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The Effect of Al Composition on Vacancy Concentration and Hardness for B2-FeAl*

铝含量对 B2 铁铝空位浓度和硬度的影响

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Abstract:【Objective】B2-FeAl has a wide range of applications in heating elements and high temperature structural materials. The studies for mechanism of strengthening and malleablizing of B2-FeAl are of great significance for the design and development of B2-FeAl-based superalloy. **【Methods】**The vacancy concentration in B2-$Fe_{1-x}Al_x$ ($x=0.40-0.51$) has been experimentally determined as function of Al composition and quenching temperatures ranging from 773 K to 1 173 K by XRD and measurement of bulk density. The hardness of the B2-$Fe_{1-x}Al_x$ alloys has also been measured by micro-hardness tester. **【Results】**The vacancy concentration increases with the increase of Al content and annealing temperature. The micro-hardness also increases with increasing Al content and annealing temperature. **【Conclusions】**With the increase of Al content, the formation energies of vacancy decrease and the concentration of vacancy increases. With the increase of the heat treatment temperature, the concentration of vacancies in the alloy increases. The increase of the micro-hardness of the alloy is mainly due to the increased inhibition of the dislocation caused by the increase of the vacancy concentration. The quantitative relationship between the micro-hardness and vacancy concentration can be approximated as $H_V=1.922+2.179C_{V_{ac}}^{1/2}$.

Key words: intermetallics, mechanical properties, point defect

摘要:【目的】铁铝合金在高温结构材料、加热元件等方面有广泛的应用,对其强化和韧化机理的研究对设计和开发新型铁铝基高温合金具有重要意义。**【方法】**采用真空熔炼方法制备不同铝含量的铁铝合金,经过不同的退火温度热处理后,用 XRD 方法测量其晶格常数,确定其 X 射线密度,采用阿基米德法测量其体密度,从而确定其空位浓度;用显微硬度计测量其硬度。**【结果】**空位浓度随着铝含量的增加而增加,随热处理温度的升高合金中空位浓度增加;显微硬度随铝含量的增加或热处理温度的升高而增大。**【结论】**随着铝含量的增加,空位形成能下降,空位浓度增加;随着热处理温度的升高,合金中空位浓度增大;合金显微硬度增大主要是由于空位浓度增加对位错的阻碍作用增强所致。显微硬度与空位浓度的关系可近似表示为 $H_V=1.922+2.179C_{V_{ac}}^{1/2}$。

关键词:金属间化合物 力学性能 点缺陷

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0 Introduction

Iron-aluminides have attracted a lot of interests due to their high melting point, high strength, good mechanical properties, excellent oxidation, corrosion resistance and low costs[1-3]. On the other hand, iron-aluminide has also been considered for applications as heating elements owing to its high electrical resistivity[4] and magnetic materials for its outstanding magnetic performance. Most studies focus on B2-FeAl alloys.

B2-FeAl exist over a wide range of compositions, from 32 at% to 50 at% Al at room temperature and from 23.5 at% to 51 at% Al at elevated temperatures[5]. Thermal defects exist at all finite temperatures and the off-stoichiometric compositions generate the constitution defects, which are dominating the total defect concentration at ambient temperatures. The point defect structure in B2-FeAl is probably of the triple defect type[6-7] having Fe anti-site atom for Fe-rich composition, Fe vacancy for Al-rich composition, and a combination of two Fe vacancies and one Fe anti-site atom at stoichiometric composition. In addition to structural defects needed to accommodate deviations from stoichiometry, several percents of Fe antisite and Fe vacancy defects were found due to lattice disorder, with greater disorder in Fe-deficient alloys[8].

The technological application of iron aluminides is also impaired by their low tensile ductility at ambient temperatures. Many efforts have been devoted to the understanding of ductility properties in B2-FeAl, e. g. quenching-in vacancy hardening[9-11], environmental embrittlement[12] and its intrinsic brittleness[13-14]. The improvement of brittleness for Fe-Al alloys depends on a thorough understanding of their deformation mechanisms in relation to the defect structures and concentration in these materials. Experimental as well as theoretical studies[9-11,15-17] suggest that the microhardness is also in correlation with vacancy concentration. However, the values of vacancy concentration, especially the dependence of concentration on Al composition, and microhardness are scarcity in the literature.

What is the effect of Al composition on the point defect concentration and microhardness for B2-$Fe_{1-x}Al_x$? The aim of the present work is to experimentally determine the vacancy concentration in B2-FeAl alloys as functions of Al composition and annealing temperatures. The microhardness and the relationship between vacancy and microhardness have been investigated to understand the mechanism of vacancy hardening for B2-FeAl.

1 Experimental procedure

$Fe_{1-x}Al_x$ (x=0.40, 0.42, 0.45, 0.47, 0.49, 0.50 and 0.51) alloys were prepared with pure Al and Fe (99.99 wt. %) by arc melting, using a non-consumable tungsten electrode and a water-cooled copper tray under an atmosphere of purified argon (99.99 wt%). During the melting process, Ti was used as an oxygen getter. The samples were remelted more than four times for good homogenization. For all of the samples, the weight loss was less than 0.5 wt% after melting, which can be considered to be negligible. All samples were sealed in evacuated quartz tubes for heat treatment. All samples were annealed at 773 K for 168 h, 873 K for 144 h, 973 K for 120 h, 1 073 K for 96 h and 1 173 K for 72 h, respectively and finally quenched into ice-water mixture. The annealed samples were powdered for x-ray diffraction (XRD) analysis, which was performed on a Dt 3500 diffractometer with a copper target, graphite monochromator, with a voltage of 36 kV and a current of 25 mA. The mass density was measured by the hydrostatic technique. The microhardness measurements were performed by Hv-1000 with 0.98 N load and held for 15 s. The effective formation energy of vacancy was evaluated with embedded atom method (EAM)[15].

2 Results and discussion

Fig. 1 shows the lattice constants for $Fe_{1-x}Al_x$ alloys after annealing at temperature 773 K. The other experimental results were also included for comparison. One can see that the present results are coincident with those of Kogachi et al[11], but larger than those of Chang et al[10], Schneibel et al[17] and Hanc et al[18], but less than those of Zhao et al[19] and Gialanella et al[20]. The agreement between the

present results and other experimental measurement indicates that present lattice constants of $Fe_{1-x}Al_x$ alloys are reliable. Fig. 2 demonstrates the lattice constants of $Fe_{1-x}Al_x$ alloys after annealing at temperature of 773 K, 873 K, 973 K, 1 073 K and 1 173 K, respectively.

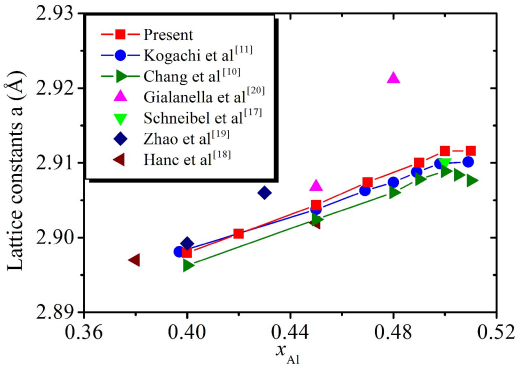

Fig. 1 The lattice constants of or $Fe_{1-x}Al_x$ alloys after annealing at temperature of 773 K

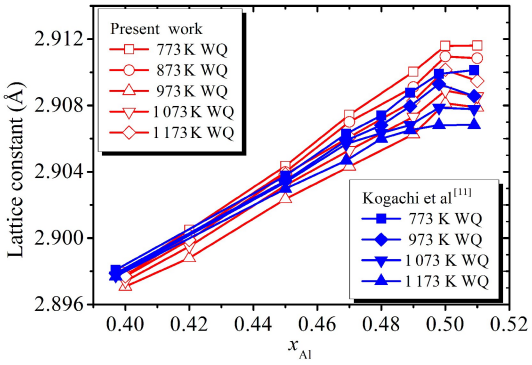

Fig. 2 The lattice constants of $Fe_{1-x}Al_x$ alloys after different annealing temperatures

The results of Kogachi et al[11] are also included for comparison. One can see that the lattice constants increase with the Al content and annealing temperature when the $x \leqslant 0.5$ and then decrease. It is obvious that the present results are in reasonable agreement with those of Kogachi et al[11].

The vacancy concentrations are determined from the room temperature measurements of the XRD density and bulk density of specimens quenched from a particular temperature as

$$C_V = \frac{\rho_x - \rho_b}{\rho_b}, \quad (1)$$

where C_V is the vacancy concentration defined as N_V/N (where N_V and N are the total numbers of vacancies and atoms, respectively), ρ_x is the so called XRD density determined from the lattice constant and the nominal composition, and ρ_b is the bulk density measured by hydrostatic method. The vacancy concentrations of $Fe_{1-x}Al_x$ alloys after annealing at different temperatures are plotted in Fig. 3. For comparison, the results of Kogachi et al[11] are also included in Fig. 3. It can be seen that the vacancy concentration increases with Al content and the annealing temperature. Though the vacancy concentration in this work is slightly larger than those of Kogachi et al[11] and Hanc et al[18], the changing tendency of the vacancy concentration versus Al content and annealing temperatures is in good agreement with that of Kogachi et al[11]. The effective vacancy formation enthalpies of $Fe_{1-x}Al_x$ alloys obtained by present experiments and EAM prediction are plotted in Fig. 4. One can see that the effective formation enthalpy of vacancy for $Fe_{1-x}Al_x$ alloys is scattered. The present experimental results are close to those of Kogachi et al[11], and larger than those of Zhao et al[19] but smaller than those of Wolff et al[21], Rivière et al[22] and Kass et al[23]. The results of EAM indicate that the formation enthalpy of Fe vacancy is less than that of Al vacancy and the magnitude of formation enthalpy decreases with Al content and changes suddenly and reversely for Al vacancy and Fe vacancy at stoichiometric composition. Both of experimental values[24] and the data evaluated with embedded atom method[15] indicate that the formation enthalpy changes on a large scale. Theoretical and experimental formation enthalpies decrease with the Al content on the whole, this results in the increase of vacancy concentration.

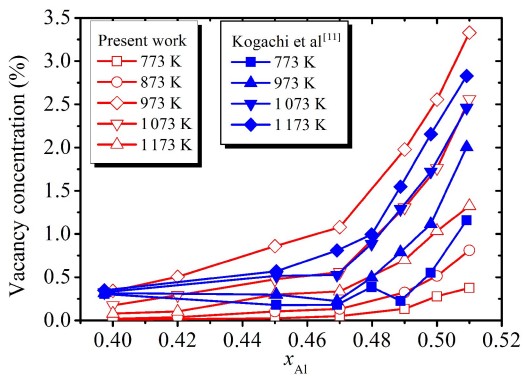

Fig. 3 The vacancy concentration of $Fe_{1-x}Al_x$ alloys after different annealing temperatures

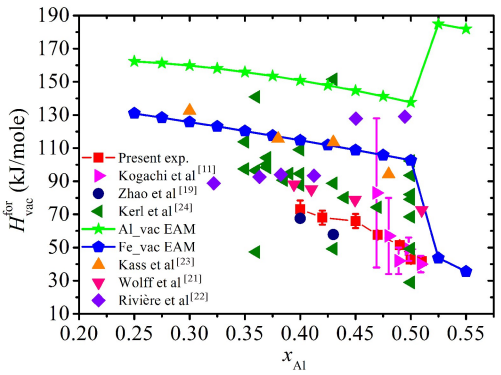

Fig. 4 The effective formation enthalpy of vacancy for $Fe_{1-x}Al_x$ alloys

In order to determine the effect of vacancy concentration on the property of $Fe_{1-x}Al_x$ alloys, the microhardness was measured and the results are displayed in Fig. 5. The microhardness increases with Al content and annealing temperature. From the figure, one can see that the present values are less than the results of Skoglund et al[25]. Their results present that the microhardness for $Fe_{68}Al_{32}$ sample annealed at different temperatures are the same. The present results are also less than those of Chang et al[10] and Gialanella et al[20]. In the whole, the changing tendency of microhardness as function of Al composition agrees with that of Chang et al[10].

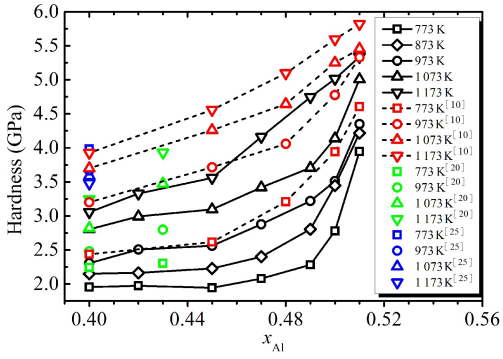

Fig. 5 The microhardness $Fe_{1-x}Al_x$ alloys as a function of Al composition from material heat treated and quenched from high temperatures

To insight the effect of vacancy concentration on microhardness or the mechanism of vacancy hardening, the microhardness was plotted as a function of square root of vacancy concentration, C_v and it is plotted in Fig. 6. It is noticeable that there is a good linear relationship between microhardness and square root of vacancy concentration, which is in good agreement with the conclusion of Chang et al[10]. One should bear in mind that there exists a large amount of Fe antisite constitutional defect as the Al composition decreases, which is independent on annealing temperature, and the hardening mechanism has a dispute[26]. However, the present results successfully confirm the relationship between the microhardness and vacancy concentration. This suggests the vacancies have a more significant effect on the microhardness than the Fe antisite-atoms. The relationship between the microhardness and vacancy concentration can be fitted with a formula as, $H_V = 1.922 + 2.179C_{Vac}^{1/2}$. This also indicates that the hardening mechanism of B2-FeAl is vacancy hardening, namely, the interaction between the vacancies and dislocations.

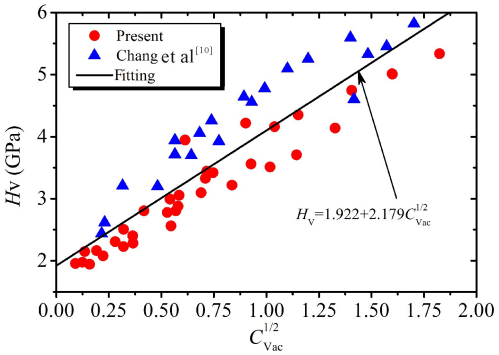

Fig. 6 Relationship between microhardness and vacancy concentration, the line is fitted to all experimental data

3 Conclusions

The quenched-in vacancy concentration in B2-$Fe_{1-x}Al_x$ has been obtained as functions of composition ($0.4 \leqslant x \leqslant 0.51$) and quenching temperature ranging from 773 K to 1 173 K by XRD and bulk density measurements. The vacancy concentration increases slowly with Al when the Al content deviated far from the stoichiometric composition and then increases rapidly as approaching the stoichiometric composition. The measured microhardness increases monotonously with vacancy concentration, which demonstrates the hardening mechanism of B2-FeAl alloys is vacancy hardening. The quantitative relationship between the microhardness and vacancy concentration can be formulated as $H_V = 1.922 + 2.179C_{ac}^{1/2}$.

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