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# X-RAY STUDY OF Al-RICH Al-Fe ALLOYS QUENCHED FROM THE MELT

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## Introduction

Al-Fe (1) phase diagram shows that the equilibrium solubility of Fe in Al is nil at room temperature and that a stable intermediate phase $Al_3Fe$ exists for about 76 at.% Al. The maximum equilibrium solid solubility of Fe in Al was found to be about 0.026 at.% at 655°C (2). Using a special quenching technique, Falkenhagen and Hofmann could obtain saturated Al-Fe solid solutions of up to 0.082 at.% Fe (3). By very rapid quenching from the melt, Bletry (4), Tonejc and Bonefačić (5) and Jones (6) have obtained supersaturated metastable Al-rich Al-Fe solid solutions of up to 5 at.%Fe. In rapidly quenched Al 4-20 wt.% Fe alloys, Jones also found a metastable $Al_6Fe$ phase which has been observed before exclusively in continuously cast Al-2 wt.% Fe (7) and in Al-1 wt % Fe solidified with a cooling rate of more than 3°C $sec^{-1}$ (8).

This paper reports on the stability of Al-Fe solid solutions and $Al_6Fe$ phase examined by X-ray diffraction.

## Experimental Procedure

Al-Fe alloys were quenched rapidly by the "two-piston" quenching technique. Details about the preparation of the alloys, the quenching procedure, and X-ray examination by means of a 114.6 mm diam Debye-Scherrer camera were reported previously (5,9). The presence of the $Al_6Fe$ or $Al_3Fe$ phase was examined by means of a Nonius Guinier-de Wolf quadruple focusing camera with crystal monochromated radiation. All the annealing treatments were carried out in a furnace with a nitrogen atmosphere. The desired temperatures were taken to lie within ±5°C for temperatures of up to 300°C, and within ±10°C for those up to 550°C.

## Results and Discussion

About 50 samples of Al-Fe alloys, containing from 1.4 to 10 at.% Fe, rapidly quenched using the "two-piston" quenching method, were examined. The results are summarized in Table I.

TABLE I

Results of X-ray Investigation of Al-Fe Alloys Quenched Rapidly from the Melt

| Sample Thickness, µm | At.% Fe | $Al_6Fe$ | $Al_3Fe$ | Al-Fe Solid Solution |
|---|---|---|---|---|
| 1 - 15 | up to 4.4 | - | - | ⊕ |
| 15 - 40 | up to 4.4 | + | - | - |
| | | + | - | ⊛ |
| 1 - 50 | 4.4 - 10.o | - | + | - |
| | | - | + | ⊛ |
| | | + | + | - |
| | | + | + | ⊛ |

+ $Al_6Fe$ or $Al_3Fe$ phase present

⊕ Al-Fe solid solution

- Absent

⊛ Partial Al-Fe solid solution

For most quenched alloys up to 4.4 at.% Fe, solid solutions were obtained in flakes not thicker than 15 µm. The $Al_6Fe$ phase was detected in thicker flakes. Partial solid solutions were also present.

When alloys with higher concentrations than 4.4 at.% Fe were quenched, both the $Al_6Fe$ and the $Al_3Fe$ phase were observed in most cases. There were also samples with $Al_3Fe$ phase and partial solid solutions but no traces of the $Al_6Fe$ phase.

The lattice parameters of Al-rich Al-Fe solid solutions decrease with increasing iron content (5). In order to examine the stability of metastable Al-Fe solid solutions, a solid solution with the 3.6 at.% Fe was chosen for isothermal and isochronal annealing.

Isochronal annealing of the Al-3.6 at.% Fe solid solution for ten minutes at temperatures up to 550°C showed (Fig.1) that the solid solution decomposed between 200°C and 350°C with a maximum increase in the lattice parameter between 250°C and 300°C. The $Al_6Fe$ pattern became detectable at 300°C. At 450°C the $Al_3Fe$ pattern became evident, while at 500°C the lines of the $Al_6Fe$ phase were disappearing. At 550°C only the equilibrium terminal phases Al and $Al_3Fe$ were present.

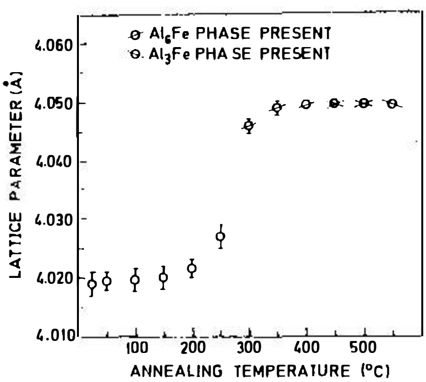


FIG. 1

The variation of lattice parameter of Al-Fe solid solution with annealing temperature for Al-3.6 at.% Fe after annealing for ten minutes at each temperature.

Isothermal annealing of the Al-3.6 at.% Fe solid solutions at 200°, 250° and 300°C showed (Fig.2) that the solid solutions are very stable at 200° and less at 250°, while at 300°C they decompose rapidly to Al and to metastable $Al_6Fe$ phase (in our experiment after 350 minutes).

Samples of Al-3.6 at.% Fe which contained mixtures of Al and $Al_6Fe$ were isothermally annealed at 300°, 400° and 550°C. The $Al_6Fe$ phase remained stable for 76 hours at 300°C but for 30 minutes only at 400°C. It gradually changed to the equilibrium phase $Al_3Fe$ within the next 15 hours at 400°C, and within about 10 minutes at 550°C. However, at 300°C the process was not yet completed even after 670 hours.

## Conclusions

Although very stable at temperatures up to 200°C, Al-Fe solid solutions decompose rapidly in Al and $Al_6Fe$ phase at 300°C. $Al_6Fe$ phase, stable at temperatures up to 300°C, decomposes and passes rapidly to $Al_3Fe$ at 400°C.

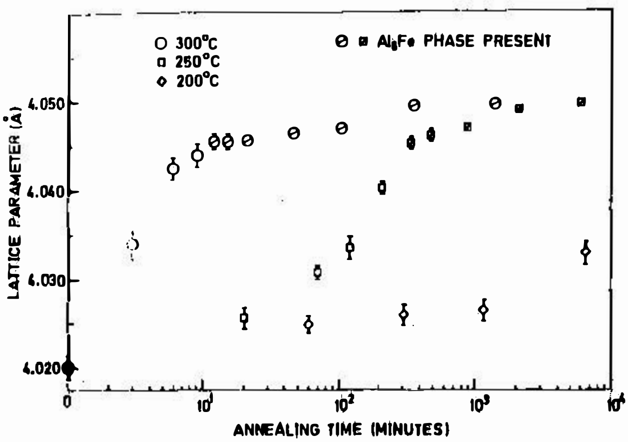


FIG. 2

The variation of lattice parameters of Al-3.6 at.% Fe solid solutions with annealing time (annealing temperatures: 200° , 250° and 300°C).

Acknowledgements

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DISCUSSION :

E. Blank : How did you state, that your flakes are solid solutions and that no precipitates like $Al_6Fe$ are within ? $Al_6Fe$ could precipitate during solidification eventually and cause the Debye-Scherrer diffraction pattern observed.

A. Tonejc : From the fact that within the experimental errors lattice parameters of solid solutions fall on a curve, and from absence of any evidence for another (i.e. $Al_6Fe$) on Debye-Scherrer patterns we conclude that our flakes are solid solutions. However, very small quantities of $Al_6Fe$, if they exist, can not be detected on X-ray patterns.

N.J. Grant : If the lattice parameter versus composition plot remains a straight line extrapolation of the equilibrium data, one must assume that there is probably no excess phase present. Transmissionelectron microscopy can be used to confirm the presence or absence of suspected second phase particles. Our data agree well with the maximum solubility data reported in this work for splat foils of 1-10 $\mu$m thickness at cooling rates estimated to be $10^8$C/s, our value was $\gg$ 4 at% Fe.

Do you have activation data for the decomposition process ?

A. Tonejc : The data for activation energies for the decomposition process will be given by Babić et al.

G.E.A. Bartsch : Do you think that there is formation sequence: $Al$-$Al_6Fe$-$Al_3Fe$, in other words that the sites of $Al_6Fe$ are the sites of nucleation for $Al_3Fe$ ?

M. Paić : You cannot see by X-ray techniques if the $Al_6Fe$ phase is a nucleus for the $Al_3Fe$ phase. But I think that thermodynamically one may suppose that the $Al_6Fe$ phase transforms into the $Al_3Fe$ phase .

G.E. Bartsch : Did you see any preprecipitation effects ?

A. Tonejc : D. Kunstelj and A. Bonefačić (Metallography 3, (1970) 79 ) made some metallographic investigation of isochronal annealed Al-0.68 at% Fe solid solution and they found that in the first stage of annealing (up to 150°C) coarse cellular structure develops with globular precipitates along the cell walls.

A. Guinier : Do you observe the $Al_6Fe$ diffraction pattern without any anomaly (position or intensity of the lines) ?

A. Tonejc : No, we did not, but because we used monochromated copper radiation, the fluorescence was very high, so if there exist some anomalies in the first stages of annealing, we could not detect them.

A. Dixmier : When annealing the sample did you see intermediate shift of the X-ray aluminium solid solution lines, and during the first stage of annealing did you see immediatelly the $Al_6Fe$ diffraction lines ?

A. Tonejc : No inmediate shift of solid solution lines was observed. $Al_6Fe$ diffraction lines were detected after the (333) line of Al appeared beside the already existing (333) $\alpha$-Al line, naturally now less intensively.