The Tensile Properties of Iridium at High Temperatures

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In this paper the authors describe their determination of the tensile strength and ductility of iridium at temperatures up to 2000°C. The tensile strength of iridium at high temperatures, in the presence of carbon vapour, compares favourably with those of tungsten, molybdenum, tantalum and niobium, all of which are attacked to a much greater degree by the carbon.

The increasing demand for metals to operate at extremely high temperatures has necessitated the investigation of their behaviour at temperatures above 1000°C. A measurement of the tensile strength at high temperatures gives some indication as to which metals are likely to possess the necessary mechanical properties. Other factors that must be considered are vapour pressure and resistance to gaseous attack—such as oxidation or carburisation. In a recent investigation into the mechanical properties of high melting point solids, Mordike (1, 2) measured the tensile properties of the sintered refractory metals tungsten, molybdenum, tantalum and niobium. It was observed, however, that carbon vapour present in the furnace at temperatures above 1500°C was contaminating the specimens, forming the corresponding metallic carbides. In the case of molybdenum, for example, at 2200°C a molten phase was present at the grain boundaries, reducing the tensile strength to zero some 400°C below the melting point of the pure metal.

Iridium is known to possess properties (3) which make its use at high temperatures attractive despite its high cost. It is extremely resistant to corrosion and maintains its strength at high temperatures; consequently it is being used increasingly as a crucible material. In addition it was hoped that since iridium is resistant to carburisation it would indicate the extent to which this attack was affecting the tensile properties of the metals originally investigated. This paper describes some preliminary observations on the tensile properties of iridium from room temperature to 2000°C.

Apparatus

For the tensile measurements at temperatures up to 1000°C a modified Hounsfield tensimeter and furnace unit was used. The original experiments on the refractory metals were carried out in an atmosphere of argon, but oxidation rates of iridium are low and since a high rate of strain was used (30 per cent per minute) the argon atmosphere was found to be unnecessary within this temperature range. A chromel : alumel thermocouple was used to measure temperature.

In the temperature range above 1000°C a graphite tube resistance furnace was used, the working pressure within the furnace being approximately 10^{-4} mm of mercury. Black body conditions were assumed to hold for the interior of the graphite tube and an optical pyrometer was used to measure the temperature. The estimated accuracy of

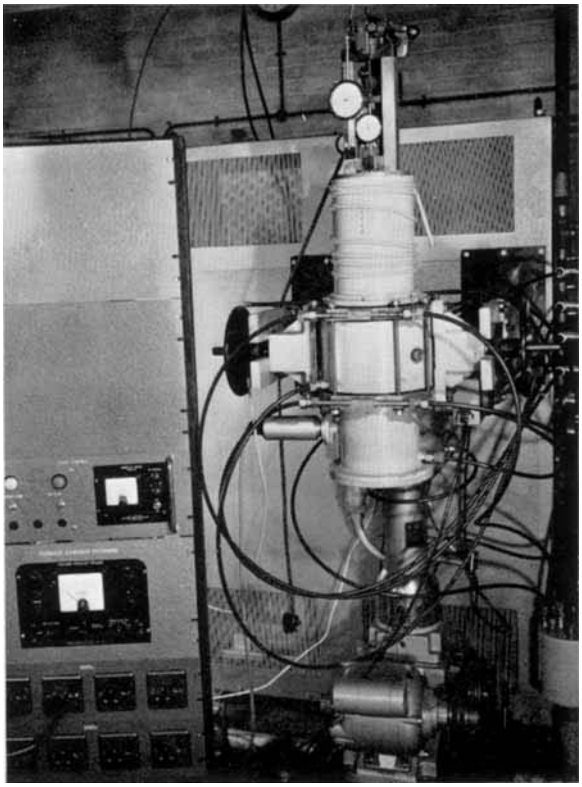


Fig. 1 General view of the apparatus used in the investigation

temperature measurement under these conditions was ±17°C. A spring type tensile device was employed, using dial gauges to record extension of the spring and the specimen elongation, while the strain rate in this case was approximately 52 per cent per minute. Details of both furnaces and the tensile measuring device are given in the thesis by B. L. Mordike (4). A general view of the apparatus is shown in Fig. 1.

Specimens

The iridium specimens were supplied by Johnson, Matthey & Co., Limited, and had been prepared from a small argon arc melted ingot. Spectrographic analysis showed it to contain the following metallic impurities only:

Rhodium	0.05 per cent
Platinum	0.01 per cent
Palladium	0.001 per cent
Copper	0.005 per cent
Silver	0.005 per cent
Iron	0.003 per cent

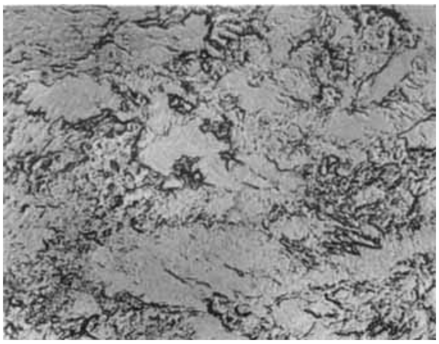


Fig. 2 Iridium in the as received condition ×75

Reduction down to the specimen thickness of 0.30 mm to 0.40 mm was carried out by hot rolling to give a very small reduction in thickness, about 0.025 mm at each pass, with interstage heating to approximately 1800°C. The gauge length of the tensile specimen was 3 cm, and either 3 or 6 mm in width. The thickness of the specimen was either 0.30 or 0.40 mm.

Metallographic examination of the metal in the original condition showed a heavily deformed cold worked structure (Fig. 2).

All the metallographic specimens shown in this paper were electrolytically etched in 10 per cent sodium hydroxide, with an alternating current of 2 amp, using iridium as the other electrode.

Results

From the dial gauge measurements obtained, a load : extension curve for each specimen was drawn; typical curves are shown in Fig. 3. Using these curves the

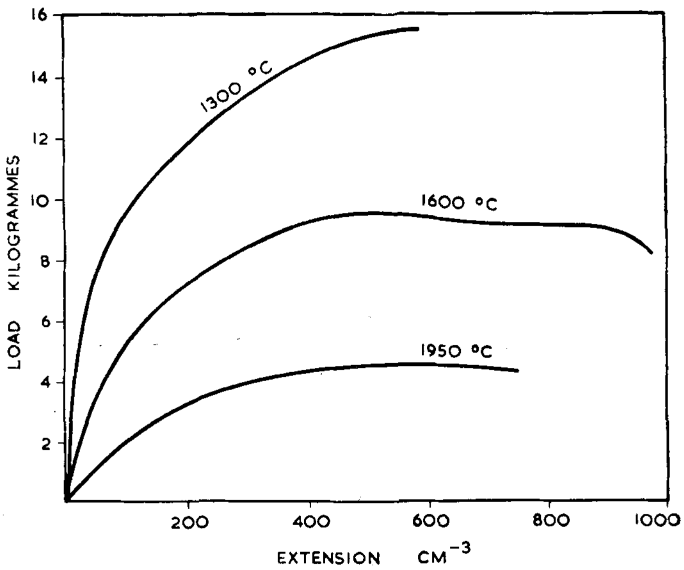


Fig. 3 Typical load: extension curves for iridium specimens

tensile strength of the specimen was obtained and also the yield stress, as indicated by a permanent extension (0.1 per cent) of the gauge length. Measurement of total elongation was made on the specimen over the gauge length of 3 cm, but measurement of reduction in area was impracticable due to the specimen dimensions. Variation of the measured tensile strength as a function of temperature is shown in Fig. 4, while Fig. 5 shows the effect of temperature on yield stress and elongation to fracture.

The results of the tensile strength tests on iridium are compared with results of tests made in the same conditions on the metals tungsten, molybdenum, tantalum and niobium in the table. In considering these results, it should be borne in mind that the other metals were all attacked by carbon vapour present in the furnace and were extensively carburised at the time of test; iridium alone was unaffected by the carbon.

Discussion

A certain amount of the scatter obtained in results at low temperature may be attributed to the effect of the cold worked structure, particularly in specimens of different thicknesses. The material in the original condition was extremely brittle and during attempts to

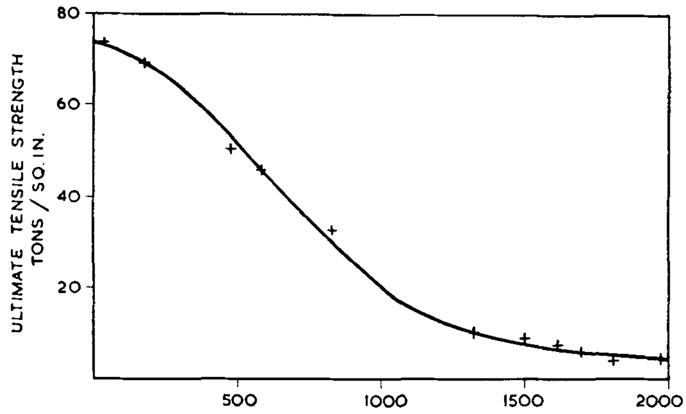
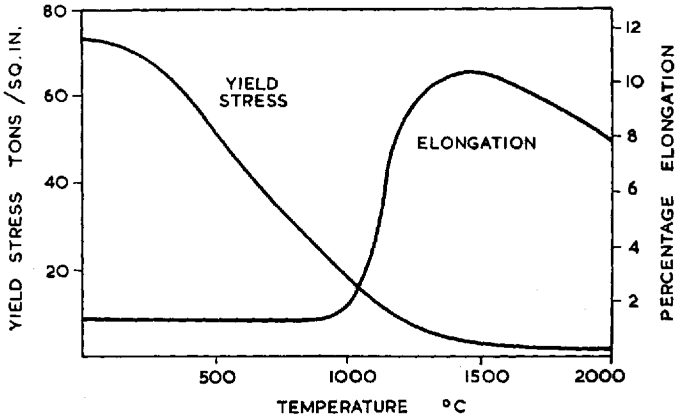


Fig. 4 Ultimate tensile strength of iridium as a function of temperature

Fig. 5 Yield stress and elongation to fracture of iridium as a function of temperature



shape the gauge length, in the initial stages of the investigation, it was observed that a certain amount of lamination appeared to be present. Conventional methods of machining either resulted in the fracture of the specimen during shaping or gave a badly damaged edge along the gauge length which considerably lowered the tensile strength at temperatures up to approximately 900°C. The method finally adopted was that of grinding with diamond impregnated wheels. Using conventionally shaped specimens a tensile strength of 68 to 100 kg/mm^2 (43 to 64 tons/in^2) was obtained, while a specimen shaped with the diamond wheel had a tensile strength of 117.9 kg/mm^2. A specimen annealed at 1600°C and pulled at room temperature had a tensile strength of 62.9 kg/mm^2 (39.9 tons/in.^{2}) with an elongation of 5.6 per cent.

The curves shown in Figs. 4 and 5 illustrate the effect of temperature on cold worked iridium. Over the temperature range from room temperature to 1300°C, the process of recrystallisation reduced the yield stress and ultimate tensile strength, while the ductility increased. Simultaneously the effect of temperature on the face-centred cubic lattice occurred, increasing the ease of slip and extending the range of plastic deformation. At temperatures up to 900°C the strength dropped rapidly, presumably due to annealing effect, but the fractures were brittle, involving some intergranular failure. This was contrary to the observations made on fractures of the body-centred cubic metals, where failure was essentially transgranular within the same temperature range. Fig. 6 shows the fracture of a specimen recrystallised at 1600°C and pulled at room temperature, clearly indicating the presence of inter-

Tensile Strengths (in tons/in^2) at High Temperatures of Iridium and Other Refractory Metals in the Presence of Carbon Vapour

	1000°C	1200°C	1400°C	1600°C	1800°C	2000°C
Iridium	20.3	12.1	6.0	4.4	3.2	2.5
Niobium	7.9	3.8	1.6	0.95	0.64	0.64
Tantalum	17.4	10.8	5.4	3.2	2.9	2.5
Molybdenum	16.8	7.9	5.1	3.2	2.2	1.3
Tungsten	20.9	16.2	12.7	10.2	7.0	4.4

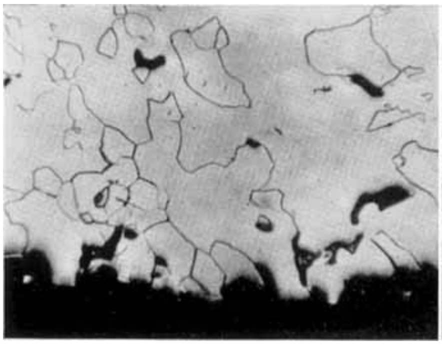


Fig. 6 Fracture of recrystallised iridium showing the presence of intergranular failure × 75

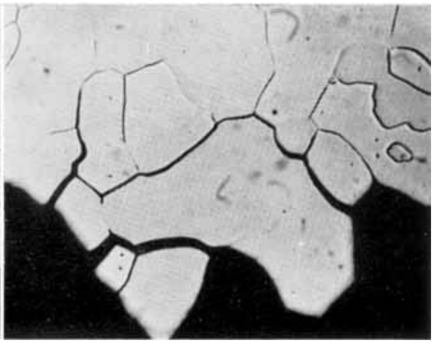


Fig. 7 Intergranular fracture of iridium at 1600°C × 75

granular failure. The high strain rate used in these experiments increased the tendency to brittle fracture and, since the extent of the brittle range varied with the different specimen sizes, it was difficult to determine the transition temperature. In the range from 900°C to 1600°C the metal became increasingly ductile and failure was partly intergranular and partly transgranular, but above 1600°C the fracture became entirely intergranular with grain boundary movement at the highest temperatures. The measurement of percentage elongation for high temperature fractures gives a false impression of the ductility, since the elongation is almost entirely due to intergranular movement with little deformation of the grains (Fig. 7).

Calverley and Rhys (5) have investigated the ductility of rhodium, which has similar mechanical properties to those of iridium. They considered that a segregation of impurity atoms might possibly increase the resistance to (111) slip. A single crystal was prepared, by the electron bombardment floating zone technique, which could withstand 90 per cent reduction in area without an intermediate anneal. Polycrystalline bars could only be deformed to a very limited extent before intergranular fracture occurred. When the wire produced from the single crystal was annealed it recrystallised and could not be further cold worked without fracture. Calverley and Rhys concluded from this that the crystal boundaries play an important part in the cold deformation of rhodium and that the presence of grain boundary impurities may also be important.

Non-uniform grain growth was observed at temperatures above the recrystallisation temperature and in one specimen the complete cross-section of the gauge length at one point consisted of a single grain. Failure, in this specimen, had occurred intergranularly in a neighbouring region of small grains, suggesting the presence of grain boundary weakness. The large twinned region shown in Fig. 8 was also observed in this specimen, and was thought to be the result of annealing twinning. Although a precipitate was not observed, it is possible that small quantities of impurities present in the iridium segregate at the grain boundaries. This segregation

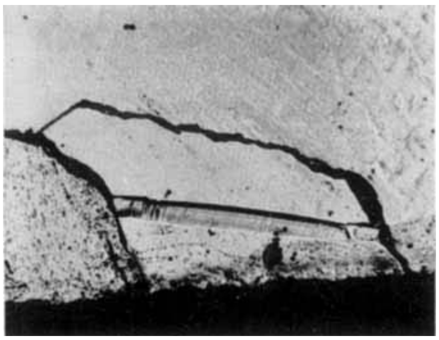


Fig. 8 Twinned region in a recrystallised iridium specimen × 94

could initiate the formation of a number of cracks at the grain boundaries, probably by a Stroh type mechanism (6), thus propagating intergranular failure. At the higher experimental temperatures it is suggested that this segregation does not occur, the distribution of the impurities remains random and a more ductile behaviour is observed. At the highest temperatures grain boundary movement occurs, and the fracture becomes entirely intergranular.

Conclusions

The tensile strength of iridium at high temperatures compares very favourably with that of the metals tungsten, molybdenum, tantalum and niobium. Of the common refractory metals only tungsten has a higher tensile strength above 1300°C and all of these metals are attacked to a much greater degree in atmospheres containing oxygen and carbon. Carbon vapour present in the furnace at high temperatures did not produce any change in the metallographic structure of the iridium.

The electrolytic etch using an alternating current and sodium hydroxide solution is most efficient, and may be usefully employed to reveal areas of high strain.

Iridium, unlike most other face-centred-cubic metals, exhibited a brittle-ductile transition and only moderate ductility at higher temperatures. Maximum ductility appeared to be within the range 1300 to 1800°C. The brittleness is considered to be due to grain boundary failure, possibly accelerated by coherent impurity segregation at these grain boundaries. It is also suggested that fracture is probably initiated by a Stroh pile up mechanism.

It is emphasised that these are no more than preliminary observations: more detailed work is in progress.

Acknowledgements

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Catalytic Reforming in the U.S.S.R.

PRODUCTION OF AROMATIC HYDROCARBONS

Russian research workers engaged on the development of petroleum reforming processes using platinum-on-alumina catalysts have designed a plant to produce not only high-octane petrol, but also a range of aromatic hydrocarbons. Two types of reforming unit developed by workers from Lengiprogas are described in a recent paper (H. B. Aspel, G. C. Golov and V. D. Pokhozaev, *Khim. i Tekhnol. Topliv i Masel*, 1960, (5), 1–7). The basis for the differentiation is a change in operating pressure. By working at a pressure of 40 atmospheres it is possible to produce high-octane petrol and xylenes, while benzone and toluene are produced at 20 atmospheres. It is shown that, by modifying and expanding existing petroleum refineries, it is possible to construct multipurpose catalytic reforming plants which may vary their products according to demand.