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Precipitation mechanisms and subsequent hardening kinetics in the β-CEZ alloy

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The ß-CEZ alloy : Ti-5%Al-2%Sn-4%Zr-4%Mo-2%Cr-1%Fe is a near ß titanium alloy. Since its processing routes include heat treatment (consisting of solution treatment and ageing), it has been decided to study the influence of the ß phase stability on α phase precipitation and hardening kinetics. Small sized specimens, coming from industrial heats, have been heat treated above and below the ß transus, and then water quenched. Therefore, several degree of ß stability are obtained. When it is increased (i.e. the solution treatment temperature is lowered), α or ω phases precipitations are delayed, as well as the hardening phenomena. Moreover, the ω stability domain is reduced. The mechanisms of α or ω precipitation are discussed, and related to the hardening kinetics.

Introduction

As a metastable ß titanium alloy, the ß-CEZ alloy (Ti-5%Al-2%Sn-4%Zr-4%Mo-2%Cr-1%Fe) may be subjected to solution and ageing treatments. Significant changes in the nature, morphology and size of the precipitates can be therefore encountered which may lead to a large choice of mechanical properties. These microstructural changes are depending on the stability of the ß phase. Minimum stability is given by 100 % ß microstructures and their response to heat treatments have been well documented [1,2]. However, very few is available for more stable structures. The objectives of this paper are to characterize the effect of ß phase stability on precipitation mechanisms and subsequent hardening after heat treatment.

Material and experimental procedure

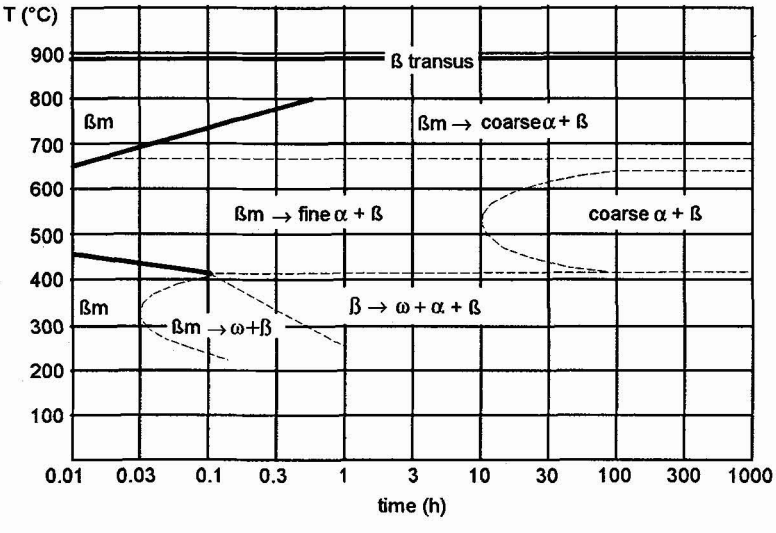
Specimens have been cut in industrial forgings. 100 % ß metastable microstructures have been produced heating at 920 °C (i.e. above the ß transus of the alloy : 890 °C). These specimens have been cooled and held to the heat treatment temperature encompassing the 250-800 °C range. A higher degree of ß stability has been obtained using a heat treatment at 830 (or 850) °C followed by water quenching to retain the ß phase. These specimens have been heated to the desired temperature in the 200-800 °C range at low heating rates (1 °C/mn). Light metallography, SEM and TEM have been used to characterize the microstructures.

For the 100 % ß metastable specimens, dilatometry has been performed to obtain the precipitation kinetics. 30 kgf Vickers hardness measurements have been run to get the hardness evolutions.

Microstructural evolutions

ß heat treated specimens (920 °C)

This study has been priorly performed [1,2], and has been used, for long time, to design the first heat treatment windows. The relevant Temperature-Transformation-Time diagram is presented in figure 1.



-Figure 1-

The diagram exhibits probably a nose of precipitation between 500 and 600 °C and for durations lower than 36 s. The temperature range can be roughly divided into three domains. For temperatures between 700 °C and the ß transus, metastable ß, ßm, transforms into coarse α. Between 400 and 700 °C, for durations t lower than 10 h, fine α phase precipitation occurs first, and for t > 10 h, the fine precipitates coarsen. For temperatures below 400 °C, ßm transforms into ω phase which can be more or less rapidly reverted into α phase. However, after 1 h, the ω+ß field no longer exists.

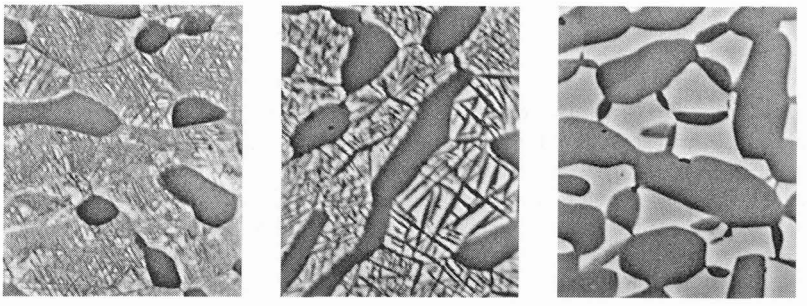
Therefore, for a fully ß microstructure, the material may be solution treated above 700 °C (typically 750 °C is chosen industrially) and aged between 500 and 650°C to avoid the ω phase domain.

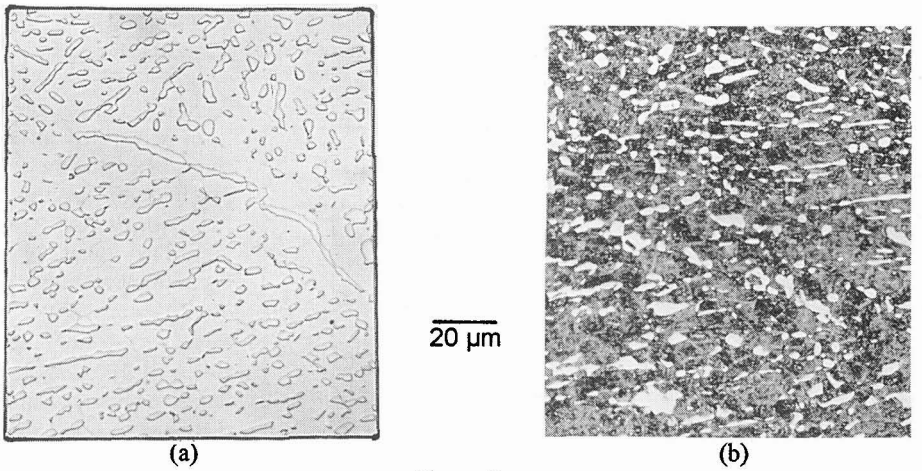
α+ß heat treated specimens (830 °C)

For temperatures above 550 °C and below 700 °C, α phase precipitation occurs and the morphology is fine and acicular. For longer durations, this phase coarsens as shown in figure 2. For temperatures higher than 750 °C, relatively coarse α precipitation takes place, and so, all the more rapidly than the ageing temperature is larger (figure 3).

Below 400 °C, ω phase has been detected between 200 and 300 °C ; within this temperature range, it appears rapidly (after 2 h duration at 250 °C for instance, the incubation time being longer at 200 °C : 100 h). Below 275 °C, even after 1000 h, ω phase precipitation is relatively dense (figure 4 (a)-(b)). Just below 300 °C, fine α particles precipitate from ω, and both phases co-exist (figure 4 (c)). For durations longer than 100 h, all the ω phase has been transformed into α. Above 300 °C, α precipitates out directly from the matrix defects such as grain boundaries (figure 4 (d)).

-Figure 2-
SEM micrographs
(a) T = 650 °C/t = 3 mn
(b) T = 650 °C/t = 5 h
(a) T = 650 °C/t = 50 h

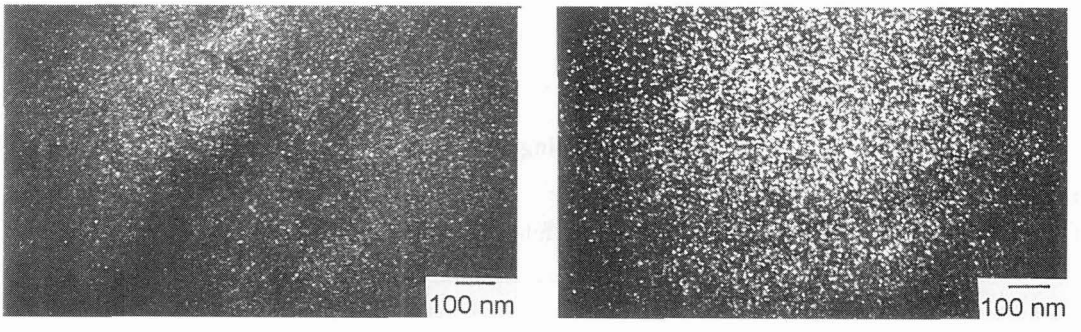




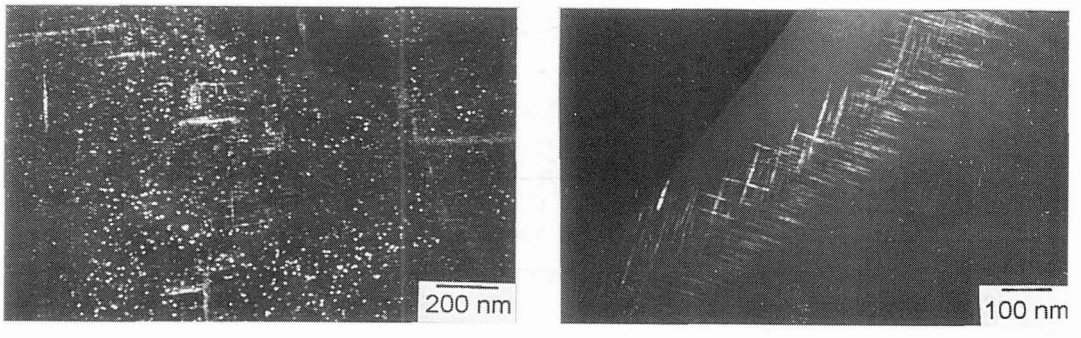
(a) (b)

-Figure 3-

Light metallography : (a) T = 550 °C /t = 3 mn ; (b) T = 650 °C/t = 3 mn



(a) (b)

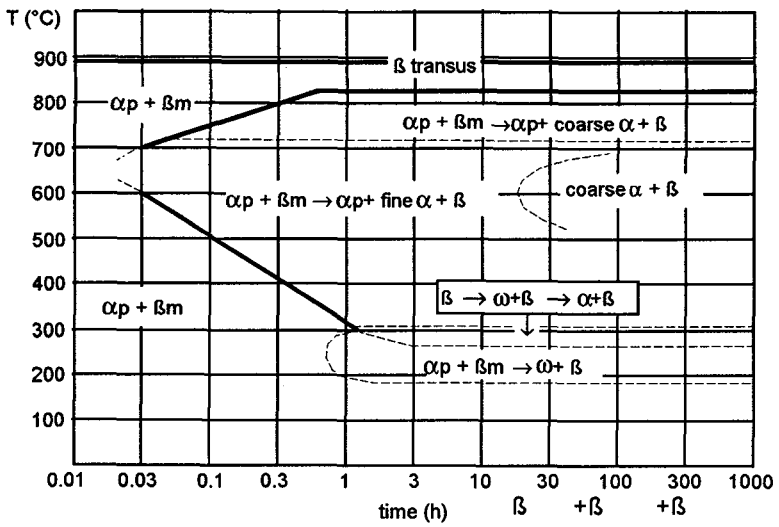


(c) (d)

-Figure 4-

TEM drak field micrographs : (a) T = 200 °C /t = 100 h ; (b) T = 250 °C/t = 500 h
(c) T = 300 °C /t = 8 h ; (d) T = 365 °C/t = 1 h

These microstructural observations allow to draw the Temperature-Transformation-Time diagram from specimens solution treated in the α+ß field, where αp denotes the primary α phase present during solution treatment at 830 °C.



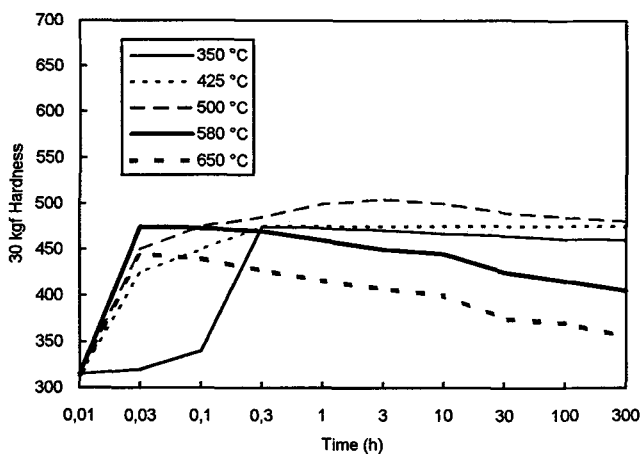
-Figure 5-

Although no experiments have been performed to confirm it, there is probably a nose of precipitation in the 600-700 °C temperature range for durations lower than 2 mn. For temperatures between 700 and 830 °C, ßm transforms into coarse α. Between 300 and 700 °C, for durations lower than 20 h, fine α phase precipitation occurs first, and for longer time, the precipitates coarsen. For temperatures below 300 °C, ßm transforms into ω phase which can be more or less rapidly transformed into α phase. However, below 275 °C, the ω+ß field is stable.

Hardening kinetics

ß heat treated specimens (920 °C)

The figure 6 illustrates the hardening kinetics of the fully ß microstructures [2].



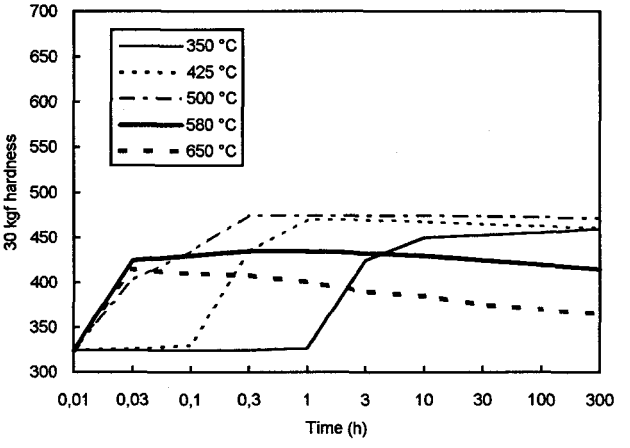
-Figure 6-

Influence of the ageing temperature on the hardness of specimens solution treated at 920 °C

Whatever the temperature in the 350-650 °C range, when the holding time increases, the material hardens abruptly first, then, the hardness reaches a maximum and gradually decreases. The amplitude of hardening is about 200 HV, which confirms the heat treatment response of ß-CEZ alloy. Best compromise of hardness is obtained at 500 °C after 3 h. At low temperatures, the increase in hardness is slightly delayed (6 mn incubation time for instance at 350 °C)

α+ß heat treated specimens (850 °C)

Figure 7 shows the kinetics on specimens solution treated in the α+ß range (850 °C) [2].



-Figure 7-

Influence of the ageing temperature on the hardness of specimens solution treated at 850 °C

Whatever the temperature in the 350-650 °C range, the hardness increases with time first, then, it reaches a maximum and smoothly decreases. The amplitude of hardening is important (about 175 HV), which confirms that the heat treatment response of ß-CEZ alloy is not altered by the 850 °C treatment. Here again, best compromise of hardness is obtained at 500 °C after 20 mn. At low temperatures, the increase in hardness is delayed (1 h incubation time for instance at 350 °C, and 6 mn for 425 °C)

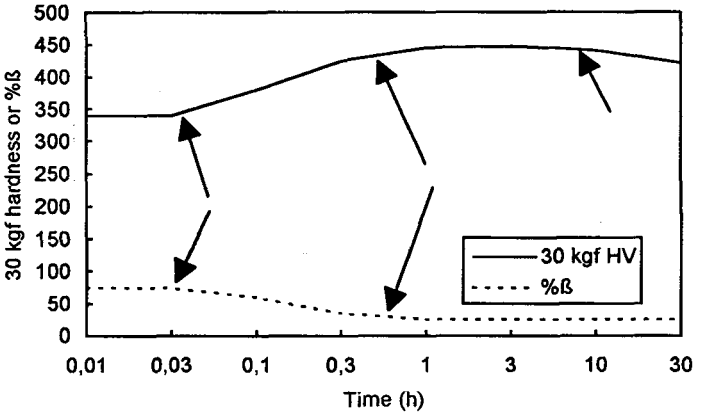
Discussion

At first, the previous results illustrate the influence of the ß phase stability (obtained through different solution treatment temperatures) on the precipitation and hardening kinetics. A reduction of solution treatment temperature increases the stability and the precipitation boundaries are shifted towards higher durations. The domain of existence of the ω phase is greatly reduced, whereas the one of the α phase is enlarged. For treatments performed below 800 °C, there is no more ω phase.

The morphology of the ω phase is related to the misfit between ß and ω. In particular, Mo or Nb give a small contraction of the ß lattice, then a limited misfit and an elliptical shaped ω. At the contrary, the ß lattice is dilated by additions of Cr, V and Fe ; the misfit is larger and the ω particles are cubic [3]. Elements such as Zr or Sn, increase ß phase stability and reduce the ω phase domain ; Al makes the α phase precipitation faster, and, consequently, lowers the ω stability duration. Considering the ß-CEZ composition, the presence of Al, Zr and Sn minimizes the ω phase domain of existence. It should be noticed that the elliptical shaped ω particles are stable at high temperatures and can be as large as 2000 Å, which is not the case here. However, in the present case [3], it has been found such morphologies.

Of course, the hardening kinetics are strongly related to the precipitation rate, the nature of the phases and their size. It can be said that either ω or α particles reinforce the ß matrix. As the ß phase stability increases, the ω and α phases precipitation rates are lowered. In the case of α+ß treated specimens, the ω phase precipitation is delayed, compared to the ß treated ones ; the hardness evolves more slowly. For higher solution treatments temperatures, the material hardens because of the presence of α phase. Compared with other alloys, the kinetics are comparable to the ones of 10-2-3 and SP 700 (metastable ß alloys) [4,5] : the heat treatment response is important and relatively fast (few mn), whereas, for Ti-64 (α+ß alloy) [6], it is fast but limited, and for Ti 15-3 (ß alloy), it is important but slow. In figure 8, the

hardening kinetic for 830 °C solution treated microstructure is shown together with the ß phase proportion evolutions, which is in turn related to α phase precipitation rate.



-Figure 8-
Comparaison of the hardening kinetic and ß phase proportion during ageing, at 550 °C, of a specimen solution treated at 830 °C.

The increase in hardness is directly related to α phase precipitation. For durations longer than 10 h, the decrease is related to the coarsening of the precipitates (cf figure 2 (c)) [7].
On the industrial viewpoint, the ß-CEZ processing includes a solution treatment in the α+ß field followed by ageing in the 500-650 °C temperature range. This study shows that this industrial practice, even performed on small sized specimens (for which water quench would retain maximum ßm quantity) is relatively immune from ω phase existence.

Conclusions

For the ß-CEZ alloy, a decrease in the solution treatment temperature enhances the ß phase stability. The ω phase precipitation is delayed and its domain of existence severely reduced. It disapears for solution treatments below 800 °C. The α phase domain of existence is also slightly shifted. The hardness levels and variations depend on the fineness of the structure and the precipitation kinetics. In the conventional ageing treatment temperature range (500-650 °C), no ω phase precipitation is to be feared and hardening is related to the kinetic of precipitation of α phase, whereas softening is due to α particles coarsening.

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