A STUDY OF MICROSTRUCTURE AND PHASE TRANSFORMATIONS IN AN ANNEALED, RAPIDLY QUENCHED, Al - 8 wt.%Fe ALLOY

M. H. Jacobs, A. G. Doggett and M. J. Stowell
Tube Investments Research Laboratories, Hinxton Hall,
Saffron Walden, Essex, England.

Introduction

This paper is concerned with the metastable structures formed in flakes of an Al - 8 wt.%Fe alloy (splats) which were solidified rapidly by the 'gun' splatting technique (1). Transmission electron microscopy (TEM) was used to study the microstructure of the splats in the "as-quenched" condition and also to follow the decomposition of this microstructure after subsequent isothermal annealing treatments at elevated temperatures. In addition to their intrinsic interest, these annealing studies were aimed at providing a fundamental microstructural background to the work of Thursfield et al (2) on the elevated temperature mechanical properties of compacted and extruded bulk material.

Our investigation was an extension of work carried out by Jones (3) in which he found that a transition in structure was revealed in Al - 8 wt.%Fe splats if a polished section, normal to a splat surface, was etched in Keller's reagent. The layers adjacent to each surface of the splat, which were characterised by only a slight response to etching, he designated as Zone A and the strongly etched central layer he designated as Zone B. In addition, he showed that the hardness of Zone A (~260 kg/mm^2) was significantly higher than that of Zone B (~100 kg/mm^2). We have carried out a detailed TEM investigation of these microstructures and our results are listed below as a precursor to our annealing studies.

Experimental Procedure

The gun splatting apparatus and the Al-Fe stock material were the same as used by Jones (3) in the earlier work. The splats were solidified on a grit blasted copper substrate at

room temperature. All the annealing treatments were carried out in an air furnace, controlled to within ± 5°C of the desired temperature. 3mm discs for TEM were prepared by the standard method and the resulting thin foils were examined in an AEI EM6G electron microscope operating at 100 KV.

Microstructure of Splats in the "As-Quenched" Condition

1. The characteristics of Zone A were as follows:-
(a) The structure consisted of grains of α-Al, ~1μm diameter in the plane of the splat, containing a fine-scale, second phase, network structure (in agreement with Jones (3)). Each α-Al grain was, usually, highly misoriented with respect to its neighbours.

(b) The cell diameters of the second phase network varied over the range ~10nm up to ~100nm, sometimes between regions only a few microns apart. Fig. 1 is an example of a Zone A network structure which contained cells ~50nm in diameter.

(c) The characteristic electron diffraction pattern from Zone A consisted of a ring pattern (usually four or more rings could be distinguished) which originated from the network phase, superimposed upon a spot pattern from the α-Al matrix (a typical pattern is shown inset in Fig. 1). From an analysis of several ring patterns it was concluded that the network consisted of a large number of small, randomly oriented, crystallites of a b.c.c. phase which was close in lattice parameter to the intermetallic compound FeAl.

2. The characteristics of Zone B were as follows:-
(a) In agreement with Jones (3), Zone B was found to consist of grains of α-Al which contained a second phase network of spacing ~400nm. In the plane of the splat the α-Al grains were ≥4μm in diameter.

(b) It was shown, by electron diffraction and dark field microscopy, that the network consisted predominantly of the equilibrium $FeAl_3$ phase which was single crystal over extensive areas (a few very small areas of $FeAl_6$ network were also observed).

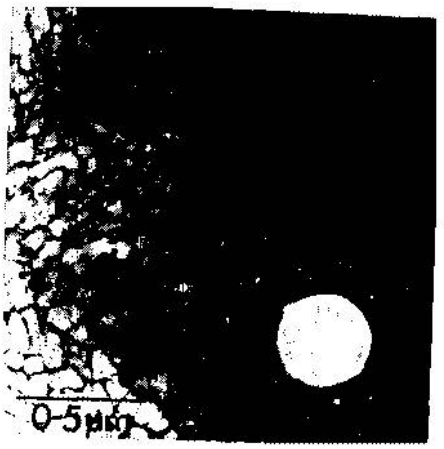

FIG. 1
The network structure of Zone A. The diffraction pattern from this area is shown inset.

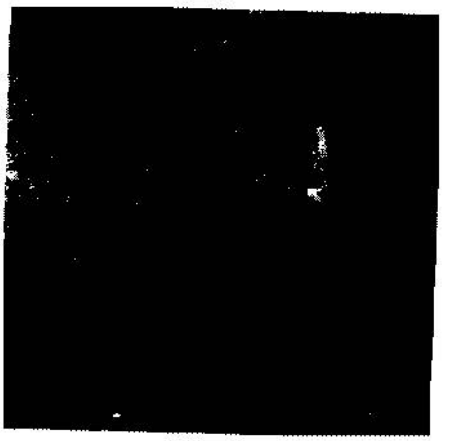

FIG. 2
Zone A annealed for 3½ hours at 300°C, showing particles of $FeAl_6$ at the α-Al grain boundaries.

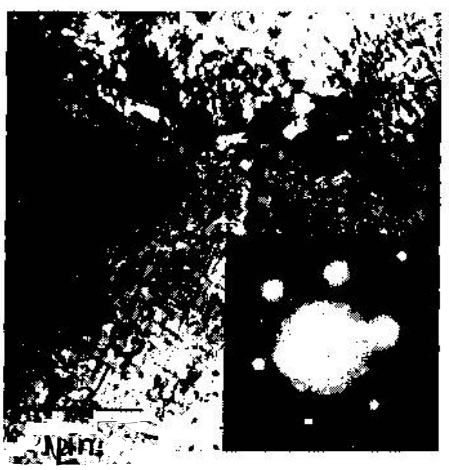

FIG. 3
Zone A annealed for 2 hours at 380°C. The inset diffraction pattern was obtained from the grain G.

FIG. 4
Zone A annealed for 4 hours at 380°C. The diffraction pattern from this area is shown inset.

Microstructural Changes During Annealing

Jones (3) studied the annealing behaviour of Al - 8%Fe splats after annealing for one hour at temperatures up to 600°C by means of subsequent microhardness measurements at room temperature. He found that the hardness of Zone A decreased considerably on annealing above 300°C, whereas Zone B underwent only a relatively small softening. The object of our work was to use TEM to study the microstructural changes associated with this annealing behaviour.

The only microstructural change detected in Zone A material annealed for up to 3½ hours at 300°C was the slow growth of particles at the α-Al grain boundaries, as illustrated in Fig. 2. These particles were identified, by selected area diffraction, as the metastable $FeAl_6$ phase (4).

More significant changes occurred when Zone A was annealed at 380°C. After annealing for 30 minutes the network had decomposed in some areas into needle-shaped particles, whereas after 2 hours it had decomposed throughout into needles (see Fig. 3). This transformation was associated with the gradual disappearance of the ring pattern, characteristic of the network structure, and this was replaced by a streaked diffraction pattern (an example is shown inset in Fig. 3) which indicated that the needles possessed an orientation relationship with the α-Al matrix and also that they were coherent with the matrix along their length. More information about the crystal structure of the needles and their orientation relationship with the matrix was obtained from a sample of Zone A, annealed for 4 hours at 380°C, in which the needles had grown sufficiently in size to give rise to a well defined spot diffraction pattern. An example of this type of pattern, obtained from an α-Al grain in cube orientation, is shown inset in Fig. 4. A careful TEM examination of this sample showed that (a) the needles were oriented with their lengths parallel to cube directions of the matrix, as demonstrated in Fig. 4, (b) the needles were of the equilibrium, monoclinic, $FeAl_3$ phase (5), and (c) that there were two equivalent orientations of needle for needles along each matrix

cube direction, e.g. for the needles along [100] α-Al these were

$$(1)\ \left\{\begin{matrix}(010)\text{ needle // (100) }\alpha\text{-Al}\\ [100]\text{ needle // [010] }\alpha\text{-Al}\end{matrix}\right\}$$

$$\text{and } (2)\ \left\{\begin{matrix}(010)\text{ needle // (100) }\alpha\text{-Al}\\ [100]\text{ needle // [001] }\alpha\text{-Al}\end{matrix}\right\}$$

The transformation from a network of FeAl to needle-shaped $FeAl_3$ particles occurred more rapidly during annealing at 500°C and after 15 minutes the needles had coarsened into large cuboidal-shaped particles. In a sample annealed for 15 minutes at 550°C the trend towards spheroidisation of the $FeAl_3$ particles was even more marked.

No changes in the network structure of Zone B were detected in samples annealed for up to 3½ hours at 300°C. However, a gradual process of spheroidisation occurred in Zone B regions annealed at 380°C, leading to isolated particles of $FeAl_3$ in an α-Al matrix.

Conclusions

Our results showed that the decomposition of Zone A during annealing at elevated temperatures (>300°C) was as follows:-

(1) particles of the metastable $FeAl_6$ phase were formed at the α-Al grain boundaries;

(2) the fine scale network of FeAl crystallites decomposed to form needle-shaped $FeAl_3$ particles, which subsequently coarsened into spheroidal-shaped particles.

Zone B was found to decompose during annealing as follows:- Coarse network of $FeAl_3$ in an α-Al matrix → spheroids of $FeAl_3$ in an α-Al matrix.

Finally, we also carried out a less extensive study of splatted material produced by (a) the centrifugal disintegration technique, and (b) the gas atomisation technique (see Thursfield et al (2)). Our results showed that material produced by these methods had as-quenched microstructures similar to those described above for single splats prepared by the gun technique and that they also underwent similar microstructural changes during annealing.

Acknowledgements

The authors are grateful to Mrs. K. M. Whitmore for assistance with specimen preparation. This paper is published by permission of the Chairman of Tube Investments Limited.

References

1) P. Duwez and R. H. Willens, Trans.AIME., 227, 362 (1963).
2) G. Thursfield, H. Jones, M. H. Burden, R. C. Dibling and M. J. Stowell (This Conference).
3) H. Jones, Mater.Sci.Eng., 5, 1 (1969/70).
4) L. K. Walford, Acta Cryst., 18, 287 (1965).
5) P. J. Black, Acta Cryst., 8, 43 and 175 (1955).

DISCUSSION :

A. Guinier: What happens to the first phase Fe Al during annealing ? Is it not surprising that the first precipitate is more iron rich than the following ones ?

M.H. Jacobs: The FeAl phase decomposes and is replaced by the needles of $FeAl_3$.

Yes, it is surprising that the first phase is more rich in iron. One possible explanation is that the formation of the equilibrium $FeAl_3$ phase is inhibited because the value of its unit cell (1.488 μm^3) is large and the small dimensions of the inter-cellular regions of zone A impose a constraint on its formation. The volume of the FeAl unit-cell (0.024 μm^3) is much smaller and it may be easier, energetically, for the metastable phase to solidify under the highly non-equilibrium conditions of splat quenching.

H. Warlimont: Regarding the identification of the "FeAl" phase: it is based only on diffraction angles of very few lines and tentative assignment of one line to obtain a lattice parameter of 2.9 Å . Thus the structure and the composition associated with it may require some closer study. But even if the determination is correct, the fact that a solute content as high as 50 at% is suggested for the metastable precipitate from its structure is not basically surprising: solute rich (up to near 100%) transitional precipitates are well known in several systems (e.g., Cu-Al, Fe-Mo). But since the phase FeAl extends to approximately 75 at% Fe on the iron rich side in stable equilibrium, a metastable extension to a higher Al content under the present conditions is also conceivable.

G.W. Lorimer : The unit cell is specified by crystallographers for their clasification and convenience. The phase that is being nucleated does not know that it must produce a simple or complex unit cell and thus a complex unit cell is not a deterrent to nucleation.

M.H. Jacobs : I think nucleation of a large unit cell may be inhibited if the available volume is of the same order of magnitude as the unit cell.

J. Dixmier: In the as quenched sample is there a shift of the diffraction lines corresponding to the percentage of iron in the sample ? (Vegard law). In other words have you in zone A a complete solid solution of 8 wt% of iron in Al ?

M.H. Jacobs: There is a shift in the α -Al X-ray diffraction lines of Zone A, but not-from zone B. The shift for zone A indicates enhanced solubility of iron but probably not a complete solid solution of 8 wt% of iron in Al, since a large proportion of the iron is believed to be present in the second phase network.

R. Roberge: How do you explain the change from the initial FeAl to the metastable $FeAl_6$ and finally to the equilibrium $FeAl_3$?

M.H. Jacobs: During annealing, the growth of particles of $FeAl_6$ is observed at the α -Aluminium grain boundaries, but not within the α -Aluminium grains. The transformation within the grains is FeAl $\rightarrow$ $FeAl_3$, that is, the highly metastable as-quenched structure decomposes progressively towards the equilibrium microstructure.

M. Paić: May be that, as it happens generally, the less thermodynamically stable phase, forms first.

M.H. Jacobs: Yes, I agree.

D. Kunstelj : Zone A- α (Al) solid solution (shift of the lines) + AlFe (?) at the cell boundaries. It is quite a complicated situation ?

M.H. Jacobs : Yes.