Investigations on High Temperature Properties of Iridium

J. Merker[1], B. Fischer[2], D. F. Lupton[3], C. Scheckenbach[1],
R. Weiland[3], J. Witte[4]

[1] Department of Materials Engineering;
University of Applied Sciences Giessen-Friedberg, Germany

[2] Department of Materials Technology,
University of Applied Sciences Jena, Germany

[3] Engineered Materials Division,
W. C. Heraeus GmbH & Co. KG, Hanau, Germany

[4] Melting Technology, SCHOTT Glas, Mainz, Germany

Introduction

The metals of the platinum group, in particular platinum, rhodium and iridium, are characterised by their outstanding chemical stability, oxidation resistance and resistance to many molten oxides. The platinum group metals are therefore ideal materials for high temperature use under simultaneous chemical attack and mechanical loading. These metals are widely used in spite of their relatively high prices.
Iridium is the most chemically resistant of all metals, although it is more sensitive to oxidation than platinum or rhodium. Because of its high melting point (2454°C) iridium is particularly suited to applications under extreme thermal (up to 2300°C) and mechanical conditions which exclude the use of platinum alloys or rhodium. Important fields of use are e.g. in crucibles for pulling single crystals, components for manufacturing and processing high melting special glasses and also for space technology.
For the design of equipment used at high temperatures and the numerical simulation of their service behaviour under industrial conditions it is necessary to have materials data on the stress-rupture strength and creep behaviour.
These properties have been investigated in a temperature range between 1650°C and 2300°C on different semi-finished products (wire, sheet) of pure iridium. The results will be discussed on the basis of metallographic

and fracture examinations (SEM) and micro-analytical investigations by means of scanning secondary ion mass spectroscopy (scanning SIMS).

Experimental

Test Facility for the Determination of High Temperature Mechanical Properties of Metallic Materials

The stress-rupture strength and the creep behaviour of high melting metals (Pt materials, Rh, Ir, Mo, Re, W and their alloys) can be measured in our specially developed testing facility. This equipment permits measurements up to 3000°C either in air or under a protective gas atmosphere (for example argon-hydrogen mixture) [1-8].

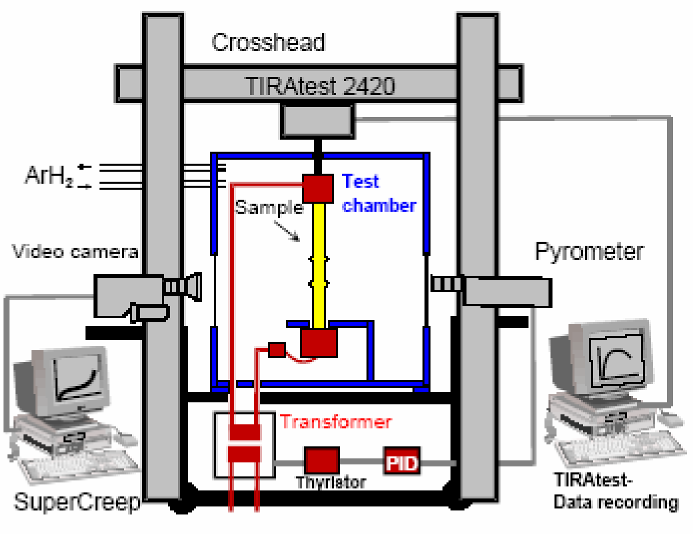

Fig. 1: Schematic diagram of the equipment used to measure stress-rupture strength and creep parameters of metals at temperatures up to 3000°C

A schematic diagram of the equipment is given in figure 1. In order to achieve high test temperatures, the sample are heated by direct electrical resistance heating. Strips, rods or wires with a cross-sectional of 1-5 mm^2 can be used as samples for the tests. The temperature is monitored by an infrared thermometer focused on the centre of the sample and connected to a controller which adjusts the sample heating current via a thyristor regulator. The small measurement point of the infrared thermometer (0.3 mm diameter) scans the length of the sample by means of a tilting mirror. The maximum value found is used for temperature measurement and control. The equipment design guarantees a constant maximum temperature throughout the test in spite of deformation of the sample. The load is applied to the sample by means of calibrated weights. The stress-rupture curve is determined by measuring the time to rupture of the samples at different loads and constant temperature. The measurements were carried out under a protective atmosphere (argon-hydrogen mixture) to prevent the evaporation of volatile oxides which would lead to a reduction in cross-section.

Samples

A special design of sample (figure 2) enables the continuous recording of the sample elongation, i.e. creep curves, with a laser scanning system or by observing it with a high resolution camera which is itself controlled by the program SuperCreep developed at the University of Applied Sciences for strain measurements using digital image analysis [4, 6, 7].

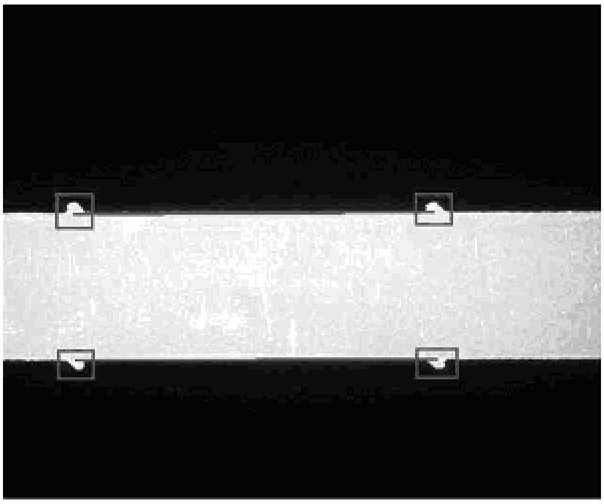

Fig. 2: Image of a tensile sample with shoulders recorded by SuperCreep

In this technique the distance between the two shoulders on each side of the sample is continuously measured. This is the central portion of the sample which has a uniform temperature. In this way it can be guaranteed that the measurement of elongation is effected without being influenced by the temperature gradient in the region of the ends of the sample. The specimens, which have shoulders of only 0.3 mm radius, are laser machined from sheet material. All creep curves presented in this paper were determined using the camera and the SuperCreep technique. Lower values for the elongation are registered when the laser scanning system is used as a result of the temperature gradient over the length of the sample.

The results given in this report were determined on samples with a length of 120 mm, a width of 4 mm and a thickness of 1 mm taken from sheet materials. The cross-section of the samples made from wire was 1x1 mm^2 with a length of 120 mm. To record the elongation of wire samples by means of SuperCreep two small wires (0.3 mm diameter) were mounted as markers on the samples at a gauge distance of 10 mm.

Results and Discussion

Stress-Rupture Strength of Pure Iridium

Stress-rupture curves of pure iridium determined on samples from sheets and wires at temperatures between 1650°C and 2300°C are given in figure 3.

The individual values obtained showed an excellent degree of reproducibility and lie almost ideally on straight lines in the stress-rupture diagram. So these results were comparable to investigations published some years before [9-12]. Measurements up to 1,000 hours permit an extrapolation of stress-rupture strength for long rupture times up to 10,000 hours (table 1). Table 1 shows typical values of stress-rupture strength of pure iridium for rupture times between 1 h and 10,000 h determined by interpolation and extrapolation respectively. There are no differences in stress-rupture strength of samples made from sheets or wires. Furthermore the results did not show any differences between different iridium heats taken from the daily production of the Heraeus company.

Iridium shows a very high stress-rupture strength at highest temperatures. Even the results determined at 2300°C offer the possibility for applications at this extreme temperature.

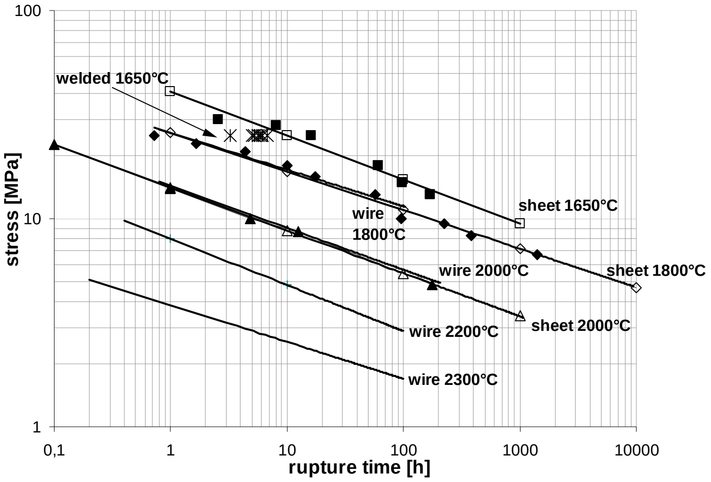

Fig. 3: Stress-rupture curves of pure iridium (wires and sheets) at different temperatures

Table 1: Stress-rupture strength of pure iridium at different temperatures for rupture times up to 10,000 hours (*by extrapolation)

Rupture time [h]	Stress-Rupture Strength [MPa] at		
	1650°C	1800°C	2000°C
1	40.8	25.8	14.1
10	25.1	16.9	8.8
100	15.4	11.0	5.5
1.000	9.5	7.2	3.4
10.000*	5.8	4.7	2.3

Creep Behaviour

The creep curves of pure iridium show a significant anomaly (figure 4).
In the range of steady-rate creep the creep curve contains plateaus, as shown in figure 4. This region is separated in time slice by an acceleration of the elongation. The acceleration of creep at these steps is clearly visible in figure 5.

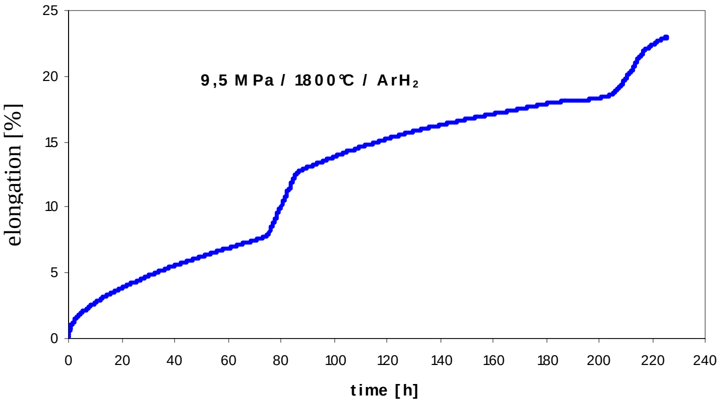

Fig. 4: Creep curve of pure iridium determined at 1800°C (load: 9.5 MPa)

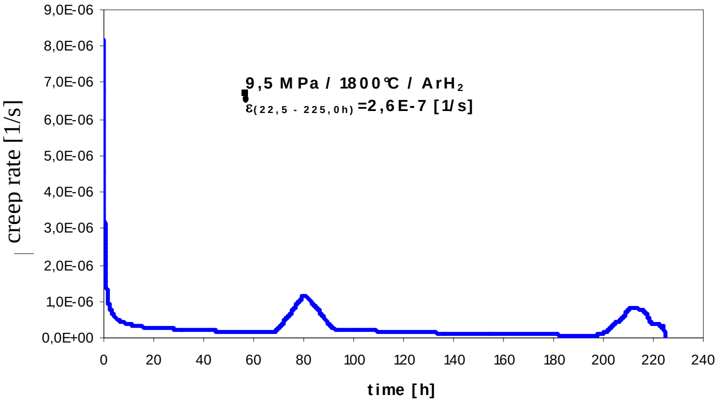

Fig. 5: Creep rate as a function of time at 1800°C

Due to this anomaly the Norton plots (figure 6) were calculated on the basis of an averages of creep rate determined for a measurement time range from 10% to 90% instead of using the minimum of creep rate.
Figure 6 shows the double logarithmic Norton plots of these calculated

stationary creep rates (averages) for pure iridium at test temperatures of 1650°C, 1800°C and 2000°C. The Norton exponent in the range between n = 5.5 to 7.4 is slightly higher than those typically found for more common pure metals (n ≈ 4 … 5) [13], but these determined values are not in a critical range.

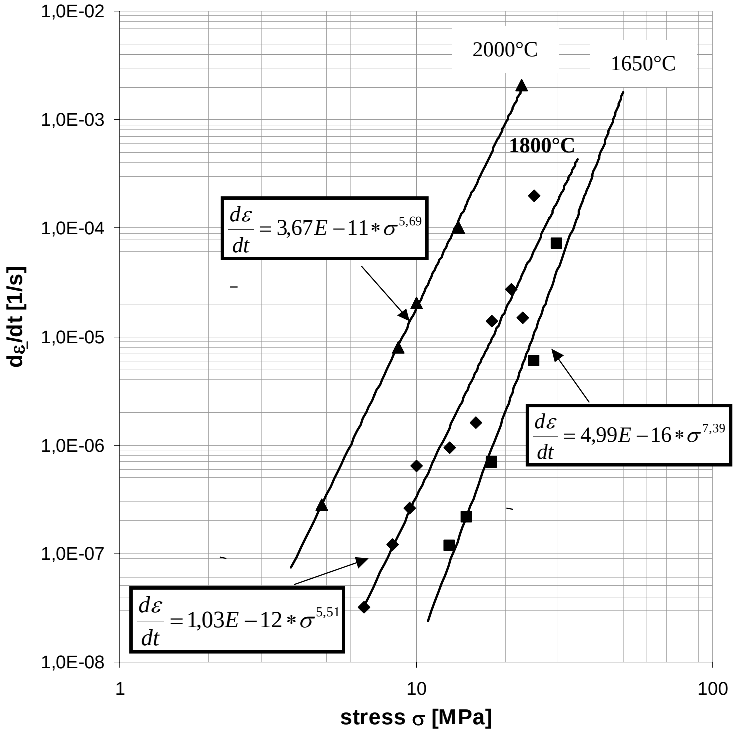

Fig. 6: Stationary creep rate of pure iridium at test temperatures of 1650°C, 1800°C and 2000°C

Iridium shows excellent ductility at the highest temperatures. Figures 7a and 7b show results of investigations of fracture surfaces by means of scanning electron microscopy (SEM). The samples show a strong necking up to a very thin knife-edge.

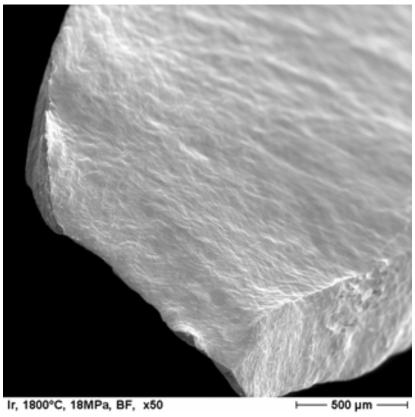

Fig. 7a: Fracture surface after creep test at 1800°C (SEM micrograph 50x)

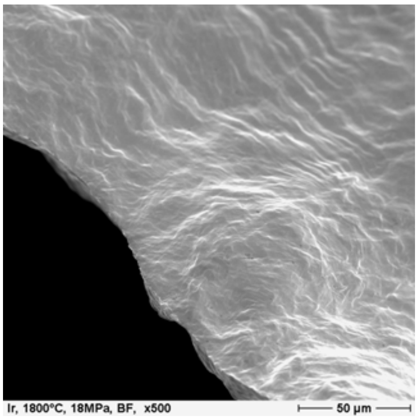

Fig. 7b: Fracture surface after creep test at 1800°C (SEM micrograph 500x)

Figures 8a and 8b show scanning electron micrographs of the surface near the fracture zone after the creep test at 1800°C. In several directions large concentrations of overlapping slip bands can be observed. This indicates that several slip systems operated successively which could be the cause of the plateaus in the creep curves as mentioned above.

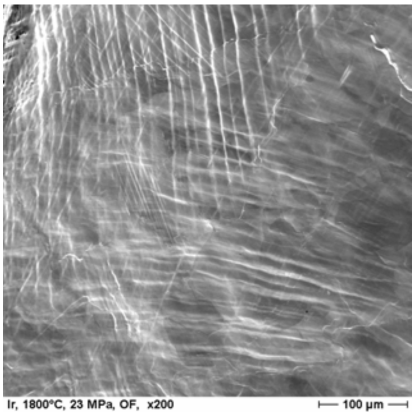

Fig. 8a:
Surface near the fracture zone after creep test at 1800°C
(SEM micrograph 200x)

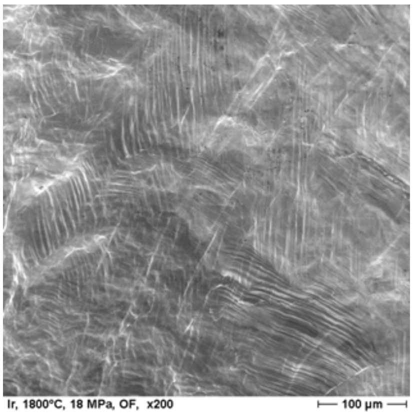

Fig. 8b:
Surface near the fracture zone after creep test at 1800°C
(SEM micrograph 200x)

Another phenomenon that could also lead to the anomaly in the creep curves was observed during the creep tests. After some hours the initiation of necking and intercrystalline cracking was observed. Some time later this damage stopped and new necking and intercrystalline cracking started again at another part of the sample and the process continued for some hours (depending on the load) until fracture occured. The cessation of the first damaging process and the start of the second period of necking and crack propagation could temporarily lead to an increasing creep rate until the adjustment of the next steady-rate creep. The scanning electron micrographs in figures 9a and 9b show intercrystalline cracking on a surface of an iridium sample generated during a creep test at 1800°C.

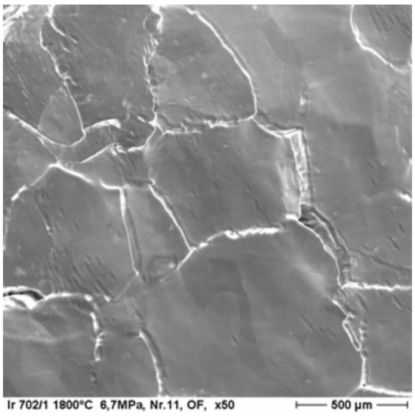

Fig. 9a:
Intercrystalline cracks on Ir surface after creep test at 1800°C
(SEM micrograph 50x)

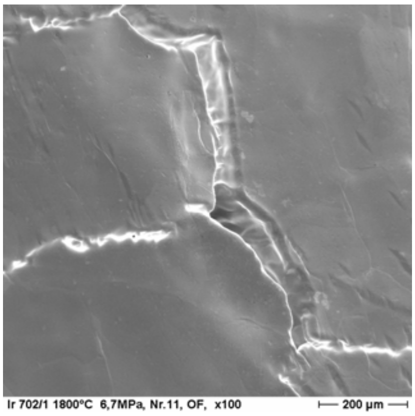

Fig. 9b:
Intercrystalline cracks on Ir surface after creep test at 1800°C
(SEM micrograph 100x)

Metallographic Examination

The aim of the metallographic examination was the evaluation of the microstructure of pure iridium depending on the influence of high temperatures and different loads during the creep tests. All investigations were carried out on longitudinal sections of samples after creep tests in comparison with the initial state. The microstructure of a sample taken from a sheet in the initial state is given in Figure 10. The sample shows a uniform structure with a grain size of about 100 µm. After the creep tests performed at test temperatures of 1800°C and 2000°C under different loads the expected grain coarsening was observed (figures 11 – 13).

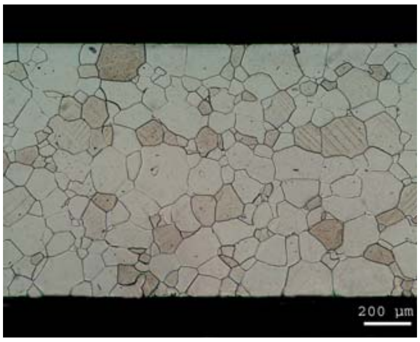

Fig. 10: Longitudinal section, Ir sheet

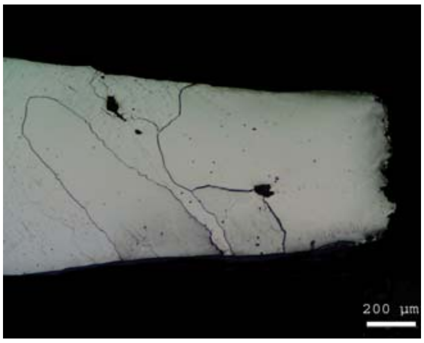

Fig. 11a:
Longitudinal section, Ir sheet
after creep test at 1800°C/13 MPa

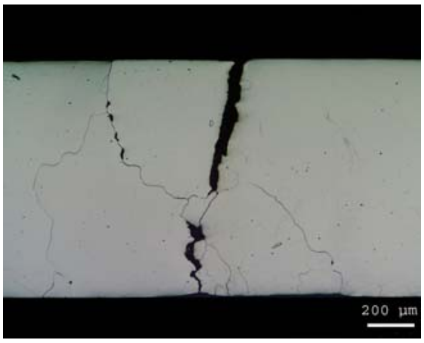

Fig. 11b:
Longitudinal section, Ir sheet
after creep test at 1800°C/13 MPa

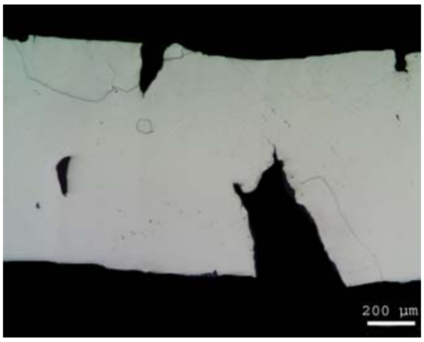

Fig. 12a:
Longitudinal section, Ir sheet
after creep test at 1800°C/6.7 MPa

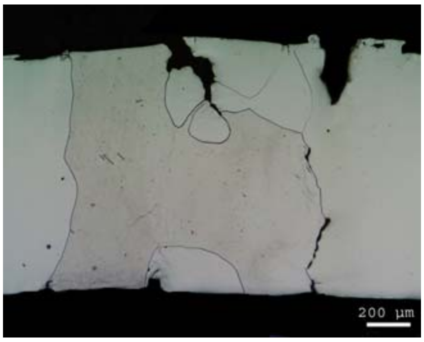

Fig. 12b:
Longitudinal section, Ir sheet
after creep test at 1800°C/6.7 MPa

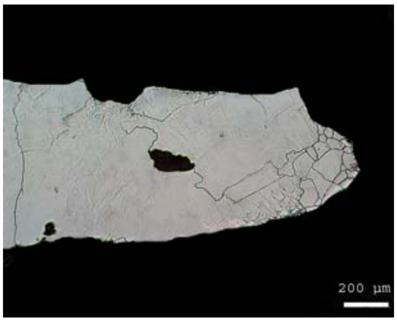

Fig. 13a:
Longitudinal section, Ir sheet
after creep test at 1800°C/14 MPa

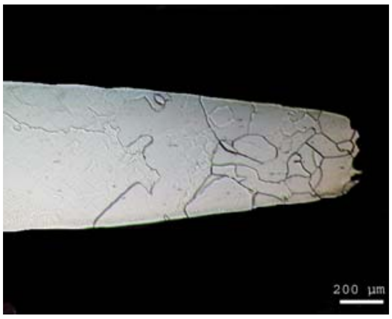

Fig. 13b:
Longitudinal section, Ir sheet
after creep test at 2000°C/14 MPa

Especially figures 11 and 12 show that pure iridium develops very coarse grains due to the high temperatures and long test time. The grain sizes after the creep test were up to 4 mm. Furthermore the samples confirmed the step by step propagation of necking and crack formation and crack stopping (mentioned above). The figures 11b, 12a and 12b show strong intercrystalline crack formation. Nevertheless the material withstood this damage and cracked at another position several hours later (e.g. figure 11a). In addition the results of metallographic examination show that in some areas of strong deformation, e.g. close to the fracture (figures 13a and 13b) and necking areas (figure 12b) dynamic recrystallisation has occured. Due to this an increase in the deformation stress is possible. As a consequence the necking or crack propagation can stop in this area and continue in another part of the sample. As a result of this process a new plateau in the creep curve can be generated.

Micro-analytical Investigations

In general iridium tends to brittle intercrystalline fracture due to trace impurities at the grain boundaries as demonstrated by previous investigations [14-20]. Investigations by means of scanning secondary ion mass spectroscopy on the iridium heats examined in this paper have shown a very high purity. Only small amounts of trace impurities (in ppm range) were detected without any enrichment at grain boundaries. All of the elements detected have shown a homogeneous distribution in the matrix.

Both this high purity and the avoidance of enrichment of trace impurities at the grain boundaries during the manufacturing process contribute to the very high stress-rupture strength and excellent ductility of the pure iridium.

Summary and Conclusions

Because of its outstanding properties iridium is particularly suited to applications under extreme thermal and mechanical conditions because of its high melting point and its chemical resistance.

The measurement of stress-rupture strength and creep behaviour up to highest test temperatures was necessary to obtain materials data for the design of high temperature equipment and the numerical simulation of their service performance.

Stress-rupture tests and the investigation of creep behaviour were performed on samples of different semi-finished products (wire, sheet) at test temperatures of 1650°C, 1800°C, 2000°C, 2200°C and 2300°C. The results have shown an excellent degree of reproducibility. All iridium samples demonstrated very high stress-rupture strength and outstanding ductility without any significant differences between sheet and wire samples or even between different materials heats. Based on long-term stress-rupture tests the prediction of stress-rupture strength by extrapolation up to rupture times of 10,000 hours was possible.

The creep behaviour of the pure iridium investigated showed a significant anomaly. In the range of the steady-rate creep the creep curve contains some plateaus. Both metallographic examination and investigations by means of scanning electron microscopy gave indications of possible causes. The activation and the effect of several slip systems were discussed as well as the influence of dynamic recrystallisation.

Micro-analytical investigations by means of scanning secondary ion mass spectroscopy have shown a very high purity of the iridium heats investigated without any enrichment of trace impurities at the grain boundaries.

Acknowledgments

The authors would like to thank Margit Friedrich (SEM investigations), Erik Hartmann and Frank Lehner (creep tests) from the Department of Materials Technology of the University of Applied Sciences Jena for their support of our investigations.

References

[1] B. Fischer, H. Töpfer, R. Helmich, German Patent DD WP G 01 N / 245 576 A3

[2] B. Fischer, H. Töpfer, R. Helmich, Silikattechnik **35**, 329-331 (1984)

[3] B. Fischer, D. Freund, D. Lupton, Tagung Werkstoffprüfung, Proceedings pp. 119-125 (1997)

[4] R. Völkl, D. Freund, B. Fischer, D. Gohlke, Tagung Werkstoffprüfung, Proceedings pp. 211-218 (1998)

[5] B. Fischer, D. Freund, D. Lupton, The Minerals, Metals & Materials Soc., Proceedings pp. 311-329 (1997)

[6] R. Völkl, B. Fischer, Experiment. Mechanics, **44**, 121-127(2004)2

[7] R. Völkl, D. Freund, B. Fischer, J. of Testing and Evaluation, 31, 1-9 (2003) 1

[8] B. Fischer, D. Lupton, A. Behrends, D. Freund, J. Merker, Platinum Metals Rev. **43**, 18-28 (1999) 1

[9] B. Fischer, D. Lupton, F. Braun, J. Merker, R. Helmich, Praktische Metallographie, Sonderband 25, 353-362 (1994)

[10] B. Fischer, D. Lupton, F. Braun, J. Merker, R. Helmich, Tagung Werkstoffprüfung, Proceedings pp. 141-145 (1994)

[11] F. Fischer, D. Freund, R. Kirchner, T. Händel, D. Lupton, J. Merker, J. Schielke, Praktische Metallographie, Sonderband 27, 405-408 (1995)

[12] D. Lupton, B. Fischer, 2nd European Precious Metals Confererence Lisbon, Proceedings (1995)

[13] R. Bürgel, Handbuch Hochtemperaturwerkstofftechnik, p. 108, Vieweg & Sohn Verlagsgesellschaft mbH, Braunschweig (1998)

[14] P. Panfilov, et al., Platinum Metals Rev., **35**, 196-200 (1991)

[15] P.Panfilov, Platinum Metals Rev., 45, 4, 179-183 (2001)

[16] H.-J. Ullrich, M. Schlaubitz, J. Merker, B. Fischer, et al. DESY-HASYLAB Annual Report, pp. 855-856 (1993)

[17] J. Merker, M. Schlaubitz, H.-J. Ullrich, D. Lupton, et al., DESY-HASYLAB Annual Report, pp. 913-914 (1994)

[18] J. Merker, D. Lupton, B. Fischer, M. Schlaubitz, et al., Praktische Metallographie, Sonderband 27, 267-270 (1995)

[29] J. Merker, D. Lupton, H.-J. Ullrich, M.Schlaubitz, B. Fischer, TMS Annual Meeting, Proceedings pp. 109-120 (2000)

[20] R. Völkl, A. Behrends, J. Merker, D. Lupton, B. Fischer, Materials Science and Engineering **A368**, 109-116 (2004)