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ATOM-PROBE STUDY OF G.P. ZONES IN Ti-Cu ALLOYS

L. HADJADJ, A. MENAND and D. BLAVETTE

Laboratoire de Microscopie Ionique, UA 808 CNRS, UER Sciences de Rouen, BP 67, F-76130 Mont-Saint-Aignan, France

Résumé - Nous avons étudié les zones G.P. dans un alliage Ti-2.3at% Cu à l'aide d'une sonde atomique. Les analyses ont révélé une structure multicouches de ces zones avec une alternance possible de plans riches en cuivre et de plans riches en titane. La composition moyenne de ces plaquettes est comprise entre 20% et 40% .

Abstract - G.P. zones in a Ti-2.3at% Cu alloy were investigated on an atomic scale by means of atom probe. Analyses revealed a multi-layer structure with a possible alternation of copper rich planes with titanium rich planes. The copper average concentration of the platelets was found to be in a range of 20% to 40%

I - INTRODUCTION

In spite of some difficulties in tip preparation, and although tip failures are very common under the field stress, A.P. studies of high content titanium base alloys are now performable /1/.

The peculiarity of titanium-copper alloys is that age-hardening is available by precipitation of copper enriched zones in the α phase /2/. Better mechanical properties than those obtained with pure titanium are therefore available with such materials /3/.

The purpose of this study is to get more informations about the structure and the composition of G.P. zones in titanium-copper alloys.

II - EXPERIMENTAL

The alloys Ti-3wt%Cu were supplied by the Office National d'Études et de Recherches Aéropatiales (O.N.E.R.A.). They were prepared by using arc-melting technique. They were solution treated 1 hour at 800°C, 1 hour air cooled and then aged 28 days at 400°C. This treatment leads to obviously visible precipitates forming coherent platelets on (1011) planes of the alpha matrix /4/ Figure 1.

The field ion microscopy specimens were prepared by anodic electropolishing in a solution of 6% perchloric acid 34% n-butyl alcohol and 60% methanol. In order to avoid passivation phenomena it has been necessary to product a circulation of the solution obtained by means of a peristaltic pump. An electropolishing cell has been specially designed to that effect.

The atom probe used in this study is a conventional one. The instrument details and performances are described elsewhere /5/. Reliable data were obtained by using a pulse fraction of 20% of the DC voltage, an a tip temperature lying between 40 K and

80 K. Analyses were carried out in a vacuum of 10^{-9} Torr. FIM observations were performed at 20 K. The figure 2 shows a FIM micrograph of a Ti-3wt%Cu specimen obtained with neon as imaging gas. A low index pole is clearly visible. The titanium is observed in the mass spectra as doubly charged ions while the copper is detected as doubly and for a smaller part as singly charged ions.

III - RESULTS AND DISCUSSION

Composition

The composition averaged over all the analyses is $1.40 \pm 0.05\text{at\%}$ of copper. This result is in good agreement with the solubility limit of copper in the alpha phase at the eutectoid point given in the literature as 1.6at% /6/, 1.25at% /7/ or 1.4at% /8/. The excess of copper in Ti-3wt% Cu i.e Ti-2.3at% Cu leads to a grain boundary precipitation of a Ti_2Cu phase /3/. Because this phase is not homogeneously dispersed we only encountered two precipitates of this kind during our investigations.

Depth of analysis

One major problem in A.P. data analysis is the determination of the depth of analysis. This is of particular significance when the studied objects are small platelets of unknown thickness.

When the ring counting technique or even layer by layer analysis are not usable (poor image quality ...), the following general relevant expression of the analysis depth p can be used /8/ :

$$p = \left(\frac{L^2}{S_D \theta} \right) \cdot \left(\frac{(E\beta)^2 \cdot V_{at}}{(m+1)^2} \right) \cdot \left(\frac{1}{\gamma(m,\theta) \cos(\theta+\Phi)} \right) \cdot \frac{N}{V_m^2}$$

where N is the number of detected ions and V_m the average evaporation voltage.

The first term of this expression depends upon the apparatus itself (detector efficiency θ , flying path L , detector area S_D).

The second factor is related to the material parameters (atomic volume V_{at} , evaporation field E and geometrical factor β , projection point m).

The third term depends upon the site of analysis (angle θ between the tip axis and the normal to the investigated pole (hkl), angle Φ between the tip axis and the detector direction).

Therefore two possibilities exist to get a practical formula :

$$p = k \cdot N / V_m^2$$

The first is to perform a layer by layer analysis on a known pole located near the tip axis where $\gamma(m,\theta) \cos(\theta+\Phi) = 1$ and to deduce k from the number of detected atoms per plane.

The second is to estimate k from the results obtained with an other material (nickel in our case) where this k parameter is known in the same atom probe configuration :

$$k_{Ti} = k_{Ni} \cdot \frac{(E\beta)_{Ti}^2 \cdot V_{atTi}}{(E\beta)_{Ni}^2 \cdot V_{atNi}}$$

The first method supposes the possibility to index the different poles. In fact this is not often the case with titanium because of the poor contrast of FIM images. However it is possible to make some assumptions between four or five low index poles and to compare the results for k to the one obtained by the second method.

Fortunately we succeed to traverse G.P. zones during layer by layer analyses, this allows to clearly recognize these planes as the habit planes (1011). Therefore this gives us a more reliable determination of k . Finally we found $k = 17 \pm 1.5 \text{ \AA kV}^2$ while the result obtained by the second method was $16.5 \pm 2.5 \text{ \AA kV}^2$.

Analysis of platelets sideways crossed

A typical profile is given in figure 3. It surely indicates that a G.P. zone has been crossed but is not very informative as regards the real structure and composition of the platelet. However some indications can be drawn from such a profile.

If p is the crossing length through the G.P. zone (figure 4), α the angle between the platelet and the analysis cylinder axis, $n.d(hkl)$ the thickness of the platelet and D the diameter of the analysis cylinder, one can write :

$$\text{tg } \alpha = D / (p - n.d/\sin\alpha)$$

which leadsto :

$$\sin \alpha = (n.d.p + D.(p^2 + D^2 - n^2 . d^2)^{1/2}) / (p^2 + D^2)$$

it is then possible to calculate each α value corresponding to the various values of n .

Afterwards the possible compositions of the G.P. zone can be derived. Since the elliptic surface of the platelet intercepted by the analysis cylinder is : $S = D^2 / (4 . \sin\alpha)$ the expected number of collected atoms for a platelet whose thickness is $n.d$ is :

$$N_p = Q.D^2.n.d / 4.V_{at}.\sin\alpha$$

with Q the detector efficiency.

Then the platelet composition is :

$$C_p = N_{Cu} / N_p$$

where N_{Cu} is the observed number of copper atoms.

Obviously since N_p depends on the α angle, it exists a couple $C_p(n)$, $\alpha(n)$ for each value of n .

The last information being used is the maximum concentration observed C_c in the central part of the profile. This concentration can also be derived from n and α :

$$C_c(n) = C_p(n) . \sum_{i=1}^n l_i / \sum_{i=1}^{n_T} l_i$$

where l_i are the intercepted length of (hkl) planes (figure 4), n the number of (hkl) planes in the platelet, n_T the number of (hkl) planes intercepted by the analysis area.

Therefore a comparison between the calculated $C_c(n)$ and the observed value C_0 can be done.

The table 1 gives the results for α , C_p and C_c for the zone of figure 3.

Table 1

n	α°	c_p	c_c
1	46	1.10	0.25
2	51	0.60	0.30
3	56	0.43	0.38
4	62	0.34	0.34
5	67	0.28	0.28

These results indicate that the observed profile could have been obtained either with a platelet of one pure copper layer crossed at 46° by the analysis cylinder (obviously 1.1 has to be taken as 1), or with platelets of 2, 3, 4 or 5 layers with decreasing concentrations traversed with the indicated angles.

But the observed composition at the central part of the zone :

$$C_0 = 16 \text{ Cu atoms} / 43 \text{ Cu+Ti atoms} = 37\%$$

indicates that a 3 or 4 layers structure seems to be more likely.

Figure 5 gives a different profile for a platelet crossed more perpendicularly. The slope of this profile indicates that the platelet is not composed of a single plane but at least of 3 or 4 planes and suggests an alterned structure Cu rich plane - Ti rich plane(s) - Cu rich plane.

The different profiles we observed were all similar to one or the other previous ones.

Layer by layer analysis

Since the random analysis of G.P. zones seems to indicate a complex structure of 3 or 4 layers, it was therefore interesting to attempt a layer by layer analysis as Hashizume and Sakurai did /9/ for Al-Cu. We tried this on different poles with the hope that one of them will be the (1011) habit plane.

Figures 6a and 6b show the copper cumulative plots obtained for two different layer by layer analyses. The flux curves vertically drawn allow to mark the different layers.

The first platelet (figure 6a) has a thickness of four layers with a similar alterned structure as suggested previously : one copper rich layer, two titanium rich layers, one copper rich layer. Of course we have to take in account the possible statistical deviations because of the very small number of collected atoms in such analyses.

The second profile obtained with a somewhat larger detection area exhibits a similar multi-layer structure with four layers. The average composition is found to be $28\% \pm 10\%$ of copper.

IV - CONCLUSION

We performed analyses on a Ti-3wt%Cu alloy containing copper enriched G.P. zones. The value of 1.4 at% of copper obtained over all the experiments in good agreement with the solubility limit of copper in the alpha matrix gives us confidence about the data reliability.

The G.P. zones randomly crossed or during layer by layer analysis are multi-layer platelets with a possible number of 3 to 5 layers. Some results suggest an alterned structure, copper rich -titanium rich layers. The average platelet compositions are ranged from 20% to 40% .

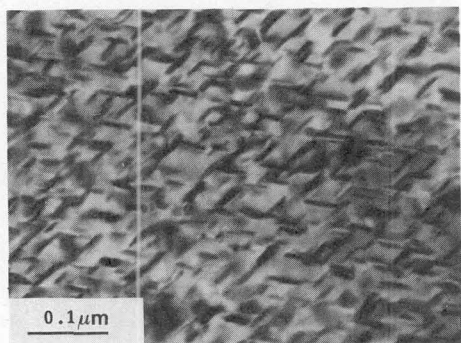


Fig 1 : Bright Field Electron micrograph of a Ti 3Cu alloy aged 28 days at 400°C (from VASSEL /4/)

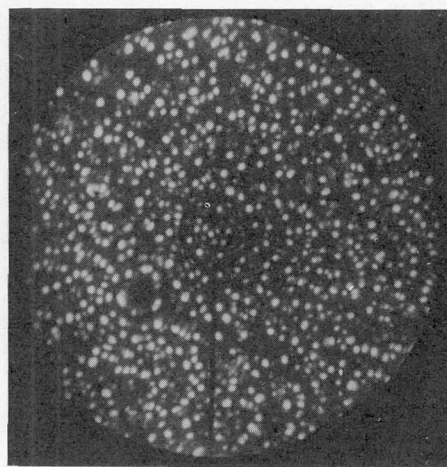


Fig 2 : FIM Micrograph of a Ti 3Cu Specimen

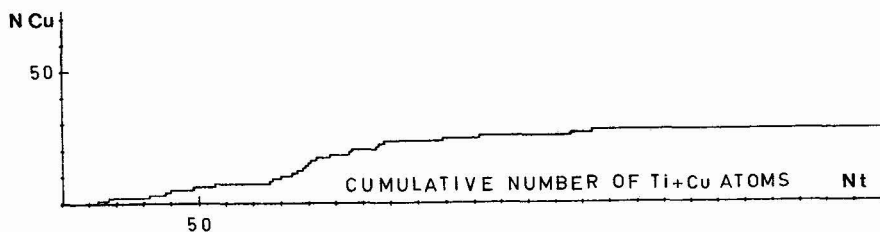


Fig 3 : Integral profile of Cu showing the crossing of a G.P. zone

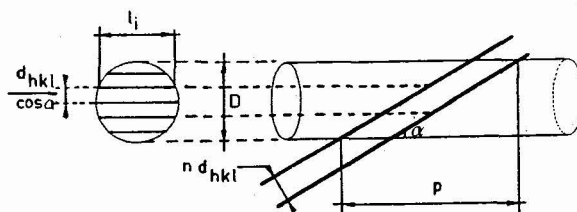


Fig 4 : Schematic of the crossing of a platelet by the analysis cylinder

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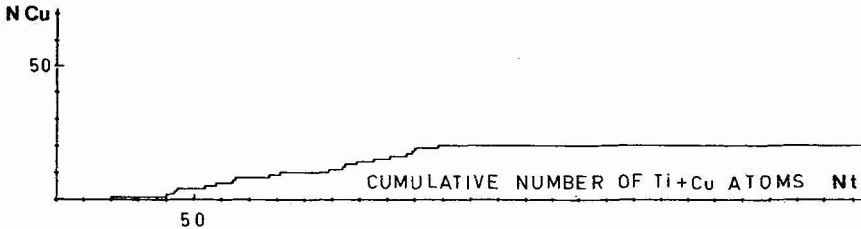


Fig 5 : Integral profile of Cu showing the crossing of a G.P. zone

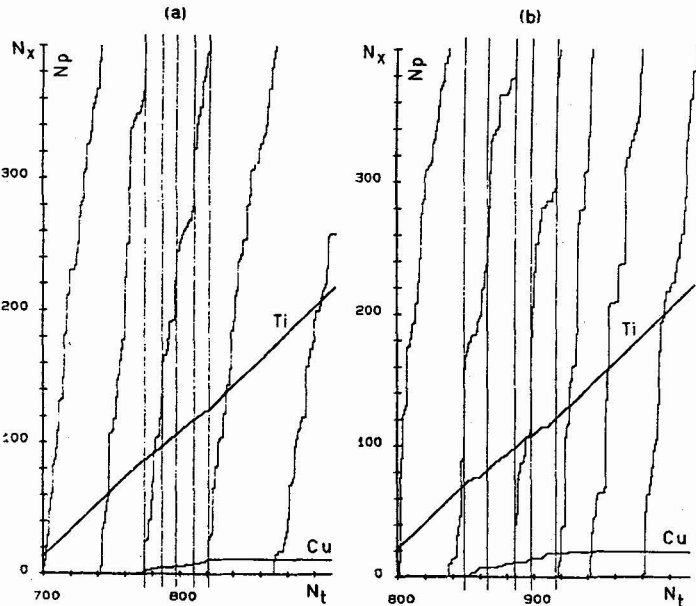


Fig 6 a and b
Integral profiles
of Cu and Ti in
layer by layer
analysis of two
G.P. zones, with
the corresponding
flux curves N_{pulse}
versus N_t (Ti+Cu).