

Two-Phase Iridium-Based Refractory Superalloys

THEIR DEVELOPMENT AND POSSIBILITIES AS HIGH TEMPERATURE STRUCTURAL MATERIALS

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A new class of alloys based on platinum group metals, which are called refractory superalloys, is proposed. These refractory superalloys have a f.c.c. and L1₂ coherent two-phase structure (similar to that of nickel-based superalloys), high melting temperatures, and good potential as structural materials for use at temperatures up to 1800°C. In this paper, we report our results on the strength behaviour, creep property, ductility and fracture mode of iridium-based refractory superalloys.

Iridium has a high melting temperature (2447°C), the highest room temperature elastic modulus (570 GPa) (1) and is one of the most stable elements against corrosion (2). Its main use is for crucibles for growing single crystals of high melting temperature oxides, but it is also used in catalysts and in ignition devices (spark plugs).

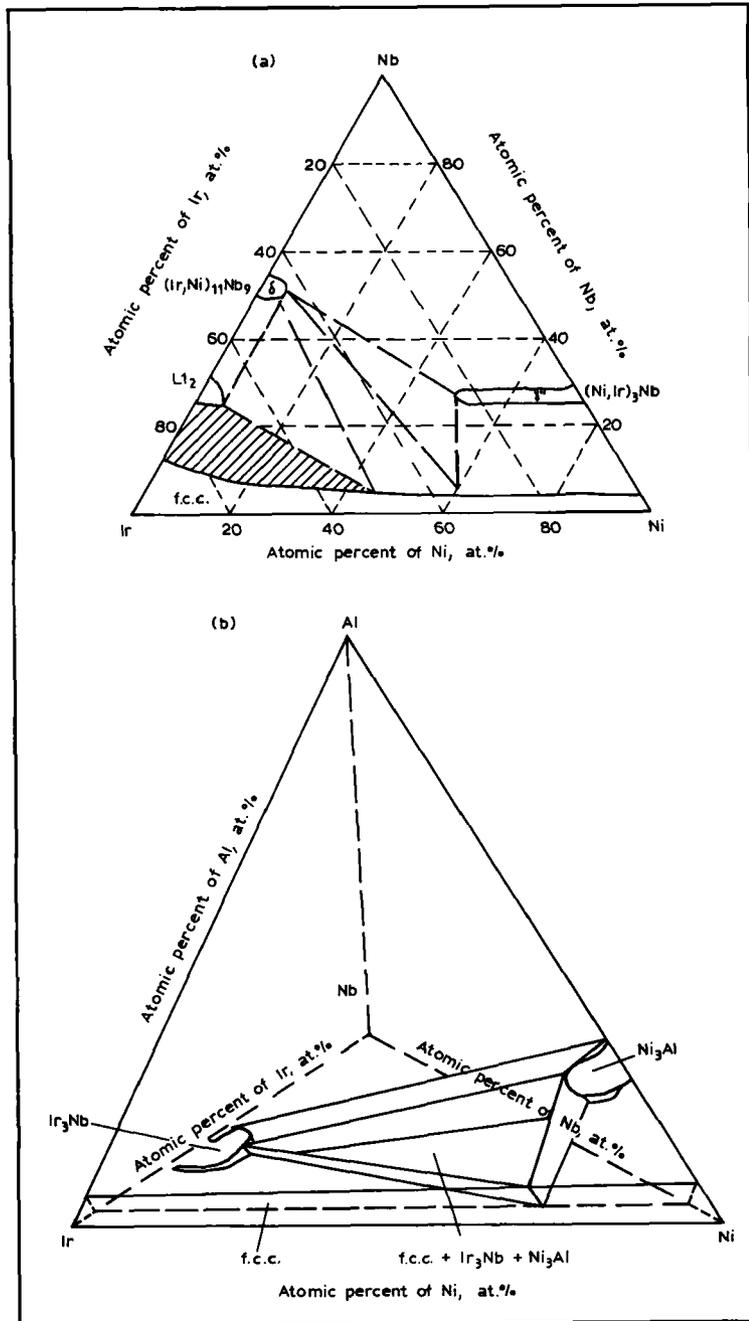
Over the last few decades, the possibility of using iridium (Ir) as a high temperature structural material has been evaluated. Several Ir-based intermetallics have been noted as having melting temperatures above 2000°C, such as IrNb with a L1₀ structure (3), IrAl with a B2 structure (4), and Ir₃Nb or Ir₃Zr with a L1₂ structure (5–6). However, Ir is an anomalous metal with a face centred cubic (f.c.c.) lattice that fails due to cleavage (7, 8).

Iridium single crystals fail in a brittle manner under tensile tests after an elongation of about 80 per cent at room temperature (9). However, polycrystalline Ir and its alloys normally exhibit intergranular or mixed intergranular and transgranular cleavage with limited ductility over a wide temperature range (10–12). Some researchers believe that intergranular fracture in polycrystalline Ir is caused by non-metallic impurities, such as carbon (C) and oxygen, or that it is environmentally induced (13). Other reports have proposed that the intergranular brittleness in polycrystalline Ir is intrinsic and not due to impurities at the grain boundary (14).

Much work has been done over the past three decades in attempts to prevent brittle fracture of Ir and its alloys and to improve their mechanical properties. Alloying (by macro-addition or micro-doping) is believed to be one of the most effective ways to achieve this. Liu and colleagues found that adding a trace amount (below 60 ppm) of thorium (Th) to an Ir-0.3W alloy (the alloy currently used as a fuel-cladding material in radioisotope thermoelectric generators) could improve its strength and ductility, and change the fracture mode from intergranular to transgranular (15). A cerium-doped Ir alloy has also shown several similarities to Th-doped alloys (16). Wolff and coworkers reported that the addition of 0.5 at.% boron (B) to an Ir-16Nb alloy could raise its strength and ductility at lower temperatures but caused a rapid fall in strength above 1100°C (17). Recently, Heatherly and colleagues investigated the effects of impurities in Ir and found that iron, nickel (Ni), chromium or aluminium (Al) at levels ranging from 50 to 5000 ppm do not embrittle Ir, whereas high levels of silicon cause severe embrittlement (18).

While these research projects concentrated on single-phase alloys based on Ir (which may be less resistant to creep deformation than two-phase alloys) we focused on two-phase Ir-based alloys consisting of f.c.c. and L1₂ phases (19). We have proposed a new class of alloys based on Ir with a f.c.c. and L1₂ coherent two-phase structure similar

Fig. 1 Phase diagrams of iridium alloys, determined experimentally:
 (a) the tertiary system Ir-Nb-Ni at 1300°C
 (b) the quaternary system Ir-Nb-Ni-Al at 1300°C



to that of Ni-based superalloys, and named them 'refractory superalloys' (20, 21). The coherent interface in the alloys appears to play an important role in strengthening alloys by preventing dislocation movements. We considered that if the Ir-based alloys (with melting temperature above 2000°C) have a f.c.c. and L1₂ two-phase coherent structure, then the alloys should show high

strength at high temperature. Such an alloy could then be used at high temperature in situations where Ni-based superalloys cannot be used. A brief introduction to our primary work was given in this Journal by Wolff and Hill (22). Here, our recent results on the strength behaviour, creep

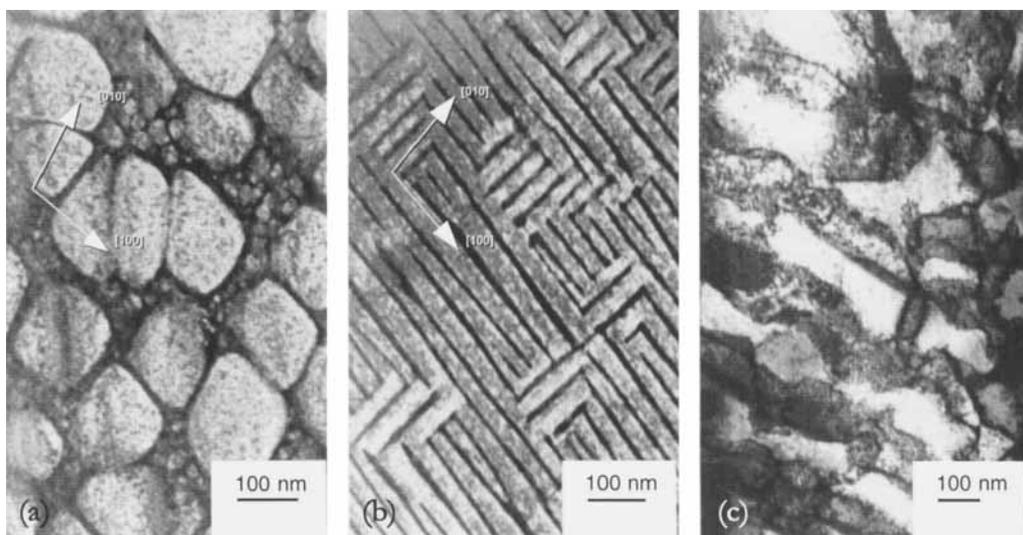


Fig. 2 Precipitate shape of (a) Ir-15 at.% Nb and (b) Ir-15 at.% Zr alloys. These dark-field images were taken from superlattice reflections from the $L1_2$ phase. (c) Bright-field image of an Ir-15 at.% Zr alloy

property, ductility and fracture mode of Ir-based alloys developed under the High-Temperature Materials 21 Project are reported.

Design Concept of Ir-Based Refractory Superalloys

According to the Ir binary phase diagrams given in Massalski (23), the f.c.c. and $L1_2$ two-phase region exists, for example, in the Ir-V, Ir-Ti, Ir-Nb, Ir-Ta, Ir-Hf and Ir-Zr systems. We investigated the strength behaviour and deformation structure in these alloys (24, 25), and Ir-Nb and Ir-Zr were found to be the most promising alloys for study with regard to their strength and microstructure up to 1200°C. However, the strength of these two binary alloys dropped off drastically above 1200°C. In attempts to improve the high-temperature strength, additions of molybdenum (Mo), tantalum (Ta) and tungsten (W) with high melting temperatures (2617, 2977 and 3380°C, respectively) were added to the Ir-Nb alloy.

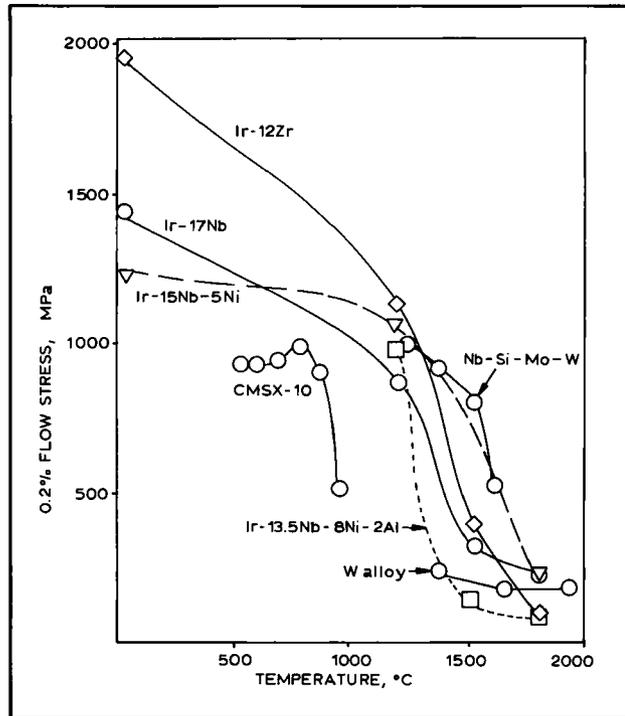
Another problem is that Ir-based alloys are very brittle and break by the intergranular fracture mode (26). In attempts to improve their ductility and change the fracture mode, different elements were added as the third element to the Ir-Nb alloy. In Ni-based superalloys, B is the element added to improve grain boundary strength and C is added

to stabilise the grain boundary. Therefore B and C were expected to improve grain boundary strength in Ir-based alloys. In addition to these trials, platinum (Pt), Ni and rhodium (Rh) were also added to Ir-Nb alloys to characterise the structural connection and to find a suitable quantity of each to replace some of the Ir. These added elements may help to improve ductility.

Another trial involved combining the two-phase Ir-Nb and Ni-Al alloys and also the two-phase Ir-Nb-Ni and Rh-Nb-Ni alloys in order to check the two-phase regions in the combined systems. These two quaternary alloys can be expected to have the advantages of both systems – high strength at high temperature from the Ir-Nb and Ir-Nb-Ni alloys, and good ductility and low density from the Ni-Al and Rh-Nb-Ni alloys. These quaternary alloys could be described, respectively, as an Ir-Ni-based alloy or as an Ir-Rh-based alloy, containing some f.c.c. or $L1_2$ phase-forming elements.

Experimentally determined phase diagrams of Ir-Nb-Ni and Ir-Nb-Ni-Al at 1300°C are shown in Figure 1 (27, 28). In the Ir-Nb-Ni system, the f.c.c. and $L1_2$ two-phase region expanded on addition of Ni. However, when too much Ni was added a third phase, $(Ir, Ni)_{11}Nb_9$, was formed. In the Ir-Nb-Ni-Al system, contrary to our expectations,

Fig. 3 Comparisons of the temperature dependence of the compressive strengths of four iridium-based alloys: Ir-12Zr, Ir-17Nb, Ir-15Nb-5Ni and Ir-13.5Nb-8Ni-2Al; with the nickel-based alloy (CMSX-10) (29), a niobium-based alloy (Nb-Si-Mo-W) (30), and a tungsten-based alloy (31)



the f.c.c. and $L1_2$ two-phase region was not connected from the Ni-Al side to the Ir-Nb side. Instead, a three-phase region, the f.c.c. and $L1_2$ -Ir₃Nb and $L1_2$ -Ni₃Al, appeared. However, there was no other phase of different structure in addition to the f.c.c. and $L1_2$ phase in the alloys that were tested. The quaternary Ir-Nb-Ni-Al alloy is very promising from the point of view of phase structure. Although exact phase diagrams for the Ir-Nb-Mo, Ir-Nb-Ta, Ir-Nb-W, Ir-Nb-Pt, Ir-Nb-B and Ir-Nb-C systems cannot be provided yet, the f.c.c. and $L1_2$ two-phase structure was confirmed in the following alloys: Ir-15Nb-5Mo, Ir-15Nb-Ta, Ir-15Nb-10W, Ir-15Nb-30Pt (all measured in at.%) and in Ir-15 at.% Nb-500 ppm B, and Ir-15 at.% Nb-500 ppm C, by observation of their microstructure. For the Ir-Rh-Nb-Ni system, the f.c.c. and $L1_2$ two-phase region was observed over a wide area.

High-Temperature Strength Precipitate Morphology Effect

Precipitate morphology depends on the lattice misfit between the f.c.c. matrix and the $L1_2$ precipitates (21). Typical microstructures are shown in Figure 2. In the Ir-Nb alloy with a small lattice misfit of 0.4%, cuboidal $L1_2$ precipitates formed, see

Figure 2a. However, plate-like precipitates formed in the Ir-Zr alloys where there was a large lattice misfit of 2% (Figure 2b), and a semi-coherent structure with many misfit dislocations also formed (Figure 2c). Plots of the temperature dependence of the strength showed that the strengths of the Ir-Nb and Ir-Zr alloys were very high (> 1000 MPa) below 1200°C, although the strengths decreased dramatically above 1200°C (Figure 3). (Figure 3 also shows the strengths of a typical commercial Ni-based superalloy, CMSX-10 (29), a Nb alloy (30) and a W alloy (31) plotted for comparison as other high-temperature materials.) In both the Ir alloys the volume fraction of the $L1_2$ precipitates was 50%. Below 1200°C, the strength of the Ir-Zr alloy was higher than that of the Ir-Nb alloy. In these two alloys, solid-solution hardening and precipitation-hardening effects were both observed (32), with precipitation hardening being larger in the Ir-Zr alloy.

The deformation mode in the Ir-Zr alloy was by shearing (25); on the other hand, shearing did not occur in the Ir-Nb alloy (33). In the Ir-Zr alloy, when a dislocation moves in the f.c.c. matrix, it

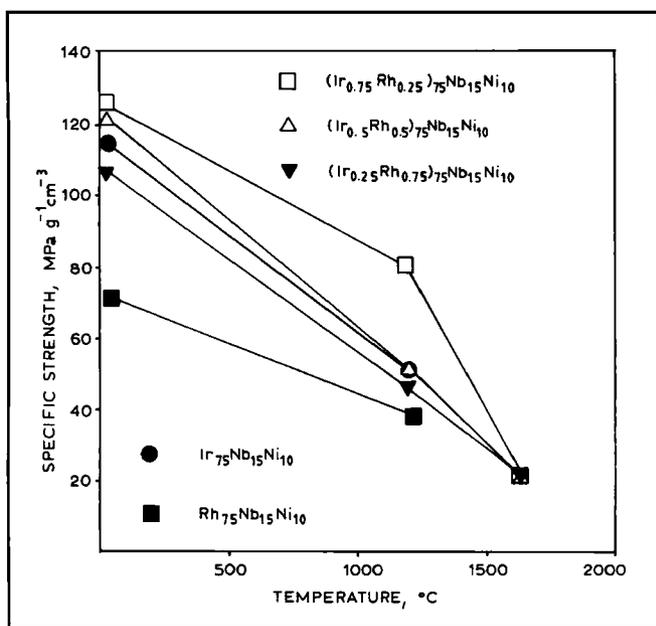


Fig. 4 The temperature dependence of the specific strength of $(Ir, Rh)_{75}Nb_{15}Ni_{10}$ alloys. Small additions of rhodium produce the strongest alloy

meets numerous interfaces in the maze structure. The coherent interface has high-coherency strain energy in the maze structure, and a large number of misfit dislocations will prevent the movement of dislocations in a semi-coherent structure. This could be attributed to the high precipitation hardening of the Ir-Zr alloy.

The Effect of Element Addition

In attempts to improve the high-temperature strength of Ir-based alloys above 1200°C, we added Mo, Ta and W to a two-phase Ir-15Nb alloy. Only Ta, at concentrations < 20 at.%, was found to be effective at improving the high-temperature strength. Additions of Ta > 20 at.% made the Ir-15Nb lose the f.c.c. and L₁₂ two-phase structure, causing the alloy strength to drop greatly at 1200°C. Adding W and Mo to the Ir-15Nb alloy only slightly improved its high-temperature strength even though W has a higher melting temperature and larger atomic size than Ta. Similar behaviour was also observed in the Ir-Nb-Ni and Ir-Nb-Ni-Al alloys (Figure 3). These results showed that a third element is not very effective at improving the strength at temperatures over 1200°C.

One of the biggest obstacles to using a two-

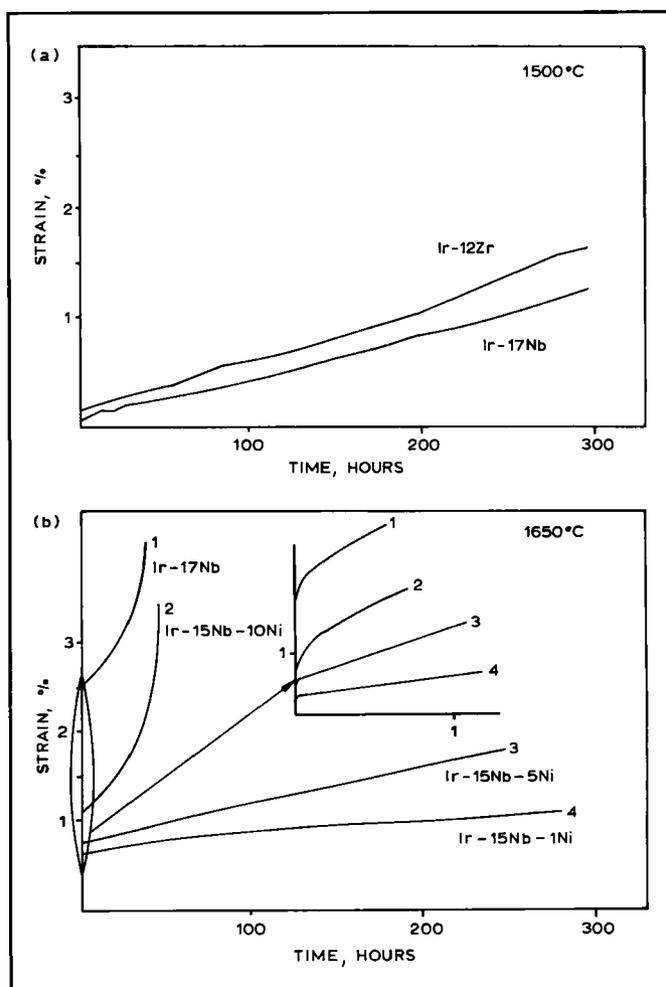
phase Ir-based alloy as a structural material at ultra-high temperatures may come from its lower specific strength (normalised strength to density), which is mainly caused by its higher density. To reduce the density, we tried replacing Ir by Rh in an Ir-15Nb-10Ni alloy, which has higher strength at both room and high temperature. The effects of the replacement on the strength and ductility are shown in Figure 4. The quaternary $(Ir_{75}Rh_{25})_{75}Nb_{15}Ni_{10}$ two-phase alloy had the highest specific strength of all the tested $(Ir, Rh)_{75}Nb_{15}Ni_{10}$ alloys: 126 MPa g⁻¹ cm⁻³ at room temperature, 81 MPa g⁻¹ cm⁻³ at 1200°C, and 24 MPa g⁻¹ cm⁻³ at 1600°C.

In the trial described previously, after the Ir-Nb alloy had been combined with the Ni-Al alloy, the density decreased; for example, the density of the Ir-10Nb-42Ni-8Al alloy with three phases was 14.8 g cm⁻³. However, the strength and melting temperature also decreased drastically.

Creep Properties

Compressive creep curves of the Ir binary and ternary alloys at 1500 and 1650°C are shown in Figure 5. Although the strength of the Ir-based binary alloys above 1500°C was not very high, the creep strain was below 2% and tertiary creep was

Fig. 5 Creep curves
 (a) for the binary iridium alloys Ir-12Zr and Ir-17Nb at 1500°C under 137 MPa
 (b) for Ir-17Nb alloy and nickel-containing iridium-niobium alloys at 1650°C, also under 137 MPa. The alloy containing the least amount (1 at.%) of nickel shows the best creep property



not observed until 300 hours. The creep resistance was higher in the Ir-Nb alloy than in the Ir-Zr alloy because discontinuous coarsening occurred from the grain boundary in the Ir-Zr alloy and its microstructure changed to a coarse structure during creep (34). This was due to the large lattice misfit in the Ir-Zr alloy. In the Ir-Zr alloys, the coherency strain energy of the interface was very high and coarsening of the maze structure was difficult to achieve. Discontinuous coarsening was also observed in the lamellar structure of the Ti-Al alloy, for example (35). When the lamellae are very fine, coarsening often occurs by migration of the grain boundary (36).

At 1650°C, a tertiary creep was observed clearly in the binary Ir-Nb alloy after 20 hours, but adding Ni to this alloy improved its creep resistance dramatically. As long as the Ni content is below 5 at.%, tertiary creep is not observed. The creep strains were below 2% after 300 hours for Ir-15Nb-xNi alloys ($x < 5$). The steady-state creep rate for the Ir-15Nb-1Ni was $1.2 \times 10^{-8} \text{ s}^{-1}$, about three orders of magnitude lower than that of the

binary Ir-17Nb alloy (10^{-5} s^{-1}). The values of the steady-state creep rates for the Ir-15Nb-5Ni and Ir-15Nb-10Ni alloys were $2.1 \times 10^{-8} \text{ s}^{-1}$ and $1.2 \times 10^{-7} \text{ s}^{-1}$, respectively. The great improvement in creep resistance of the Ir-15Nb alloy on adding Ni might be due to the effect of Ni on improving the grain-boundary strength and reducing the coarsening process.

Ductility and Fracture Mode

Our previous investigation showed that polycrystalline binary Ir-based two-phase alloys normally exhibit intergranular fracture with limited ductility even in compression tests, as does pure Ir and its single-phase alloys (26). This result implies that the grain boundary in binary Ir-based two-

phase alloys is still a weak point. The large difference between the cohesion of the grain boundary and of the bulk is likely to cause the grain boundary to break before any dislocations form, as discussed by Hack *et al.* (37).

An interpretation of the enhanced ductility in alloys prone to intergranular fracture (which happens in many intermetallic alloys and other alloy systems) based on improved grain-boundary cohesion caused by B segregation has been at least partially successful. The fracture behaviour and compression properties of the Ir-15Nb alloy doped with 80 to 2000 wppm B were investigated. The results showed that doping with B can change the fracture mode from intergranular (for the binary Ir-15Nb alloy) to transgranular (for the B-doped alloys). However, we found that doping with B only slightly improves the ductility of the alloy. We also found that even though the fracture mode for Ir-15Nb can be changed from intergranular to transgranular by adding Ni, W, Ta, Pt or Ni-Al, there is no obvious improvement in compression ductility by this change. The main reason is that the Ir-15Nb alloy, despite having additions of various elements, still fractures by transgranular cleavage at room temperature. This is due to apparently very strong and directed atomic binding forces.

To stabilise the structure of polycrystalline Ni-based superalloys against high-temperature deformation, carbide formation is required. However, C is reported to be the main impurity causing polycrystalline Ir to crack in intergranular fracture (13). Our research has found no harmful effects due to C additions on the properties of the two-phase Ir-Nb refractory superalloy, even when the C additions were up to 2000 wppm. The compression ductility for C-free and C-doped alloys had almost the same value.

Possibilities for Ir-Based Refractory Superalloys

The high-temperature strength of Ir-based refractory superalloys above 1200°C did not improve on addition of a third element. Compared with the Nb-Si-Mo-W alloy in Figure 3, the strength of the Ir-based alloys above 1200°C is not remarkable considering their high melting temper-

ature. On the other hand, the creep property of the Ir-based alloys is remarkable. We tested a Ni-based superalloy, TMS-75, which has a rupture life of 196 hours under 98 MPa in a tensile condition at 1150°C (38). Under compressive stress at 1200°C, the sample buckled, and the strain could not be measured accurately. Another comparative test indicated that the compressive creep strain rates of an IrAl single-phase alloy with a B2 structure at 1100°C were between 10^{-6} and 10^{-5} s⁻¹ under 100 to 220 MPa (39). The strain rate of IrAl at 1100°C was one or two orders of magnitude larger than that of our alloys (10^{-7} s⁻¹) at 1500°C.

This shows that our alloys, with the f.c.c. and L1₂ two-phase structure are more promising materials because of their high creep resistance. We also found that the creep life of Ir-Nb increased dramatically at 1650°C by addition of a third element, such as Ni. This shows that the Ir-based refractory superalloys may possibly be regarded as ultra-high temperature materials. Furthermore, the change in fracture mode on addition of Ni showed that there is a potential for designing high-temperature Ir-based alloys with both high-temperature strength and good ductility by addition of a suitable element.

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