

Experimental investigation and thermodynamic reassessment of the Ir-rich zone in the Al–Ir system

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The homogeneity range of the B2-(AlIr) phase and the phase equilibria in the Ir-rich region of the Al–Ir system were experimentally investigated using scanning electron microscopy/energy-dispersive spectroscopy and electron probe microanalyzer. Thermodynamic assessment of the Al–Ir binary system was performed using the calculation of phase diagram approach based on the experimental data from literature and this work. A self-consistent thermodynamic description for this binary system was developed and the calculated Al–Ir phase diagram agrees with the experimental data very well.

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It is well known that improved material performance at elevated temperatures is the key to obtain higher efficiency of energy conversion from gas turbine engines. Ni-based superalloys, the most widely used high-temperature materials in gas turbine engines, are currently used at temperatures as high as 1100 °C, which is up to 90% of their melting point. It imposes a natural ceiling for further improvement [1]. The Ir-based alloys have been investigated intensively in recent years [2–6] due to the fact that their high melting temperatures and excellent chemical stability [7] have provided the designer with more flexibility in improving engine durability, fuel consumption and overall engine life-cycle costs. Among these alloys, the Al–Ir-based alloys consisting of the two-phase structure of B2-(AlIr) and face-centered cubic (fcc) Ir have been identified as candidates for potential high-temperature applications because of their high-temperature strength, high creep and oxidation resistance at elevated temperatures and high melting point. At the same time, their relatively high impact strength at room temperature [8] is another attractive property. Knowledge of phase equilibria and thermodynamic properties in the Ir-rich region of the Al–Ir binary system is of fundamental importance for better understanding the microstructure and subsequent property

enhancement of Ir-based alloys. The purpose of the present investigation was to develop a reliable thermodynamic description of the Al–Ir system in the Ir-rich region.

In the following, B2 stands for B2-(AlIr) and (Ir) for fcc-(Ir). The B2 phase in the Al–Ir binary system was first reported by Esslinger and Schubert [9], and was confirmed by Schulz et al. [10]. Lattice parameters of B2 single crystals were obtained by Axler and Roof [11] and the congruent melting point of B2 phase was reported to be about 2120 °C [12]. The phase boundaries between the B2 phase and the (Ir) phase were also investigated by Axler et al. [12], who suggested that the phase boundary of B2/B2 + (Ir) is located at ~52% Ir and that this boundary is temperature independent. All compositions are given in atomic per cent in this paper unless otherwise stated. Jiang and Gleeson [13] thermodynamically modeled the Al–Ir binary system using the calculation of phase diagram (Calphad) approach, in which the first-principles calculated energetic data are used as input parameters. However, the homogeneity range of the B2 phase was not considered in their modeling. Abe et al. [14] also developed a thermodynamic description of the Al–Ir binary system using the Calphad approach. The B2 phase was described by a two-sublattice model with the formula of $(\text{Al, Ir})_{0.5}(\text{Ir, Va})_{0.5}$ according to the experimental data of Axler et al. [12]. However, our prior study [15], which investigated the fcc-phase stability using the cluster/site approximation

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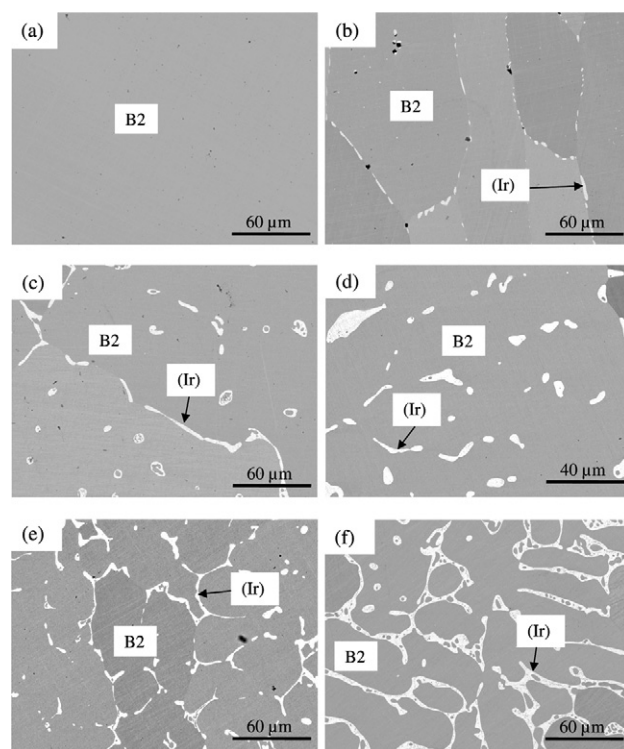
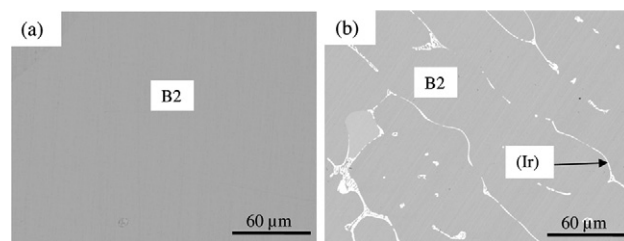
Table 1. Bulk alloy compositions and EPMA-measured compositions for phases in equilibrium

Alloy No.	Composition (at.%)	Phases (annealed)	Al (1500 °C) (at.%)	Al (1300 °C) (at.%)	Al (1130 °C) (at.%)
1	Ir–49.88Al	B2	49.88	49.93	49.83
2	Ir–49.03Al	B2	49.03	48.79	49.28
3	Ir–47.98Al	B2	–	–	–
4	Ir–47.48Al	B2	–	–	–
5	Ir–46.99Al	B2	47.78	48.10	48.99
6	Ir–46.20Al	B2	47.95	48.21	48.89
		(Ir)	9.87	7.92	5.98
		(Ir)	9.83	8.01	6.02

(CSA) method coupled with first-principles calculations, showed that the Ir contents in the B2 phase compositions in equilibrium with (Ir) is decreased with temperature and the calculated compositions of B2/B2 + (Ir) are different from the literature data, especially at temperatures below 1000 °C. In order to develop a reliable thermodynamic description of the Al–Ir system, the B2/B2 + (Ir) phase boundary needs to be accurately determined first. Experiments were thus carried out, and six typical alloys sitting in both the B2 single-phase field and the B2 + (Ir) two-phase region were selected for detail experimental investigation of phase boundary of B2/B2 + (Ir). The bulk alloy compositions are listed in Table 1, together with the equilibrium phase compositions measured by electron probe microanalyzer (EPMA).

The starting materials were commercially pure aluminum with a purity of 99.999% or higher and iridium powder purchased from Engelhard and Carr with a purity of 99.9%. The gross weight for each sample was about 30 g. The alloys were fabricated by arc-melting under high-purity argon (99.998% Ar), and with titanium as an oxygen-getter. Each button was inverted and re-melted at least five times to ensure homogeneity. These alloys were cut into small pieces (~5 g each) using a diamond wheel cutter. All samples were then wrapped in tantalum foil and subjected to annealing in an Ar-flow atmosphere. Samples were annealed at 1500 °C for 100 h, 1300 °C for 550 h and 1130 °C for 1100 h, respectively. The annealed samples were carefully grinded to get scratch-free surfaces. The microstructure of each sample was examined using a JEOL6100 scanning electron microscopy (SEM) in backscattered electron (BSE) imaging mode. The equilibrium phase compositions were determined by EPMA performed with a CAMECA SX51 electron probe. Operating conditions were 15 keV and 20 nA. On-peak counting times were 10 s, with backgrounds measured for the same time. Intensities were corrected by matrix correction using the Bastin original algorithm. Standards were pure Ir and Al–Si slug (99 wt.% Al, 1 wt.% Si).

Figure 1 shows BSE images of the six binary Al–Ir alloys annealed at 1300 °C for 550 h. As shown in Figure 1a, alloy 1 (Ir–49.88Al) is a single-phase alloy. EPMA and SEM/energy-dispersive spectroscopy analyses show

**Figure 1.** BSE images of alloys annealed at 1300 °C for 550 h. (a) Alloy 1 (Ir–49.88Al); (b) alloy 2 (Ir–49.03Al); (c) alloy 3 (Ir–47.98Al); (d) alloy 4 (Ir–47.48Al); (e) alloy 5 (Ir–46.99Al); (f) alloy 6 (Ir–46.20Al).**Figure 2.** BSE images of alloy 2 (Ir–49.03Al) annealed under different conditions: (a) 1500 °C for 100 h; (b) 1130 °C for 1100 h.

that the composition of this phase is Ir~50Al, which indicates that this phase is B2 phase. The microstructure of alloy 2 (Ir–49.03Al) as shown in Figure 1b reveals the coexistence of two distinct phases. EPMA measurements suggest that these two phases are B2 (grey phase) and (Ir) (bright phase). The majority amount of the B2 phase implies that the overall composition of this alloy locates very closely to the boundary of B2/B2 + (Ir). BSE images of alloys 3–6 annealed at 1300 °C (Fig. 1c–f) clearly show the two-phase mixture of B2 (grey phase) and (Ir) (bright phase) at this temperature. The volume fraction of the (Ir) phase increases with iridium concentrations in the bulk alloys, which is consistent with the lever rule.

Figure 2 shows the microstructures of the same alloy 2 (Ir–49.03Al) subjected to different heat treatment conditions. Figure 2a represents the microstructure obtained from alloy 2 annealed at 1500 °C for 100 h,

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