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ATOM PROBE STUDY OF A Ti-10V-2Fe-3Al ALLOY : PRELIMINARY RESULTS

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Résumé : Nous avons étudié à la sonde atomique l'évolution de la phase ω qui apparaît, après trempe depuis le domaine β (ou $\alpha + \beta$), dans un alliage Ti-10V-2Fe-3Al. Cette étude est effectuée pour des durées de revenu à 300°C allant jusqu'à l'apparition de la phase α intragranulaire.

Les premiers résultats montrent que les particules de la phase ω isotherme se caractérisent par un enrichissement en titane et un appauvrissement pour tous les autres éléments.

Abstract : We studied the evolution of the ω phase which appears after quenching from the β (or $\alpha + \beta$) field in a Ti-10V-2Fe-3Al alloy. Studies are performed for different aging times at 300°C up to the growth of α phase secondary precipitates.

Preliminary results reveal that isothermal ω phase precipitates are enriched in titanium and depleted in all additional elements.

I - INTRODUCTION

Because of their relatively low density and high mechanical strength titanium base alloys are of great interest in aerospace applications. The Ti-10wt%V-2wt%Fe-3wt%Al alloy is an industrial alloy mainly used for making structural components of aircraft.

After quenching from the β field, the alloy is composed of two phases : the BCC β matrix and precipitates of the hexagonal athermal ω phase whose composition is the same as the matrix. During aging at 300°C precipitates of the hexagonal isothermal ω phase appear either by transformation of athermal ω phase or directly. After a few days at 300°C small needle shaped α phase precipitates are visible by TEM [1].

The purpose of the atom probe study is to determine the composition as well as the size and the volume fraction of the isothermal ω phase and to follow the evolution of this phase from the quenched state to the growth of the α phase.

In this paper we will report some preliminary results for aging times of 1h and 8h at 300°C.

II - EXPERIMENTAL

The alloys were supplied by the Office National d'Etudes et de Recherches Aéronautiques (ONERA). The atom probe studies are connected with TEM observations, hardness measurements and mechanical tests performed at ONERA by M. H. CAMPAGNAC [2]. The alloys were solution treated 2h at 780°C in the $\alpha + \beta$ field then quenched. They are composed of large β phase grains ($\sim 10\mu\text{m}$) and of α phase primary precipitates which form at grain boundaries.

The field ion microscopy specimens were prepared by anodic electropolishing in a solution of 6 % perchloric acid 34 % n-butyl alcohol and 60 % methanol cooled at -40°C. In order to prevent passivation phenomena a circulation of the solution is produced by means of a peristaltic pump.

The atom probe used in this study is a conventional one. The instrument details and performances and the acquisition system are described elsewhere [3][4]. Reliable data were obtained by using a pulse fraction of 20 % of the DC voltage and a tip temperature between 40 K and 80 K. Analyses were carried out in the range 10^{-10} to 10^{-8} Torr.

III - RESULTS AND DISCUSSION

As shown in Fig.1 all the elements Al, Ti, V, Fe are observed as doubly charged ions and are clearly separated in mass spectra.

Primary phases α and β

The phase composition as given by atom probe analyses in the two primary phases α and β are compared with microprobe analyses in tables 1 and 2. The results obtained by both techniques are close together except for Al.

	Ti	V	Al	Fe
Atom probe	87.6	3.2	8.9	0.60
(2 σ)	0.6	0.3	0.5	0.08
Microprobe	88.16	3.5	8.15	0.19

Table 1 : Composition of Primary α phase obtained by means of atom probe and microprobe analyses.

	Ti	V	Al	Fe
Atom Probe	82.3	10.6	4.9	2.2
(2 σ)	0.3	0.2	0.15	0.1
Microprobe	82.2	10.45	5.51	1.84

Table 2 : Composition of β phase obtained by means of atom probe and microprobe analyses

ω phase

Atom probe analyses of alloys heat treated 1h at 300°C revealed the presence of titanium enriched zones, as shown in Fig.2 a. These zones are interpreted as ω phase precipitates. Matrix β and ω phase compositions are given in table 3, the error bars are taken as two standard deviations.

	Ti	V	Al	Fe
Matrix (2 σ)	79.8 0.7	12.16 0.32	5.72 0.22	2.35 0.15
ω Phase (2 σ)	92.5 0.6	4.6 0.5	2.01 0.32	0.91 0.22

Table 3 : Compositions of ω phase and matrix β for aging time of 1h at 300°C

The atomic fraction F_a deduced from compositions is $F_a = 20\% \pm 5\%$ which gives a volume fraction $F_v = 21\% \pm 5\%$ taking in account the difference between atomic volumes for the two phases.

The mean precipitate size ϕ can be derived from the mean apparent length $\bar{\ell}$ of particles crossed by the analysis cylinder whose diameter is ϕ_a by using [5] :

$$\phi = \frac{3}{2} \bar{\ell} \cdot \frac{(1+\eta)^2}{F(\eta)}$$

with $\eta = \phi_a / \phi$ and $F(\eta) = 1 + 3\pi\eta/4 + 3\eta^2/2$

This method gives $\phi = 145 \text{ \AA}$ with $\bar{\ell} = 100 \text{ \AA}$. This result agrees with the values directly observed in composition profiles for precipitates nicely crossed (as in Fig.2). Of course the use of the simple expression between $\bar{\ell}$ and ϕ is not actually correct as particles are not spherical but cuboidal as shown by TEM observations [1].

After aging 8h at 300°C the precipitated fraction increases as shown in Fig.2 b. Matrix and ω phase compositions for this aging time are given in table 4.

	Ti	V	Al	Fe
Matrix (2 σ)	74.7 0.7	15.8 0.6	7.2 0.4	2.26 0.22
ω phase (2 σ)	93.1 0.5	4.55 0.38	1.61 0.22	0.73 0.15

Table 4 : Composition of ω phase and matrix β for aging time of 8h at 300°C

The atomic fraction F_a deduces from composition is $F_a = 37.5\% \pm 5\%$ which gives $F_v = 39\% \pm 5\%$. The mean particles size is $\phi = 170 \text{ \AA}$ although particles the size of which are larger than 200 $\text{\AA}$ are frequently observed in composition profiles.

IV - CONCLUSION

We analysed alloys aged 1h and 8h at 300°C. The ω phase appears to be a titanium rich phase depleted in all additional elements. The volume fraction increases from $21\% \pm 5\%$ for an aging time of 1 hour to $39\% \pm 5\%$ for 8 hours. These preliminary experiments will be extended for longer aging times up to the growth of α phase secondary precipitates.

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Fig.1 - Atom probe mass spectrum of a Ti-10V-2Fe-3Al alloy

(a)

(b)

Fig.2 - Composition profiles of a Ti-10V-2Fe-3Al alloy
780°C 2h W.Q. and: (a) 300°C 1h, (b) 300°C 8h.