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The age hardening effect in Ti–6 Al–4 V due to ω and α precipitation in the β grains

ALAIN LASALMONIE, MARC LOUBRADOU
Office National d'Etudes et de Recherches Aérospatiales (ONERA), 92320 Châtillon, France

The structure at room temperature of a quenched TA6V titanium alloy has been investigated. This structure is $\alpha + \alpha'$ or $\alpha + \beta$ according to the treatment temperature; it is always metastable. During ageing the β grains decomposed by the reaction $\beta \rightarrow \beta + \omega + \alpha \rightarrow \beta + \alpha$; this decomposition was accompanied by a large increase of the 0.2% yield stress. No structural modification was observed in α. The α and β phase of TA6V were separately investigated in the form of single-phase alloys. The hardness of α was insensitive to ageing, while β was considerably hardened by ω and α; we deduced that the strengthening of the minor phase β during ageing is mainly responsible for the hardening of TA6V.

1. Introduction

TA6V is a commercial alloy with 6 wt% vanadium and 4 wt% aluminium. Below the β transition at about 980° C, TA6V is a mixture of aluminium-rich α grains and vanadium-rich β grains. Upon quenching β may transform into α' martensite, isostructural with α, so that the structure at room temperature of TA6V can be $\alpha + \alpha'$, $\alpha + \alpha' + \beta$ or $\alpha + \beta$, depending on the annealing temperature. The structure is metastable and age hardening effects are generally observed after tempering treatments.

Several mechanisms have been proposed to explain age hardening:

(1) The decomposition of the α' martensite into $\alpha + \beta$

(2) The formation of $\alpha_2(Ti_3Al)$ precipitates within α [2–4]

(3) The ordering of interstitials within α [4].

All these mechanisms lead to a hardening of the α phase, which has the highest volume fraction in the alloy. Few results have been published on the behaviour of the β grains during the tempering treatments. This phase however plays an important role in the strength of α–β alloys. Indeed, the law of mixtures does not apply to these alloys, since the strength of the two-phase alloy, in some cases, is close to that of the continuous phase [5]. Among the β decomposition products, one of the most potent strengtheners is ω. The ω precipitates might be responsible for the hardening after heat treatments at temperatures below about 400° C, but their existence in TA6V is yet to be proved [6]. It is the aim of this work to describe the decomposition of the β phase at a temperature where ω can precipitate. The temperature chosen in this particular case is 360° C. An attempt will be made to correlate various structures with the mechanical tests and discuss if hardening, due to tempering, plays a major role in the plastic deformation of TA6V.

2. Experimental

The composition of the TA6V rods used was: Ti–6.14% Al–4.04% V (weight per cent). The main impurities were: Fe (1160 p.p.m.), O (1500 p.p.m.), N (100 p.p.m.), H (163 p.p.m.), and C (40 p.p.m.).

For the tensile tests the deformation rate was: $\dot{\epsilon} = 1.5 \times 10^{-4}$ sec^{-1}. All the specimens were heat-treated for 3 h at 910° C and oil-quenched. They were then annealed for 4 h at temperatures between 680 and 850° C and water-quenched. These treatments were conducted under a vacuum

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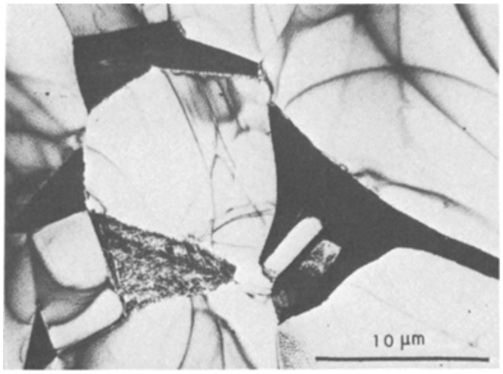


Figure 1 Microstructure after quenching from 910° C.

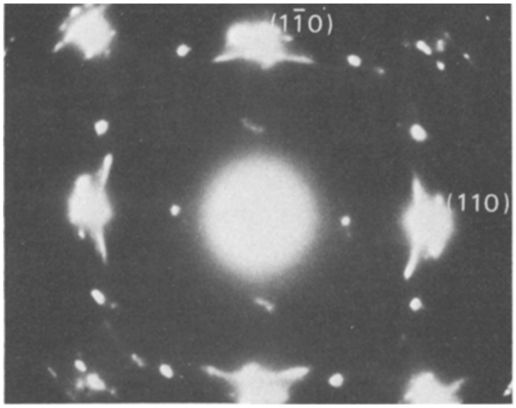


Figure 2 Diffraction pattern of α' after annealing at 850° C.

of 10^{-5} Torr. For tempering at 360° C, the samples were enclosed in a sealed silica tube and immersed in a molten salt.

The thin foils were prepared by conventional methods (jet polishing at $-40°$ C with the Struers A3 electrolyte).

3. The microstructure of TA6V

3.1. The crystallography and composition of the phases quenched from high temperatures

After quenching from 910° C, equiaxed α grains (diameter $\sim 10\,\mu$m) are embedded in an α' matrix (Fig. 1). Annealing at 850° C slightly modified the microstructure. Some grains decomposed into $\alpha + \beta$, and the diffraction patterns given by such grains are complex as shown on Fig. 2. This diagram is derived from the (0 0 1) zone of β. The peculiar feature is the presence of arced reflections which have been observed by Rhodes *et al.* [7] for Ti–Mo alloys. They have been attributed to the precipitation of lamellae, which do not follow the Burgers orientation relations, but rather are in a $(10\bar{1}2)\langle 10\bar{1}1\rangle$ twin orientation with respect to the Burgers orientation α.

The indexing of the diagram is given in Fig. 3 with two Burgers-orientated α_1 and α_2 families and their twins; however, this does not explain the whole diagram. Electron microscopy and X-ray

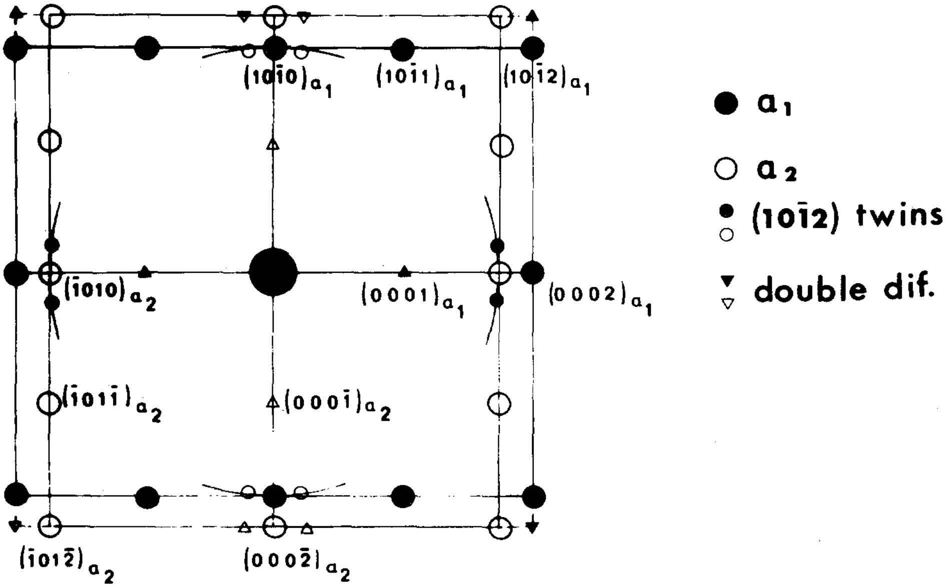


Figure 3 Indexing of the diagram with the Burgers oriented families α_1 and α_2, and their $(10\bar{1}2)$ twins.

Figure 4 Alloy annealed at 750° C.

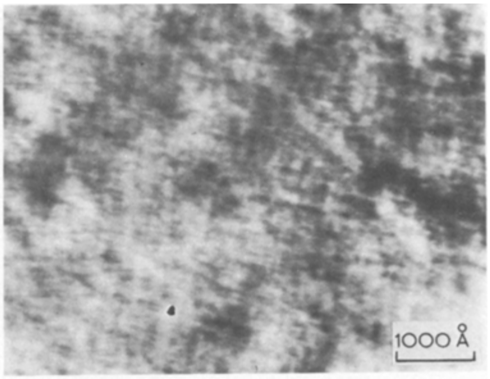


Figure 6 Mottled contrast in β.

experiments showed that, after the 750° C and 7000° C annealing treatments, β was sufficiently stabilized to be quenched at room temperature without transformation. The microstructure, as observed on optical micrographs (Fig. 4), is a mixture of equiaxed α grains and areas with intermingled α and β plates. In the specimens quenched from 800° C the three phases α, α' and β are found.

In all cases, the β grains exhibit an ω-type instability. This gives rise, on the diffraction patterns, to diffused intensity lines (Fig. 5) and to a mottled contrast of the bright-field images (Fig. 6). The phases α, α' and β were chemically analysed with the electron microprobe. The results, shown in Fig. 7, are in accordance with the data published by Castro *et al.* [2]. The arrows on Fig. 7 show that the measured amounts of Al and V in β for the alloy annealed at 800° C are maximum and minimum values respectively (the β grains were too small to allow an accurate analysis). In the specimens annealed below 800° C no analysis could be made as a result of the small size of the β areas.

It is concluded from these results that β can be quenched at room temperature, without undergoing any transformation, provided the amount of V is higher than 10% (the amount of Al is less than 5%).

3.2. The effect of tempering at 360° C

Our observations evince the decomposition $\beta \rightarrow \beta + \omega + \alpha \rightarrow \beta + \alpha$ during tempering at 360° C. In the specimens quenched from 800° C, ω is clearly observed in the β grains after 30 min at 360° C. The transformation of β into $\beta + \alpha$ is complete after 150 min.

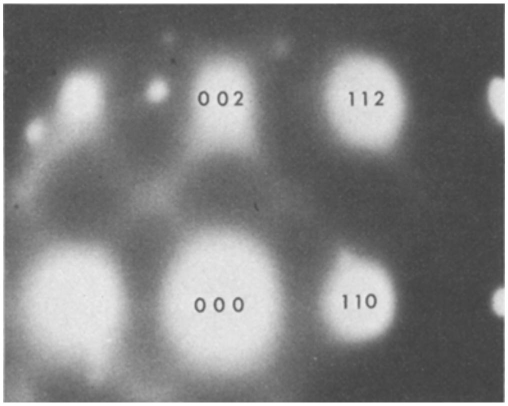


Figure 5 $(1\bar{1}0)$ diffraction pattern of β with $\langle 112 \rangle$ diffused intensity lines.

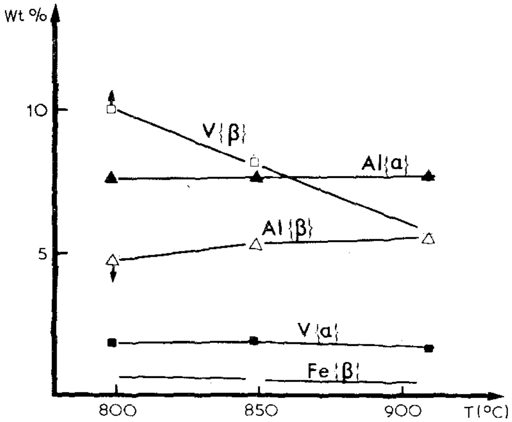
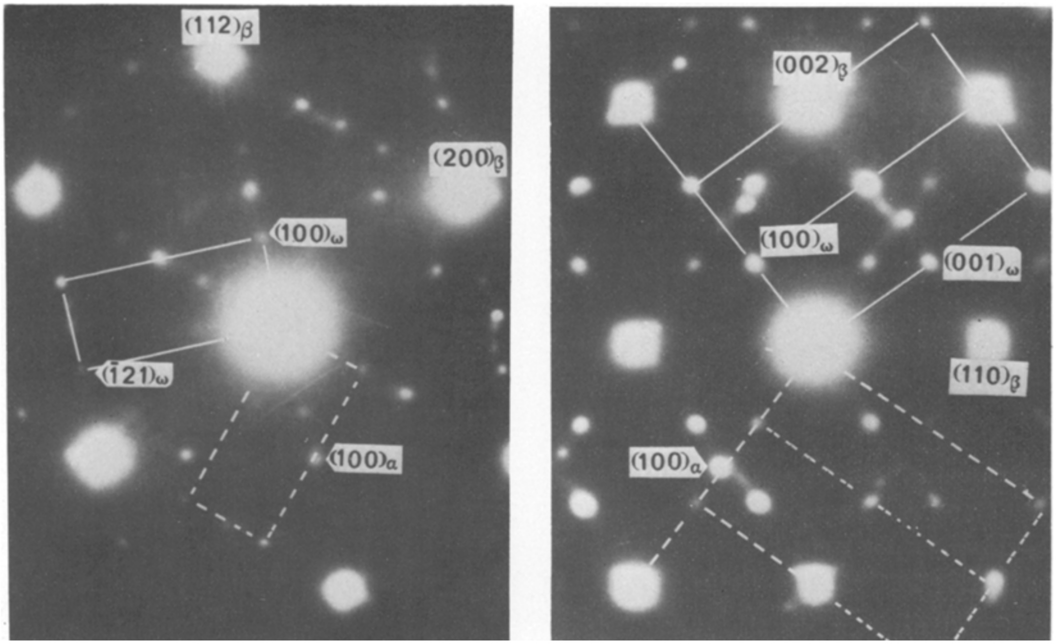


Figure 7 Chemical analysis of α and β (wt %).



Figures 8 and 9 Alloy quenched from 750° C. Diffraction patterns of β after 1 h at 360° C.

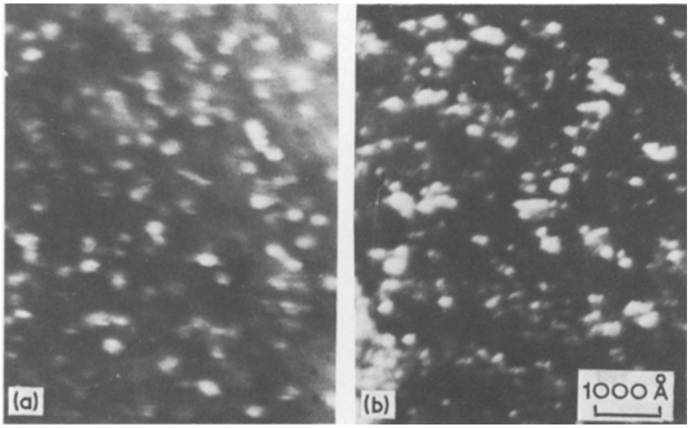


Figure 10 Dark-field images of (a) ω and (b) α.

In the samples quenched from 750° C, reflections from ω and α appeared in the diffraction patterns after one hour at 360° C. Typical diagrams are shown in Figs. 8 and 9 which are (0 2 1) and (1 1 0) zones of β. Figs. 10a and b are dark-field images of ω and α respectively, the size of these precipitates being about 150 Å. After 24 h at 360° C, ω has disappeared and only α lamellae are left within the β grains (Fig. 11).

As regards the specimens quenched from 700° C, no precipitation could be detected in the β grains after the tempering treatment. All these results are summarized in Table I. The problem of oxygen ordering at 360° C will be discussed in the

TABLE I Effect of tempering at 360° C on the microstructure of β in the quenched alloys

Quenching temperature (° C)	Time at 360° C					
	0 min	10 min	30 min	1 h	2 h 30 min	24 h
800	β	β	$\beta + \omega + \alpha$	$\beta + \omega + \alpha$	$\beta + \alpha$	$\beta + \alpha$
750	β	β	β	$\beta + \omega + \alpha$	$\beta + \omega + \alpha$	$\beta + \alpha$
700	β	β	β	β	β	β

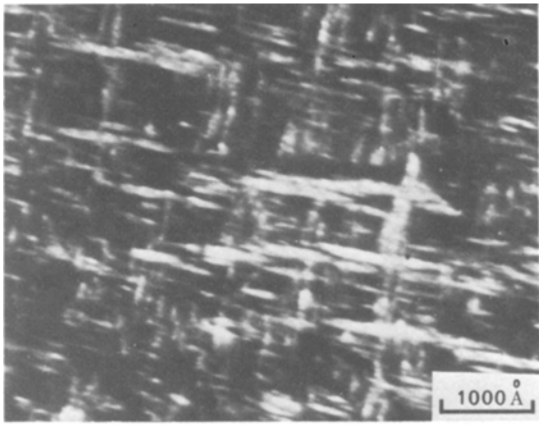


Figure 11 Precipitation of α platelets in β after 24 h at 360° C.

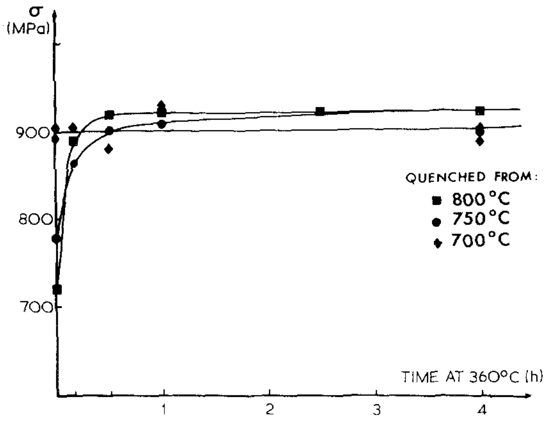


Figure 13 Influence of tempering at 360° C on the yield point.

next section. In α' the $\alpha' \rightarrow \alpha + \beta$ reaction was observed.

4. Mechanical properties

4.1. Yield strength

4.1.1. Quenched alloys

The 0.2% yield stress at room temperature goes through a minimum for the alloys quenched from 800° C (Fig. 12). The existence of this minimum has been reported for a number of α–β titanium alloys [8] and can be well explained qualitatively: When the annealing temperature is increased from 700° C to 800° C the volume fraction of β becomes higher; therefore, the yield strength decreases. Above 800° C, β is transformed after quenching into the stronger α' phase, leading to an increase of the yield strength. From a quantitative standpoint the level of strength reached after quenching from 900° C seems rather suprising, since α' is a "soft" martensite; the high value of stress presumably arises from the strengthening of this phase.

4.1.2. Tempered alloys

The tempering treatment at 360° C has no effect on the yield strength of the alloy quenched from 700° C. The structure of this alloy is the closest to the thermodynamic equilibrium conditions and no transformation of its phase occurs. For all the other alloys the yield stress increases and reaches a common value of 910 MPa (Fig. 13).

The hardening rate is especially high in the alloy quenched from 800° C: after 10 min at 360° C the yield point has increased from 720 MPa to 890 MPa.

Age hardening did not result in embrittlement. The homogeneous deformation was around 8% in all the cases.

4.2. Dislocation microstructures

The dislocation configurations were studied within the α grains. We looked for arrangements in tempered specimens which could suggest interstitial ordering (pairing of dislocations for instance).

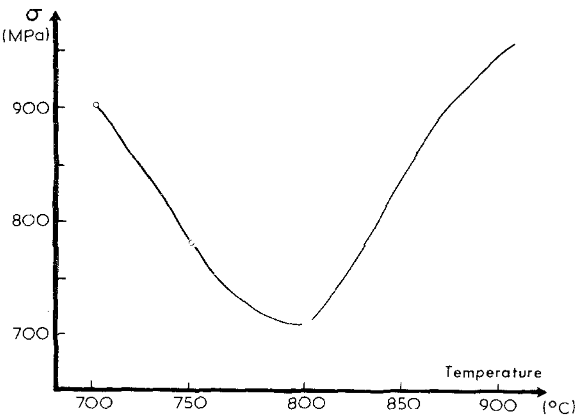


Figure 12 Yield point of the quenched alloys.

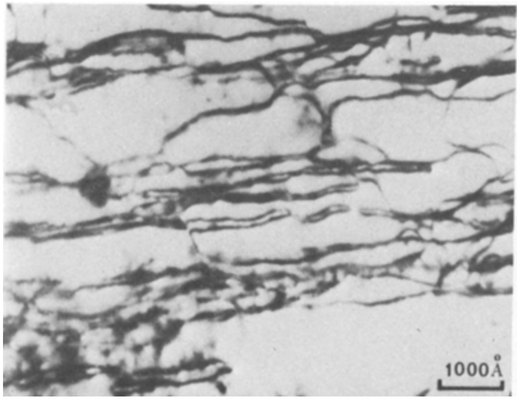


Figure 14 Dislocation dipoles in an α grain.

No evidence of pairing could be found, even in a deformed specimen which had been previously annealed for 350 h at 360° C. In this specimen numerous elongated dipoles which, at first sight, could be confused with dislocation pairs, were observed (Fig. 14). Whatever treatments may have been applied, the Burgers vector was $\mathbf{a} = \frac{1}{3}\langle 1\,\bar{1}\,2\,0\rangle$ and the slip plane $(1\,0\,\bar{1}\,0)$. In some cases basal slip was found to exist (Fig. 15), and occasionally the pyramidal plane of first kind $(1\,\bar{0}\,1\,1)$ and the $(1\,\bar{0}\,1\,3)$ plane were identified as slip planes in grains where the resolved shear stress on the prismatic planes was very low.

5. Discussion

The age hardening effect at 360° C can be very high in TA6V, since, for the alloy quenched from 800° C, the yield stress increases by about 30%. The most noticeable effect of tempering was the decomposition of β and α′. The ω precipitates are well-known effective strengtheners of β, and a good correlation is observed between the hardening and the decomposition of β to β + ω. We notice in particular, that when β remains stable (TA6V quenched from 700° C) throughout the tempering treatment, no hardening is obtained.

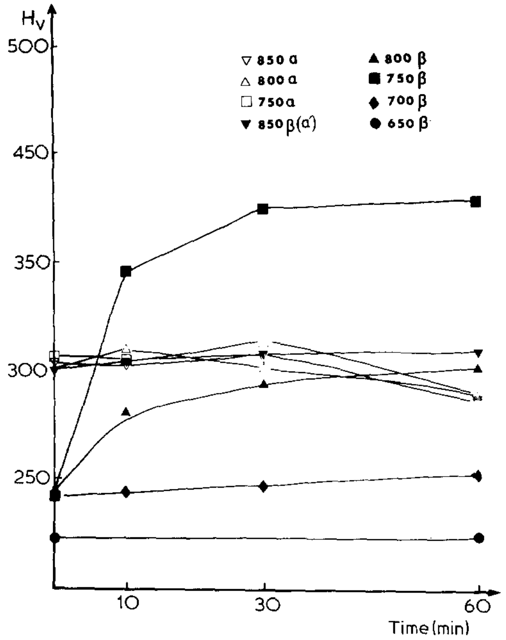


Figure 16 Effect of tempering at 360° C on the hardness of α and β alloys.

To check the influence of tempering on the mechanical properties of each phase, single-phase alloys were prepared whose compositions were close to those of α and β in TA6V at various temperatures (700° C, 750° C, 800° C, 850° C).

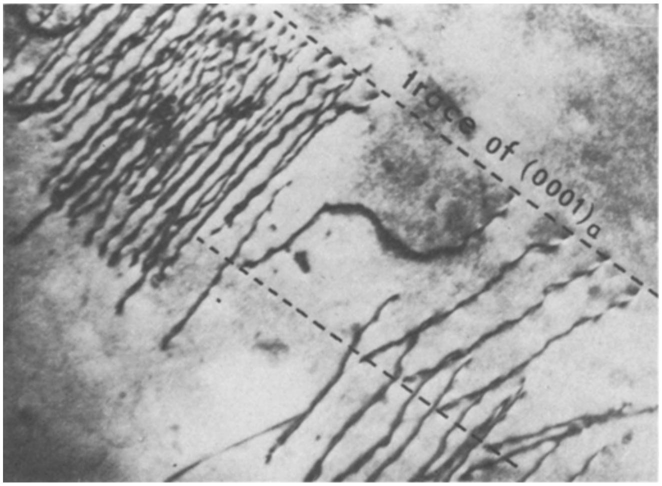


Figure 15 Basal slip in α.

TABLE II Compositions of the α and β alloys

Alloy T-phase	wt % V	wt % Al
850 β	8.7	4.6
850 α	2	6.9
800 β	11.4	4.3
800 α	2.2	6.5
750 β	14	4.1
750 α	2.4	6.2
700 β	18.8	3.7

The exact compositions are given in Table II (for all the alloys the amount of intersititials is 1500 p.p.m.).

The effect of 360° C tempering on the hardness is given in Fig. 16. There is no effect on the hardness of the α alloys, which rules out any possibility of α strengthening at that temperature. The precipitation of ω and α was observed in the β alloys as expected from the increase in hardness seen on Fig. 16.

These results clearly show that the age hardening at 360° C of TA6V alloys having a α–β structure is the consequence of ω and α precipitation within the β grains.

In the 800° C quenched alloy, the volume fraction f of β, calculated from the chemical analysis, is about 0.30, while the increase of the yield point after tempering is $\Delta\sigma \simeq 200$ MPa. If the law of mixtures is applied, the hardening of β should be $\Delta\sigma_\beta^0 = \Delta\sigma/f = 660$ MPa, but such a high value of $\Delta\sigma_\beta$ is unrealistic. Indeed, the hardening of β is mainly an increasing function of the volume fraction f_ω. In binary Ti–V alloys, the increase of the yield point, in cases where $f_\omega > 0.6$, is less than 600 MPa [9, 10]. In the β phase of TA6V quenched from 800° C, f_ω, as observed on thin foils, was only around 0.5; moreover the hardness increment of the 800_β single phase alloy is only $100 H_\mathrm{v}$, which corresponds to a value of $\Delta\sigma_\beta =$ 300 to 400 MPa, $\sim \Delta\sigma_\beta^0/2$. The actual contribution of β to the yield point of TA6V is therefore much larger than $f\sigma_\beta$. This behaviour may arise from the tendency of the beta phase to remain at the grain boundaries or at triple points.

This observation emphasizes the role played in the mechanical properties, by the morphology of a minor phase while it forms a quasi-continuous network around the grains of the major phase.

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