Compressive Strength and Creep Properties of Ir-Nb-Zr Alloys between 1473 and 2073 K

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The microstructure and strength at 1473 and 2073 K and creep properties at 2073 K were investigated in three Ir-Nb-Zr alloys with the fcc and L12 two-phase structure. The microstructure and lattice misfit were investigated using scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffractionmetry (XRD). Compression and creep tests were performed, and their deformation structures were observed using SEM and TEM. At 1473 K, the strength of the Ir-Nb-Zr alloys was higher than that of the binary Ir-Nb and Ir-Zr alloys, but they were almost equivalent at 2073 K. However, the ternary alloys showed great improvement on creep at 2073 K. The time for the 2 pct creep strain of the Ir-Nb-Zr alloy was about 100 hours, while it was 1 hour for the binary alloys. The deformation mechanisms for compressive strength and creep resistance in these Ir-Nb-Zr alloys are discussed in terms of the deformation structure.

I. INTRODUCTION

There is the demand for high-temperature materials that can be used at around 2073 K in new and challenging application fields, such as a heat exchanger in a ram jet engine or a thruster in a satellite. However, the suitable application fields, such as a heat exchanger in a ram jet that can be used at around 2073 K in new and challenging application fields, such as a heat exchanger in a ram jet engine or a thruster in a satellite, have not yet been studied systematically. In this study, the microstructure and high-temperature mechanical properties of the Ir-Nb-Zr alloys with the fcc and L12 two-phase structure were investigated for first time. Then, the strength and creep properties were discussed in terms of the lattice misfit, precipitate morphology, and deformation structure.

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II. EXPERIMENTAL PROCEDURES

Three alloys with nominal compositions of Ir-12.5Nb-3Zr, Ir-8.5Nb-6Zr, and Ir-4.25Nb-9Zr (at. pct), were prepared as 15 g button ingots by the arc-melting method. These alloy compositions are located on the line between the Ir-17 at. pct Nb and Ir-12 at. pct Zr alloys with 50 pct of the volume fraction of the L12 precipitates in each binary system, as shown in Figure 1. In Figure 1, the square symbols show the nominal composition. The solid symbols represent the actual composition of the alloy, determined experimentally by electron-probe microscopy analysis (EPMA) in a previous study. The composition of alloy A deviates from the nominal composition, but the others almost have the targeted compositions.

Cylindrical samples, 3 mm in diameter and 6 mm in height, were cut from the button ingots and heat treated at 1773 K for 72 hours in a vacuum furnace. The samples were then cooled in the furnace. These heat-treated samples were sliced into 200-μm-thick disks for all samples. One piece of the sliced sample in each alloy was embedded in a resin, mechanically ground with sandpaper, and then electrolytically etched in an ethyl alcohol solution of 5 pct HCl to observe the microstructure using scanning electron microscopy (SEM, PHILIPS* XL30). They were also used for the investigation of lattice parameters by X-ray diffractometry (XRD). The measured 2θ was between 40 and 120 deg. Each peak from the fcc or L12 phases was clearly detected using XRD. Then, the lattice parameter was determined by least-squares refinements. The lattice misfit (δ) was calculated by the following equation:

\[ \delta = \left( \frac{a_f - a_{L12}}{a_f} \right) \]

where \( a_f \) and \( a_{L12} \) are the lattice parameters of the fcc and the L12 phases, respectively. Other sliced samples were polished to obtain a thickness of 30 μm using a dimpling machine.

Fig. 2—Secondary electron images of (a) alloy A (Ir-12.5 at. pct Nb-3 at. pct Zr), (b) alloy B (Ir-8.5 at. pct Nb-6 at. pct Zr), and (c) alloy C (Ir-4.25 at. pct Nb-9 at. pct Zr) heated at 1773 K for 72 h.

They were then ion milled into thin foils to observe the precipitate morphology using transmission electron microscopy (TEM, PHILIPS CM200).

The cylindrical samples heat treated at 1773 K for 72 hours were used for compression tests at 1473 and 2073 K and for compression creep tests at 2073 K under 137 MPa. The test was carried out at a strain rate of 10^-4/s in a vacuum atmosphere using an Instron 8560 or a Shimazu (Kyoto, Japan) AG-I testing machine. Before testing, the sample was kept at the testing temperature for 15 minutes. The resulting deformation structures were characterized using SEM and TEM techniques.

III. RESULTS

A. Microstructure

The typical microstructures of alloys heat treated at 1773 K for 72 hours are shown in Figures 2(a) and (b). In alloy A,
the dendritic structure with the fcc structure was present (Figure 2(a)). A coarse fcc and L12 lamellar structure was observed around the dendrite arms. Fine precipitates were observed in the dendrite arms as well as in the interdendrite area. From the contrast of the image, we can identify the fine L12-phase precipitates in the fcc phase and the fine fcc-phase precipitates in the L12 phase. In alloy B, the microstructure consisted of dendrite arms and the coarse fcc particles with fine L12 precipitates (Figure 2(b)). Between dendrite arms, the L12 phase with fine fcc precipitates was observed. In alloy C, irregularly shaped coarse fcc and L12 phases with fine precipitates were observed in both phases.

The precipitate morphology of alloys A through C was observed using TEM. Dark-field images from the L12 phase are shown in Figures 3(a) through (f). As shown in the observation by SEM, the microstructure was not homogeneous. Thus, we found a different precipitate morphology in each alloy. Broadly, the structures can be classified into three groups. The first group (type A) includes fine cuboidal L12 precipitates in the fcc phase 100 nm in size, as shown in Figures 3(a), (c), and (f). In this case, the precipitates are surrounded by {100} habit planes, and they tend to align along the [001] orientation. Furthermore, we also found platelike fcc precipitates in the cuboidal L12 phase (Figures 3(a) and (f)). These fcc plates formed when the composition of precipitates with a nonequilibrium state formed from the as-cast microstructure, and then the composition changed to the equilibrium state from the nonequilibrium state during the heat treatment. The second group (type B) is a platelike fcc phase in large L12 particles, as shown in Figures 3(b), (d), and (g). The habit planes of these fcc plates were also the {100} planes. The group A and B structures were coherent, but the third group (type C) was an incoherent or semicoherent L12 phase with an irregular shape, as shown in Figures 3(e) and (h). The maze structure observed in the Ir-Zr alloy was not found in these alloys.

B. Lattice Misfit

The lattice parameters of the fcc and L12 phases of the alloys are shown as a function of the Zr composition in Figure 4. For reference, the lattice parameters of the Ir-based binary alloys represented by the Ir-15Nb and Ir-15Zr binary alloys are also plotted. Although we do not have the lattice-parameter data for the Ir-17Nb and Ir-12Zr alloys, it is considered that the lattice misfits of each binary alloy are equivalent in the two-phase region. The lattice parameters of the fcc phase of the alloys did not depend on the alloy composition. However, the lattice parameter of the L12 phase increased with increasing Zr content, because the lattice parameter of Ir3Zr is larger than that of Ir3Nb. The lattice misfit estimated from the data in Figure 4 is shown in Figure 5. The lattice misfits of ternary alloys were between

![Fig. 3—Dark-field images of (a) and (b) alloy A (Ir-12.5Nb-3Zr), (c) through (e) alloy B (Ir-8.5Nb-6Zr), and (f) through (h) alloy C (Ir-4.25Nb-9Zr) heat treated at 1773 K for 72 h. These images were taken from the L12 superlattice spot.](image-url)
Fig. 4—The lattice parameters of the Ir-Nb-Zr alloys heat treated at 1773 K for 72 h as a function of the Zr concentration.

Fig. 5—The lattice misfit of the Ir-Nb-Zr alloys as a function of the Zr concentration.

Fig. 6—The compressive strength of theIr-Nb-Zr alloys at 1473 and 2073 K.

Fig. 7—The creep curves of the Ir-Nb-Zr alloys at 2073 K under 137 MPa.

D. Deformation Structure

Figure 8 shows the deformation structure observed using SEM of alloy A (Ir-12.5Nb-3Zr), tested at 1473 and 2073 K. The dendritic structure did not change during the test at both testing temperatures (Figures 8(a) and (d)). It is seen from thehigh-magnification images (Figures 8(b) and (e)) that coarsening did not occur, and the cuboidal precipitates retained their size and shape. However, cracks at the interdendrite region were observed in Figures 8(c) and (e). The deformation structure was almost equivalent at 1473 and 2073 K. On the other hand, although the samples retained their dendritic structure after the creep test at 2073 K, their microstructure clearly coarsened, as shown in Figures 9(a) and (b). The fine precipitates in the fcc phase changed to coarse particles (shown in Figure 9(a)) or a rod or plate shape (shown in Figure 9(b)). The coarse L12 phase was also observed in the interdendrite region. Cracks were also observed between fcc particles or the fcc/L12 interface.

The deformation structure in the transverse cross section of alloy A (Ir-12.5Nb-3Zr) after the compression test was observed in TEM (Figures 10(a) through (d)). After the compression test with the strain of 4.8 pct at 1473 K, a large number of dislocations bowed out and made dislocation loops in the fcc-phase channels in the type-A structure (the fcc phase with cuboidal L12 precipitates) (Figure 10(a)).
A similar structure was observed in the Ir-Nb alloy tested at 1473 K.\cite{14} Although the Burgers vector was not determined in this observation, they are $a/2(110)$-type dislocations, based on the analysis of dislocations observed in the Ir-Nb alloy. This structure is similar to that of Ni-based superalloys during the incubation period of creep.\cite{15,16} Using the stereo imaging method, it has been established that these dislocations in Ni-based superalloys are located in the fcc matrix channel.\cite{15}

Thus, the dislocation loops observed in the Ir-Nb-Zr alloy are in the fcc matrix channel. We believe that we observed the dislocation loops, which were located in the horizontal channel to the applied stress. In the type-B structure ($L_1$ phase with platelike fcc precipitates), a few dislocation loops were observed (Figure 10(b)). These dislocation loops expanded from the platelike fcc phase. We believe that we observed the dislocation loops, which spread in the invisible fcc channel normal to the visible fcc channel in the micrograph. In Figures 10(c) and (d), the deformation structure of the sample tested with the strain of 0.86 pct at 2073 K is shown. In the type-A structure, dense dislocations were observed in the whole area studied (Figure 10(c)).

Three kinds of images of alloy A (Ir-12.5Nb-3Zr), taken under different diffraction conditions using TEM after the creep test at 2073 K under 137 MPa, are shown in Figures 11(a) through (c). In most of the area, the precipitates changed to an irregular, long shape. Thus, it is considered that all three kinds of structures (types A through C) changed to the same irregular structure. Compared with the secondary

The dislocations were spread more widely than in the sample tested at 1473 K, and a “zigzag” morphology was observed in many dislocations. The “zigzag” morphology consisted of dislocations along the $(110)$ and $(100)$ orientations, as shown by the arrows marked “s” and “m,” respectively. This suggests that mobile dislocations with different Burgers vectors meet and react and, as a result, dislocations along the $(110)$ slip direction with a curve along the mismatch $(100)$ orientation form, as reported by Field et al. in crept Ni-based superalloys.\cite{17} In the type-B structure as well as in the sample tested at 1473 K, a few dislocation loops were observed (Figure 10(d)). We did not find a deformation structure in the type-C structure in both samples tested at 1473 and 2073 K.
kinds of Burgers vectors, our observation showed that this network consisted of three type Burgers vectors were invisible (Figures 11(b) and (c)). Using \[ \frac{a}{2} [0 1] \] and \[ [0 0] \], dislocations with \( a/2 \) direction of \[ [1 1] \] (Figure 11(a)). When they were observed using TEM, the dislocation network was clearly observed at the interface in Figures 11. The dislocations with an \( a/2[1 0 1] \)-type Burgers vector were invisible in the beam direction of \([\overline{1}1\overline{1}]\) (Figure 11(a)). When they were observed using \([\overline{1}1\overline{1}] \) and \([0\overline{2}0] \), dislocations with \( a/2<1\overline{2}0 \) and \( a/2[1\overline{0}1] \)-type Burgers vectors were invisible (Figures 11(b) and (c)). Our observation showed that this network consisted of three kinds of Burgers vectors, \( i.e., a/2[1\overline{0}1], a/2<1\overline{2}0, \) and \( 2/a[1\overline{0}1] \), and would, thus, show a hexagonal configuration on the \([111]\) plane, as suggested by Lasalmonie and Strudel.\(^{[13]} \) In addition to the dislocation network, dislocations in the \( L_1_2 \) phase suggest that creep deformation occurs by shearing of \( L_1_2 \) phase in the Ir-Nb-Zr alloy.

IV. Discussion

A. Precipitate Morphology and the Lattice Misfit

It was difficult to get a homogeneous microstructure, because the solid-solution treatment cannot be performed on the tested alloys. However, after heat treatment, fine \( L_1_2 \) and fcc precipitates were formed in the primary fcc and \( L_1_2 \) phase, respectively. The lattice misfits of ternary alloys were between positive 1.3 and 1.8 pct. They are larger than those of conventional Ni-based superalloys with cuboidal precipitates, which is below 1 pct in absolute value. In a previous study, we reported that the precipitate shape depends on the lattice misfit.\(^{[4]} \) When the lattice misfit was small (around 0.3 pct), cuboidal precipitates were formed, and, for a large misfit of around 2.5 pct, platelike precipitates were formed. The present study showed that the coherent cuboidal \( L_1_2 \) phase existed in all tested alloys (type A in Figures 3(a), (c), and (f)), even though their lattice misfits were quite large, in the range from 1.3 to 1.8 pct. We also found a coherent structure consisting of the platelike fcc phase in the \( L_1_2 \) phase (type B in Figures 3(b), (d), and (g)) in the same samples. This suggests that, in the Ir-Nb-Zr alloys, the precipitate morphology did not depend on the lattice misfit alone. Instead, it was determined by which phases were matrix and precipitate, \( i.e., \) when the fcc phase precipitated in the \( L_1_2 \) phase, the precipitate shape became platelike. This may be due to the difference in the elastic constants in each phase. A maze structure could be formed if the density of the fcc phase was large in the type-B structure. Thus, we consider that the maze structure in the Ir-15Zr alloy also formed by precipitation of the platelike fcc phase in the \( L_1_2 \) phase. This is possible for the Ir-15 at. pct Zr alloy, because the primary phase of the ascast sample was the \( L_1_2 \) single phase.

B. Compressive Strength

In the type-A structure tested at 1473 K, dislocations spread in the narrow fcc channel (Figure 10(a)). When cuboidal precipitates align along the cuboidal orientation, it is difficult to form Orowan loops around individual precipitates and, thus, a mobile \( a/2[110] \)-type screw dislocation slips on one \([111] \) plane, leaving segments of mixed character. As a result, dislocation loops form.\(^{[19]} \) This is also a bypass mechanism, but not an Orowan mechanism. In our observation, cuboidal precipitates aligned along the cuboidal orientation (Figure 3). The dislocation loops in the fcc phase in Figure 10(a) suggested that deformation of the Ir-Nb-Zr alloy was not governed by an Orowan mechanism, but by another type of bypass mechanism similar to the one operating in Ni-based superalloys. The local deformation by dislocation loops is often observed during the incubation period of creep in the Ni-based superalloys.\(^{[15]} \) Note that the local deformation by dislocation loops was observed in the compression-tested sample in the Ir-Nb-Zr alloy. There is not enough time to spread dislocations for a long distance during the compression test. Thus, only small dislocation loops of 100 to 200 nm in size were observed, and deformation occurred locally. Another interesting point is that deformation also occurs in the fcc plate in the \( L_1_2 \) phase, but not in the large \( L_1_2 \) area in the type-B structure (Figure 10(b)). We observed only a few dislocation loops in the type-B structure. The fcc plates formed independently in the type-B structure and, as a result, the formation of the fcc channel was incomplete, compared to those of the long continuous fcc channel in the type-A structure. Thus, it was difficult to spread dislocation loops for long distances in the type-B structure. This result indicates that the deformation resistance is higher in the type-B structure than in the type-A structure.

The strength of the ternary alloys was higher than that of the binary alloys at 1473 K. It is interesting to note that the deformation structure of type A was similar to that of binary alloys such as the Ir-Nb alloy.\(^{[14]} \) However, the dislocation density was higher in the Ir-Nb alloy than in the Ir-Nb-Zr alloy. It is believed that the solid-solution hardening effect...
The dislocation structure of alloy A (Ir-12.5Nb-3Zr) tested at (a) and (b) 1473 K and (c) and (d) 2073 K. The zone axis and diffraction direction are (a) [101] and [020], (b) [101] and [110], (c) [001] and [002], and (d) [101] and [002].

in the fcc phase determines the deformation resistance, because the fcc phase is mainly responsible for deformation. As a consequence, the resistance to dislocation motion in the fcc phase in the Ir-Nb-Zr alloy was higher than that in the Ir-Nb alloy. In the Ir-Nb-Zr alloys, the maze structure was changed to the semicoherent structure with piled-up dislocations at the interface during the test. On the other hand, in the Ir-Nb-Zr alloy, both the type-A and type-B structures were very stable during the test. This suggests that the microstructural stability of the Ir-Nb-Zr alloy causes higher deformation resistance than that in the Ir-Zr alloy. It can be said that the existence of the type-B structure increases deformation resistance in the tested alloys, because it was not deformed.

At 2073 K, dense dislocations spread in the fcc phase in the type-A structure (Figure 10(c)), while in binary alloys, shearing into precipitates by dislocations was observed at 2073 K. In spite of the difference of the deformation structure, ternary alloys showed strengths that were almost equivalent to those of the binary alloys. This suggests that both bypass and shearing mechanisms are not effective in improving strength at 2073 K. However, it is interesting to note that we observed only a few dislocation loops in the fcc phase in the type-B structure. We consider that the type-B structure is still stable and can prevent the movement of dislocations at 2073 K.

C. Creep Behavior

Although the strength at 2073 K did not improve in the ternary alloys, the creep life improved drastically. During the creep test, the precipitate shape changed to the irregular shape with the dislocation network at the interface. The dislocation network is often observed in Ni-based superalloys during creep tests. It is suggested that the formation of dislocation networks occurs by local reactions between line segments of dislocations with a slip orientation of a/2(110). Thus, the dislocation network has a sessile structure and obstructs dislocation motion. When deformation is governed by a shearing mechanism, a mobile dislocation will trap at the interface with the dislocation network by reaction between a mobile dislocation and dislocation network. Thus, we consider that the formation of the dislocation network causes the creep resistance to be high. The dislocation network was also observed in Ir-Nb-Ni alloys which showed a superior creep property similar to that of the Ir-Nb-Zr alloy at 2073 K. We do not confirm the deformation structure yet in binary alloys crept at 2073 K, because the sample...
quickly deformed and failed. However, shearing of precipitates by dislocations was observed in the Ir-Nb alloy at 1923 K.\textsuperscript{[21]} From that observation, it can be said that the dislocation network prevents shearing of precipitates and results in the improvement of the creep property. Another important point is that discontinuous coarsening, which was observed in the Ir-Zr alloy with the lattice misfit of 2.5 pct, did not occur in the Ir-Nb-Zr alloy due to the lattice misfit below 2 pct. Microstructure stability seems to play an important role in improving the creep properties.

D. Design of High-Temperature Materials

We have tried to add third elements such as Ni, Mo, Ta, C, B, and Pt to increase the strength at high temperatures.\textsuperscript{[22]} Although Ni-, C-, and Pt-added alloys improved the strength at 1473 K, we have not succeeded in improving the strength above 1773 K. The addition of other elements causes a change in the constituent phase or a loss of interface coherence, resulting in a decrease of strength. In this study, Ir\textsubscript{3}Nb and Ir\textsubscript{3}Zr were fully miscible and only the fcc and L\textsubscript{1}\textsubscript{2} two-phase region was, thus, formed without any other phase. Furthermore, the coherent structure was also formed. From the viewpoint of the phase constitution and microstructure, the Ir-Nb-Zr is a very promising system. However, the strength at 2073 K was not improved. The results suggest that it is very difficult to improve the strength at 2073 K by a simple addition of a third element. An alternative idea is microstructure control. As shown in Figures 10, the platelike fcc precipitate in the L\textsubscript{1}\textsubscript{2} phase (type-B structure) seems to have a high resistance against deformation because of a lack of dislocations in the deformed structure. If we can obtain a large amount of the type-B structure and an L\textsubscript{1}\textsubscript{2} matrix strengthened by the solid-solution hardening effect, it may be possible to improve the strength at 2073 K. On the other hand, the addition of a third element is very effective in improving the creep-life properties at 2073 K, as shown in the case of Ir-Nb-Zr and Ir-Nb-Ni alloys. The rod-type structure with a well-developed dislocation network formed because the lattice misfit of the Ir-Nb-Zr alloy was found to be in the range of 1.3 to 1.8 pct, which is higher than that of the Ir-Nb alloy but lower than that of the Ir-Zr alloy. The present study showed that the dominant change of lattice misfit improved the creep resistance. However, a more detailed investigation is necessary.

V. CONCLUSIONS

The microstructure and strength at 1473 and 2073 K and creep behavior at 2073 K were investigated in three Ir-Nb-Zr alloys (Ir-12.5Nb-3Zr, Ir-8.5Nb-6Zr, and Ir-4.25Nb-9Zr at. pct). The lattice misfits of these alloys were between 1.3 and 1.8 pct. Two kinds of coherent structures coexisted in the alloy, i.e., cuboidal L\textsubscript{1}\textsubscript{2} precipitates in the fcc phase and platelike fcc precipitates in the L\textsubscript{1}\textsubscript{2} phase. The strength of the ternary alloys at 1473 K was higher than that of the binary Ir-Nb and Ir-Zr alloys. This is because the structure with platelike fcc precipitates in the L\textsubscript{1}\textsubscript{2} phase prevents the movement of dislocations. However, the strength of the ternary alloys at 2073 K was almost equivalent to that of the binary alloys. This is because the coherent structure cannot prevent the dislocation motion in ternary alloys as well as those in binary alloys. On the other hand, the creep property at 2073 K was drastically improved in the ternary alloy. The creep life for the 2 pct creep strain was about 100 hours, while the binary alloys deformed more than 5 pct during the first 2 hours. This enhancement has been attributed to the formation of a dislocation network at the interface, which prevents shearing of the L\textsubscript{1}\textsubscript{2} phase.

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