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Decomposition of β-metastable phase in β-Cez alloy during continuous heating

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Abstract

The decomposition of the metastable β phase in β Cez alloy has been studied during continuous heating up to 830°C. The heating rate varied between 0.25 and 5°C/s. Electric resistivity measurements, and hardness measurements revealed different phase transformations as temperature increased. Different samples were quenched from different temperatures during the heating range and were then studied by X-ray and TEM, allowing to correlate the changes in resistivity measurements to the beginning of the precipitations of the ω and α phases and to establish the continuous heating transformation diagram. A change in the resistivity variations, not dependent on the heating rate was observed near 700°C. X-ray quantitative analysis and EAM studies revealed that the structure consisted of a maximal content of α phase, surrounded by β rich zones in the matrix. For temperatures greater than 700°C, the content of α phase diminished and the β phase was homogenized.

Introduction

The β metastable decomposition during continuous heating is of considerable importance since it determines the morphology and distribution of the α precipitates during ageing treatments in the α + β domains.

For the pseudo β titanium alloy, the optimal microstructure is obtained by a sequence of thermomechanical and thermal treatments. Also the final microstructure is highly dependent on the various parameters of these treatments.

Also we investigated the effects of the cooling rate from the β range solution treatment to room temperature on the further phase transformations, during a further solution treatment in the α + β temperature range.

In this paper we focus on the phase transformations occuring during heating, the initial structure being a β metastable phase obtained by quenching after a solution treatment in the β phase. The effect of the cooling rate from the β solution treatment on the microstructure is presented elsewhere (1).

Experimental methods

The alloy used in this study was a "pseudo-beta" alloy named β-Cez which was produced by Cezus. The nominal composition in weight percent of the as-received material was Ti-5Al-2Sn-4Zr-4Mo-2Cr-1Fe and the dimensions of the cylindrical samples were D =6.0 mm and L = 25.0 mm;

Specimens were solution treated in secondary vacuum at 920° for 30 min. and were then water-quenched. The specimens were then reheated up to 830°C at different rates, while changes in electrical resistivity were recorded. Moreover, specimens were helium quenched from different temperatures in the heating range in order to study the phases in presence by X-ray and TEM. All X-ray bulk samples were cutted and then mechanically polished. A cobalt anticathod (Co - K α_1; λ = 1.7889 Å) was used to obtain the X-ray diffraction patterns. Thin foils were prepared by Blackburn-Williams (2) solution with the Bainbridge-Thorne (3) Jet polish technique , for investigations by TEM and AEM.

Hardness measurements were made too on the bulk samples, using the Hv30 standard measurements test.

Results

a - Electrical resistivity

The electrical resistivity variation curves were recorded versus time and temperature. Slope changes were observed at different temperatures which depended on the heating rate as shown figure 1. Four domains can be considered of which limit temperatures versus heating rate are given in table 1. A slope change not dependent on the heating rate, was observed near 700°C.

Domains	Heating rates Temperature	0.25°C/s	0.50°C/s	1.25°C/s	5.0°C/s
1	20°C up to T_1	300°C	300°C	350°C	450°C
2	T_1 up to T_2 =	350°C	350°C	400°C	550°C
3	T_2 up to T_3 =	700°C	700°C	700°C	700°C
4	T_3 up to T_4 =	830°C	830°C	830°C	830°C

Table 1 - Limit temperatures of the domains for different heating rates

b - Hardness measurements

The variations of hardness of the samples obtained after quenching from the different temperatures are shown figure 2 for a rate of 0.5°C/s. No significant increase in hardness is observed in the first domain ; an increase in hardness is noticed in the second and third domains. At quench temperatures greater than 700°C, the hardness falled down, and was nearly constant after 60 min at 830°C. A maximal hardness value was observed near 700°C, for all heating rates used.

c - Microstructure

X-ray measurement of the initial β water quenched phase revealed that the structure consisted of beta metastable phase with anisothermal ω phase. Electron diffraction patterns taken from the beta-metastable structure in the first domain revealed weak and diffusional streaks, which later could be observed too and identified by X-ray as being the athermal omega phase. This phase is produced by rapid quenching after solution treatment and vanishes at the

end of this domain. It was difficult to discern the shapes in that scattering dark shades. On the morphological point of view it looks like the G.P. zones found in the Al-Zn-Mg systems (4-7).

Specimens quenched from a temperature in the second domain revealed changes in contrast when observing the matrix. EDAX analysis showed that the transformed matrix has a light heterogeneousness of the composition and so we called it as beta transient (β_T). Electron diffraction patterns taken from β_T revealed peaks of the isothermal omega phase (fig. 3). However, that phase which was always associated with β_T could not be observed in dark field condictions. Lattice parameters of the hexagonal ω athermal or isothermal phases have been found as a = 4.53 Å and c =2.78 Å. The Silcock orientation relationships $<111>_\beta$ //$<0001>_\omega$ and $\{110\}_\beta$ //$\{11\bar{2}0\}_\omega$ were obtained. The isothermal ω phase vanished on the initial stages of the third domain during continuous heating, while alpha phase was beginning to precipitate.

Selected area diffraction patterns taken from regions around alpha precipitates in the third domain showed splitting and diffuse spots. The only observable feature of the electron diffraction pattern, apart from the diffuse streaking, was the splitting of the matrix spots.

X-Ray diffractograms and TEM electron diffraction patterns showed that the structure consisted of the hc α phase and the bcc β phase ; EDAX analysis revealed that the composition of the α precipitates is roughly constant, however that of the β phase is enriched in Fe, Cr and Mo around the α phase.

The variations in chemical composition of the zones around the α phase lead to a decrease in the "d spacing" resulting to the splitting of the matrix spots. This zones are called rich beta phase (βr). It was observed that the nucleation of the alpha phase within a grain occurs near the interfaces between the βr and the matrix which we thought to be ex-omega. This results is in agreement with Narayanan and Archbold (8), Campagnac and Vassel (9).

In the fourth domain, we observed a reduction of the α lamellae density followed by its coalescence. While the α phase content diminished, at temperatures greater than 700°C the composition of the β rich zones and the matrix was homogenized to form an equilibrium beta phase. The Bürgers orientation relationships $<111>_\beta$//$<11\bar{2}0>_\alpha$ and $\{110\}_\beta$//$\{0001\}_\alpha$ were obtained when the temperature reached about 830°C (Fig. 4).

d - X-Ray

X-ray diffraction patterns were obtained for the as quenched samples to determine the structure parameters in the present phases and to estimate the content of the α phase. The ratio $I\alpha / (I\alpha + I\beta)$ (fig. 5) shows an indication on the α phase content vatiations versus the temperature for a heating rate of 0.5°C/s; where $I\alpha$ and $I\beta$ are the sum of pics intensities of the α and β phases respectively.

The ratio $I\alpha / (I\alpha + I\beta)$ increases up to 700°C, indicating a maximal content of alpha phase. For temperatures higher than 700°C this ratio falls down to establish near 830°C.

Discussion

Changes in electrical resistivity as in hardness measurements observed during continuous heating revealed the occurence of different phase transformations. The T.E.M. and X.R. studies allowed to define that the decomposition of the metastable beta phase of the beta Cez alloy during continuous heating consisted of a sequence of successive phase transformations :

$$\beta_m + \omega_{aniso} \rightarrow \beta_m \rightarrow \beta_t + \omega_{iso} \rightarrow \alpha + \beta r + \beta \rightarrow \alpha + \beta e$$

were β_m , β_t , β_r , β and β_e refer to the metastable, transient, enriched, matrix and equilibrium β phases respectively.

At temperatures below T_2 [350 to 550°C, depending on the heating rate], the different observations indicate that the decomposition of the metastable β phase into ω phase could proceed through a diffusional clustering mechanism. Such a result has been pointed out by Narayanan and Archbold on Ti-13V-11Cr-3Al previously (8).

At temperatures above T_2, the precipitation occurs. The chemical composition of the β phase is heterogeneous, β zones enriched in Cr, Fe and Mo surrounded the α lamellae. A maximal content of α phase not dependent on the heating rate is obtained near 700°C as shown by X-ray intensity ratio variations as by a maximum in the hardness measurements.

Selected area diffraction patterns and EDAX analysis suggest that the precipitation could occur through composition fluctuations in the matrix leading to the formation of solute rich zones which subsequently drive to a maximal content of the alpha phase.

Therefore, it is reasonable to believe that the metastable β phase decomposition mechanism is dependent on the matrix composition fluctuation, during continuous heating . The fluctuations can be related to a diffusional vacancy mechanism as suggested by different authors (4-8).

Acknowledgments

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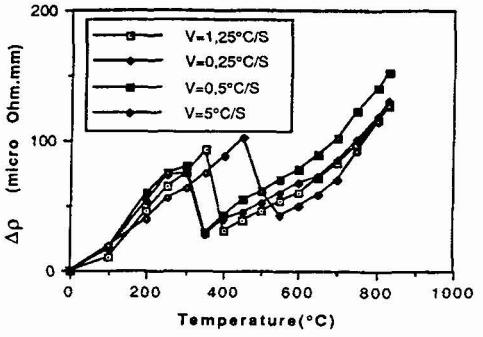


Fig. 1 - Electrical resistivity variations versus temperature during heating up to 830°C for different heating rates

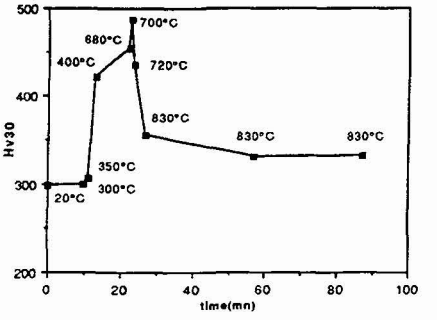
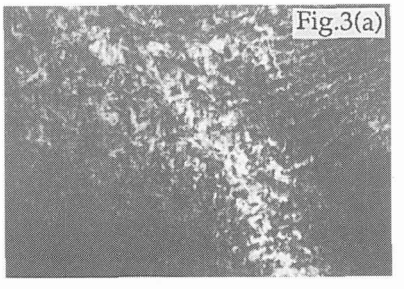


Fig. 2 - Hardness measurement variations for specimens cooled at different times (or temperatures) during heating (heating rate 0.5°C).



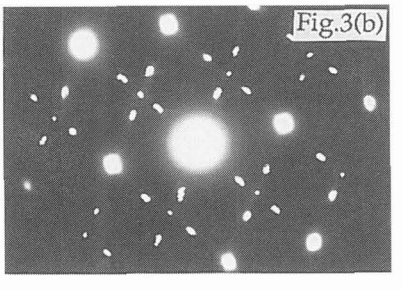
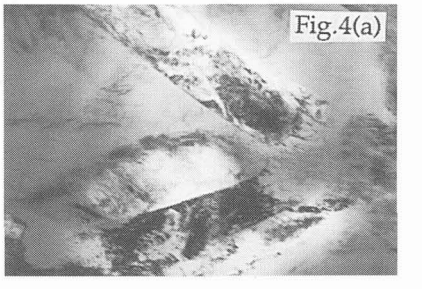


Fig. 3 - (a) Dark field image taken from the second domain showing a transformed beta transient matrix (βt) T=350°C. (b) The ω phase was observed in SAD pattern, although this phase was not observed in dark field image. Magnification 100000X.



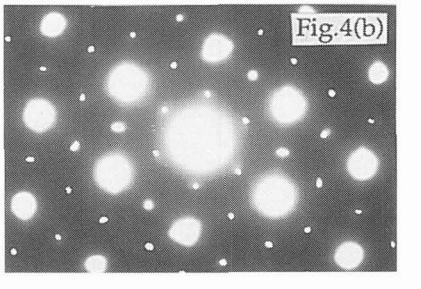


Fig. 4 - (a) Bright field image taken from the fourth domain T=830°C. A reduction of the α lamellae density was observed. (b) SAD pattern showing the Bürgers orientation relationships. Mag. 73000X.

Fig. 5 - Ratio of X-ray intensities between α phase and the total α+β phases.