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A positron annihilation study of two Fe-Al alloys in the B2 region

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ABSTRACT

In order to investigate the nature of defects in the B2(*h*) and B2(*l*) regions of Fe–Al phase diagram, two alloys with aluminum contents of 40 at.% and 45 at.% have been quenched from temperatures in the range 950 °C–600 °C. Positron annihilation spectroscopy and Vickers microhardness have been used to follow the changes produced by the quenching treatments. Two different behaviours could be distinguished in the evolution of the positron lifetime for both alloys. Between 950 °C and 800 °C the lifetime remains constant and it drastically falls down at 750 °C. Between 600 °C and 750 °C the lifetime monotonically increased with the quenching temperature. A dependence with the Al content was also observed; higher positron lifetimes were obtained for the alloy with a 45 at.% of Al over all the temperature range. The constant lifetime value obtained between 800 °C and 950 °C has been assigned to the presence of a triple defect formed by two Fe vacancies bound to a Fe on an Al site as main defect. The stage from 600 °C to 750 °C has been assigned to the presence of Fe vacancies and an increasing number with the quenching temperature of triple defect. The detected step between 750 °C and 800 °C is in good agreement with the solvus line between B2(*h*) and B2(*l*) regions.

1. Introduction

Fe–Al alloys with compositions on the B2 region of the phase diagram have special interest as high temperature structural materials because of their relatively high melting point, good oxidation resistance, high strength, low density and low cost. The B2 phase is based on an FeAl stoichiometry and it extends to iron-rich compositions. It is well known that physical and mechanical properties are affected by point defects and, in the case of B2 FeAl alloys, they have been object of many investigations to understand the mechanical properties for practical fabrication [1,2]. During the annealing at high temperatures, very high concentrations of vacancies easily form in the B2 structure and are largely retained by the lattice, even when the alloys are cooled to room temperature at low cooling rates [3,4]. In the most accepted phase diagram for Fe–Al system proposed by Kubaschewski [5] the B2 region is subdivided into three domains: B2′, B2(*l*) and B2(*h*). Nowadays, it is accepted that the nature of defects in the B2(*l*) and B2(*h*) regions are different [6–8]. However, this difference it is not well established. Wolff et al. [6,9] propose that a double vacancy is the defect present in the B2(*h*) domain, whereas in the low temperature region B2(*l*), a triple defect is present. Positron annihilation measurements and lifetime calculations conclude that iron vacancies are the main defects present in the B2(*l*) domain [10]. Moreover a triple defect formed by two Fe vacancies in the <100> direction bound to an Fe on an Al site is the dominant defect in the B2(*h*) phase. The aim of this work is to study the nature of defects retained after quenching in two Fe-Al alloys with compositions in the B2 region of the phase diagram.

2. Experimental

Two binary Fe–Al alloys containing Al nominal concentrations of 40 and 45 at.% were prepared at the Max-Planck-Institute für Eisenforschung (Düsseldorf). Iron and aluminium with a purity of 99.98% and 99.9995% respectively, were used. The alloys were induction melted and cast into ingots under a pressure of 40 kPa of argon. Eight sets of samples from each composition were annealed in air at temperatures between 600 and 950 °C and quenched into water at room temperature. The temperatures and annealing times were 600 °C for 5 days, 650 °C for 1 day, 700 °C for 16 hours, 750 °C for 10 hours, 800 °C for 3 hours, 850 °C for 3 hours, 900 °C for 2 hours and 950 °C for 2 hours. According to previous results reported in the bibliography [11,12] the annealing times at every temperature were long enough to be sure that only B2 order was present in the samples.

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Positron lifetime measurements have been performed by using a fast-fast system with a resolution of 215 ps. A conventional source of ^{22}NaCl evaporated onto a Kapton foil was used as positron source. A constant component of 380 ps with a intensity of 13.5% was present in all samples and was subtracted from all spectra as the source contribution. The positron lifetime spectra were satisfactorily analysed with one component by using the POSITRONFIT program [13]. The statistics of every recorded spectra was 3×10^6 counts. For the microhardness measurements the samples were loaded with 200 grams for 20 seconds.

3. Results

The positron lifetime evolution as a function of the quenching temperature for the two studied alloys is showed in Fig. 1. In all cases only one component was possible to solve from the positron lifetime spectra and each point represents the arithmetic mean of three measurements, as error bar the higher difference between the mean value and every lifetime was used. The plot indicates that the positron lifetime depends on both the thermal treatment given to the sample and on the aluminium content. Two different behaviours can be observed in the evolution of positron lifetime with the quenching temperature for both Fe40Al and Fe45Al studied alloys. Between 600 °C and 750 °C a slight increase in the lifetime can be observed; for temperatures between 800 °C and 950 °C the positron lifetime stays almost constant. The lifetime values in the alloy with an Al content of 45 at.% were always higher than the obtained for the sample with 40 at.% Al in 2 or 3 ps. According to previous results [10] the measured positron lifetimes are too high to be assigned to bulk Fe–Al structures and thus we can conclude that defects are present in the samples for all conditions. For comparison the theoretical values [10] for both a triple defect (two Fe vacancies bound to an Fe on an Al site) and an Fe vacancy in the B2 structure are also shown in Fig. 1.

In Fig. 2 the evolution of Vickers microhardness with the quenching temperature is shown. Each value is the arithmetic mean of twenty measurements. The hardness shows the same behaviour of positron lifetime, i.e. it increases between 600 and 750 °C and stays almost constant from 800 °C. From Fig. 2 it can be observed that the alloy with a higher Al content (45 at.%) is harder than the one with a 40 at.% content.

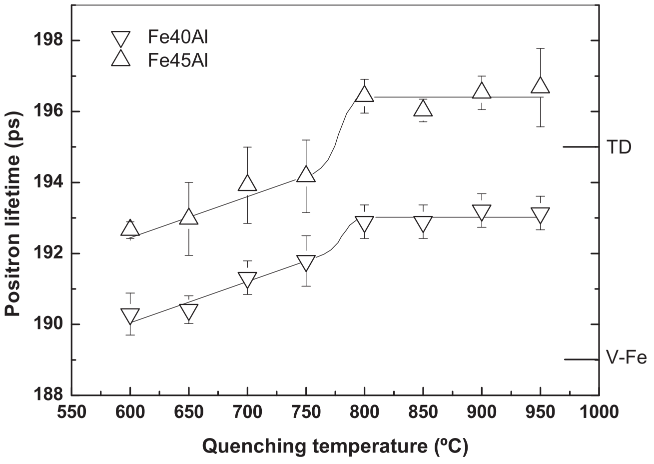

Fig. 1. Positron annihilation lifetime as a function of the quenching temperature for the Fe-40at.%Al and Fe-45at.%Al alloys. The lines have been drawn as a guide for the eye. TD and V–Fe represent the lifetimes calculated by de Diego et al. [10] for the triple defect and iron vacancy.

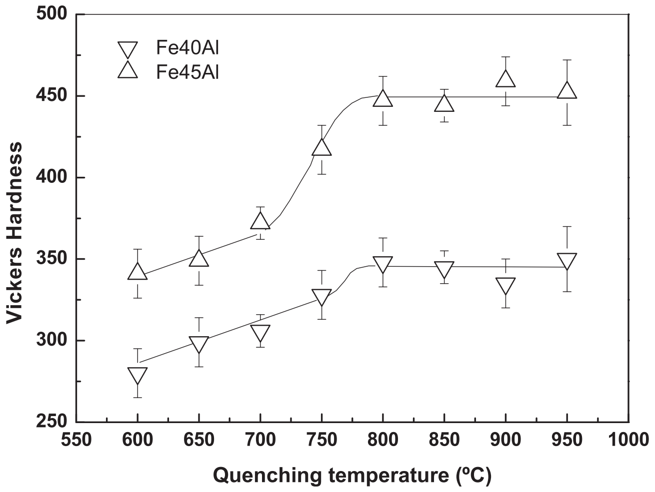

Fig. 2. Vickers hardness as a function of the quenching temperature for the Fe-40at.% Al and Fe-45at.%Al alloys. The lines have been drawn as a guide for the eye.

4. Discussion

According to the phase diagram of Fe–Al system proposed by Kubascheswski [5] the B2 region is subdivided into three domains, i.e. B2′, B2(l) and B2(h). These domains are supported by changes in physical properties such as the dilatation coefficient or the Young's modulus [14]. The work performed by Kerl et al. [8] describes changes in the vacancy concentration in these zones. The alloys studied in this work are in the B2 region of the phase diagram and the temperatures of the quenching treatments given range from the B2(h) to the B2(l) domain. As can be seen in Fig. 2 the effect of the thermal treatments is an increase in the hardness of both alloys. On the other hand the evolution of the positron lifetime (Fig. 1) indicates the presence of point defects retained during the quenching. The nature and number of these defects depend on the quenching temperature as can be deduced from the increase of positron lifetime in both alloys; moreover, the differences between positron lifetimes in both alloys for the same temperature indicate a dependence of the positron lifetime with the Al content and therefore, a different concentration of point defects in each alloy for similar quenching temperatures.

Positron lifetime has a similar behaviour with the quenching temperature in Fe-40Al and Fe-45Al alloy, increasing between 600 °C and 750 °C and staying almost constant between 800 and 950 °C. In all cases only was possible to solve one component from the spectra indicating a saturation trapping for positrons. This result indicates a very high vacancy-type defect concentration, above the detection limit of the technique ($\sim 10^{-3}$), retained during the quenching. However, the slight increase observed in the lifetime with the quenching temperature clearly demonstrates the sensitivity of the lifetime parameter to detect the defect modifications between B2(l) and B2(h).

For the Fe-45at.%Al the positron lifetime of 196 ps obtained for samples quenched from 950 °C is in good agreement with the previously reported in an Fe–Al alloy with an Al content of 48 at.% and quenched from 1000 °C [10]. This value, supported by lifetime calculations, was assigned to a triple defect formed by two Fe vacancies in the <100> direction bound to an Fe on an Al site. This configuration would be the dominant defect between 800 °C and 950 °C as it can be inferred from the constant value of the lifetime in this temperature range. Between 800 °C and 750 °C a drop to 194 ps can be observed in the lifetime value. According to the phase diagram proposed by Kubaschewski [5] these temperatures are respectively above and below the solvus line B2(h)–B2(l) for