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Relationship between the structure and mechanical properties in ß III titanium alloy

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The ageing reactions that take place during the heat-treatment of solution-treated β III titanium (11.5 wt % Mo, 6 wt % Zr, 4.5 wt % Sn, balance Ti) were followed by detailed structure characterization using electron microscopy. The variations in mechanical properties with heat treatment were also followed systematically. The electron microscopy investigations indicated that the omega phase formed on quenching. The size and volume fraction of the omega phase increased on subsequent ageing. β, ω and α phases were found to co-exist at ageing temperatures between 800 and 900° F (427 and 482° C) for short ageing times. From the observations of interfacial dislocations at the β/ω interface and the precipitation of fine alpha near the omega particles, with a morphology that is characteristic of the prior ω morphology, it is suggested that the α-phase forms directly from the omega phase. The observed increase in yield strength over the solution-treated condition, due to the precipitation of ω phase, was found to agree well with that predicted by the Orowan hardening mechanism. Since the precipitation of fine ellipsoidal α-phase was found to increase the yield strength of the alloy with reasonable ductility, it is suggested that the optimum heat treatment to produce high strength with good ductility in β III titanium is to age at 900° F (482° C) for 10 to 25 h.

1. Introduction

β-phase titanium alloys are a commercially useful group of alloys owing to their good ductility in the solution treated condition and their excellent response to thermomechanical treatments [1]. It is relatively easy to obtain strength levels in excess of 1400 MPa [2] in these alloys by simple heat treatment procedures [3]. However, during ageing, the structural changes which occur in β titanium are very complex and depend critically on time, temperature and prior deformation [3]. This investigation was undertaken with the objective of systematically studying the structural changes occurring on ageing of the β III titanium alloy and relating these to changes in Young's modulus and tensile properties. The β III alloy, of nominal composition Ti–11.5 wt % Mo–6 wt % Zn–4.5 wt % Sn, was selected because it is known to respond well to thermomechanical treatments during which it develops a wide range of engineering properties [3, 4].

In the β III alloy, Mo, Zr and Sn act together to stabilize the b c c beta phase to temperatures much below the $\alpha \rightarrow \beta$ equilibrium transformation temperature. The metastable β phase formed on quenching of β III transforms to the hexagonal omega and/or hexagonal close packed alpha phases depending on ageing time and temperature [5]. The orientation relationship between the ω phase and the β matrix has been established by the early X-ray work of Silcock *et al.* [6] and Bagaryatski *et al.* [7]. The mechanism of ω formation [8, 9], the morphology of the ω phase [10, 11] and the effect of misfit on the morphology of ω phase [12] have also been dealt with in detail. The precipitation of the equilibrium α-phase upon ageing at higher temperature has been reviewed in detail by Williams [4]. In general, ageing at temperatures higher than the stability limit of the ω phase results in precipitation of coarse α which produces low tensile ductility. The precipitation of α is more uniform and fine when the alloys are aged

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at lower temperatures to form α from ω phases which results in superior ductility and higher strength levels [5, 13]. Similar results have been obtained by deformation and then ageing because of the higher density of available nucleation sites [14, 15].

In a previous paper [16] the authors reported the variation of the Young's modulus of β III titanium upon ageing. The modulus varies from 75 GN m^{-2} in the solution-treated condition to 127 GN m^{-2} in the condition aged for ω phase. Since the ω phase has a higher modulus [17] than the β phase it is apparent that the increase in modulus on ageing is due to the precipitation of ω phase on low-temperature ageing. A mathematical relationship has been developed relating Young's modulus of the aged material with volume fraction of the ω phase [18]. This theory was used to predict the volume fraction of the ω phase knowing the Young's modulus of the aged material. The predicted volume fraction was found to agree very well with volume fraction obtained from direct observation. However, in the above formulation a fixed value for the Young's modulus of β phase in the solution-treated condition was used. Vigier *et al.* [19] have suggested that the Young's modulus of β phase should be a variable because of the enrichment of the β phase with Mo during the ω precipitation [5] and the subsequent increase in the Young's modulus of β phase [17]. In this investigation, the variations in mechanical properties with heat treatment of β III titanium were measured, and correlations between mechanical properties and microstructure were determined.

2. Experimental procedure

2.1. Material

The β III alloy was supplied by the Metals and Ceramics Division of the Air Force Materials Laboratory, WPAFB, Ohio, in the form of 6.25 mm diameter rods. The composition of the alloy (wt %) is given in Table I. The as-received material was swaged at room temperature without intermediate annealing to 3.2 mm diameter rods for preparing specimens for transmission electron microscopy (TEM) and hardness measurements. Part of the as-received material was also swaged to 1.50 mm diameter wires for tensile mechanical property measurements.

TABLE I Chemical analysis of β III titanium Alloy

Element	W%
Mo	10.95
Zr	5.86
Sn	4.48
Fe	0.11
O	1515 ppm
C	224 ppm
N	80 ppm
H	45 ppm

All the specimens were solution treated at 1500° F (816° C) for 30 min followed by a quench into water. The specimens were then aged at temperatures between 400 and 900° F (204 and 482° C) at 100° F (55° C) intervals for times varying from ½ to 50 h, after which they were furnace-cooled. All heat treatments were done in a dry nitrogen atmosphere. Specimens were cleaned before and after each heat treatment by chemical polishing in a solution of 30% HF and 70% HNO_3 (by volume) to remove any surface contamination. Specimens for TEM were prepared by cutting thin discs 0.50 mm thick from all aged material using a Micromatic wafering machine and electropolishing on a twin jet electropolishing unit. A solution of 45% methyl alcohol, 45% butyl alcohol and 10% perchloric acid was used as the electrolyte. The solution was cooled by keeping the system in a bath of dry ice and methyl alcohol. The thin foils were observed on an Hitachi HU-125 electron microscope operated at 125 kV. The yield strengths of the heat-treated specimens were measured in tension on a floor model Instron using specimens of 1.50 mm diameter and 25.4 mm gauge length at a nominal strain rate of 3×10^{-4} sec^{-1} at room temperature. One set of specimens was prestrained in tension 5% prior to ageing. The yield strengths of the prestrained material were also measured after each heat treatment. Wire specimens of 3.2 mm diameter and 12.7 mm length were used for hardness measurements. Knoop hardness measurements were made on longitudinal sections of the specimens using a 400 g load on a Tukon microhardness tester.

3. Experimental results

3.1. Microstructural observations

The diffraction pattern shown in Fig. 1 is from a specimen in the solution-treated condition. The presence of strong diffuse intensity streaking in $\langle 1\,1\,2\rangle_\beta$ directions and also the presence of intensity maxima at the spots corresponding to the ω structure indicate that the ω phase nucleates

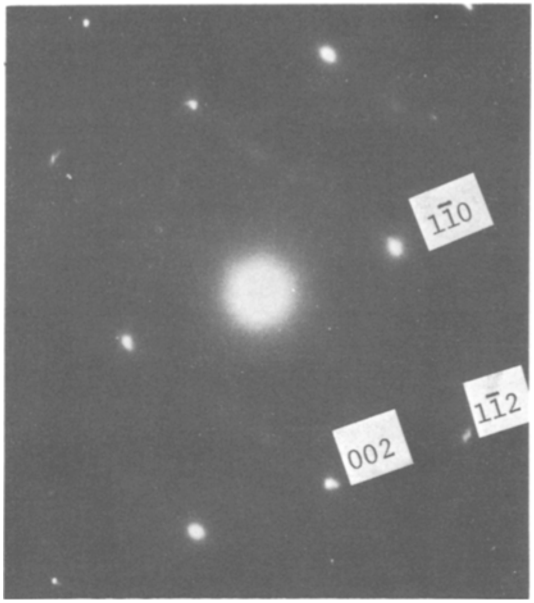


Figure 1 Selected-area diffraction pattern of β III-Ti solution-treated at 1500° F for 30 min and water quenched. Zone normal = $[1\ 1\ 0]_\beta$.

during the water quench from 1500° F (816° C). This is in confirmation with the work reported by Rack *et al.* [20] and Feeney and Blackburn [5]. However, the ω particles are not resolved in the electron microscope until after ageing at a temperature of 500° F (260° C) for 25 h. This structure is shown in Fig. 2. In general, the precipitation appears to be uniform throughout the matrix. The ellipsoidal morphology of the ω phase becomes evident after ageing for 50 h at 600° F (316° C), as shown in Fig. 3. As can be seen in this figure, the particles orientated normal to the foil surface have a circular cross-section. A typical dark-field micrograph of one variant of the ω phase taken from a specimen aged at 700° F (371° C) for $\frac{1}{2}$ h

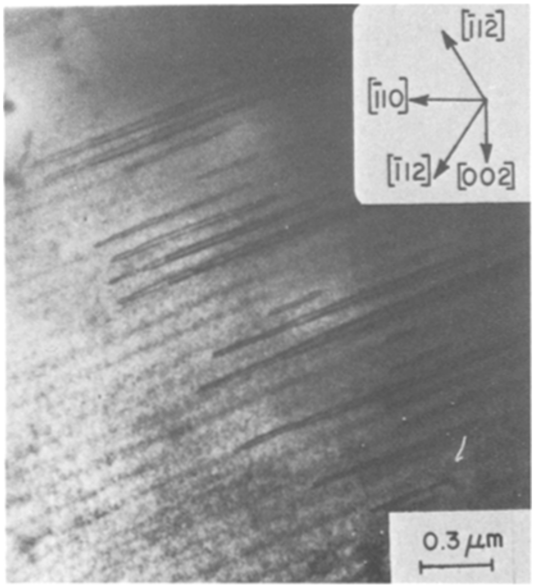


Figure 2 Electron micrograph of solution-treated β III-Ti aged at 500° F for 25 h. Bright-field. Zone normal = $[1\ 1\ 0]_\beta$.

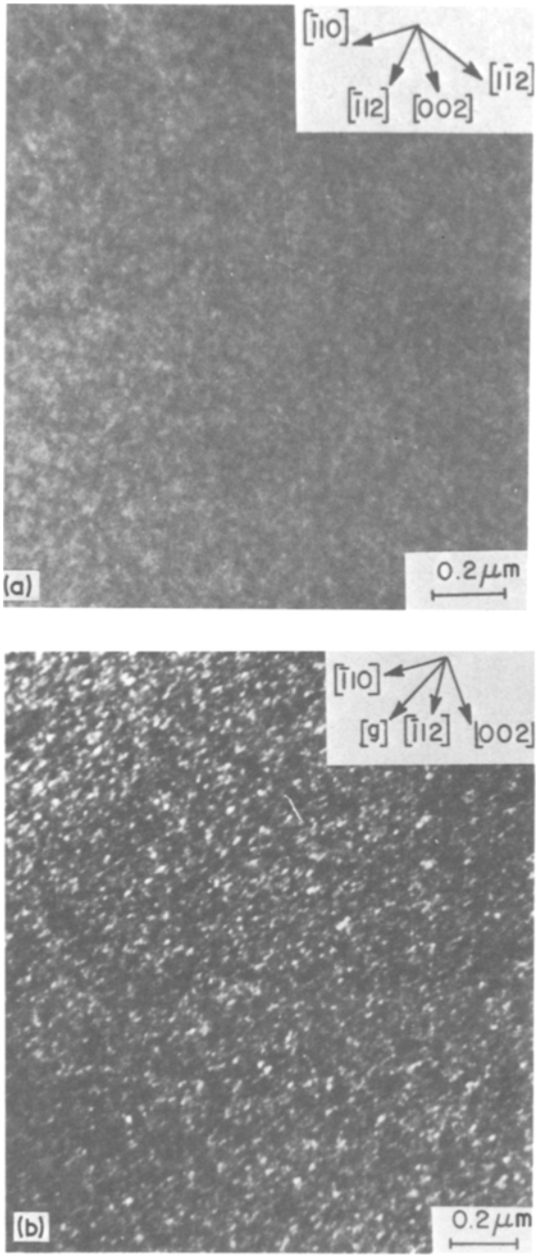


Figure 3 (a) Bright-field electron micrograph of solution-treated $[1\ 1\ 0]_\beta$ (b) Dark-field micrograph of a $(0\ 0\ \bar{1})_\omega$ spot.

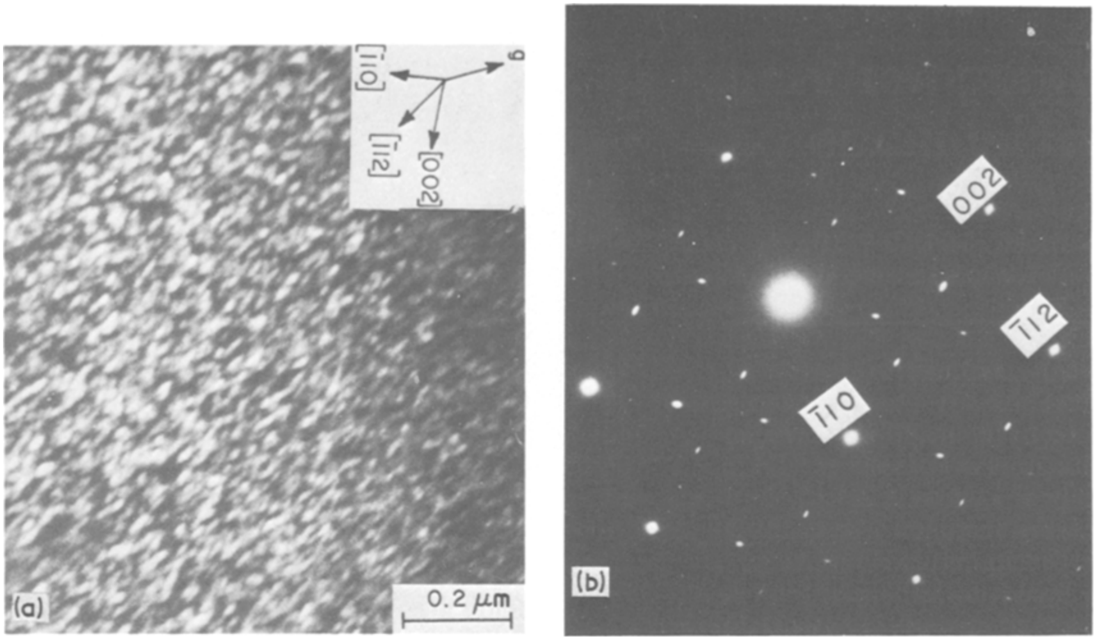


Figure 4 (a) Electron micrograph of solution-treated β III-Ti aged at 700° F for ½ h, dark-field. Zone normal = $\langle 1\ 1\ 0\rangle_\beta$. $g = (0\ 2\ \bar{2}\ 1)_\omega$. (b) Diffraction pattern of (a). Foil orientation is $\langle 1\ 1\ 0\rangle_\beta$.

TABLE II Values of Young's modulus and volume fraction of ω phase for specimens of β III titanium aged at 700° C

Ageing time (h)	Young's modulus (GN m^{-2})	V_f(% experimental)
½	110.0	48.8
1	112.8	50.6
5	119.3	54.6
10	122.0	56.3
25	125.8	58.6
50	128.6	60.4

is shown in Fig. 4a, and Fig. 4b is the corresponding diffraction pattern. Similar pictures were obtained for specimens aged at 700° F (371° C) for different ageing times. The volume fraction of ω phase was measured by point counting and the data are given in Table II. The results indicate that the volume fraction increases with ageing time. The total volume fraction of omega was determined by assuming that the ω forms equally in all the form variants. Luhman and Curzon [21] have shown that in Ti–22 at % Nb alloy unequal populations of the form ω-phase variants are possible because of preferential reversion of some variants of the ω-phase in the parent β-phase as a result of stress relaxation of the thin areas of the previously deformed foils. In these cases the diffraction pattern showed the absence of ω spots corresponding to a particular variant. However, in the present investigations no such deviation was observed as typified by the diffraction pattern shown in Fig. 4b. The particle density, N_v, obtained from these micrographs, was approximately 2.5×10^{14} cm^{-3}. This is in agreement with the measurements made by Jon *et al.* [22] on β III titanium.

Upon ageing at temperatures greater than 700° F (371° C) the α phase begins to nucleate. Fig. 5 is a bright-field electron micrograph taken from a specimen aged at 800° F (427° C) for 5 h. The microstructure contains ellipsoidal ω precipitates in a matrix of β phase. The major axis of the ellipsoids are parallel to $\langle 1\ 1\ 1\rangle_\beta$ directions. Interfacial dislocations are visible at the β/ω interfaces, and are considered to be likely nucleation sites for the precipitation of α.

The diffraction pattern shown in Fig. 6a taken from a specimen aged for 10 h at 800° F (427° C) contains reflections from β, ω and α phases. Fig. 6b is a dark field micrograph of the $(\bar{1}\ 0\ 1\ 1)_\alpha$ spot and Fig. 6c is a dark-field micrograph of the combined $(\bar{1}\ 0\ 1\ 1)_\alpha$ and $(0\ \bar{1}\ 1\ 1)_\omega$ spots. Comparing the areas marked A and B in Fig. 6b with the corresponding areas in Fig. 6c it can be seen that the ω phase particles are adjacent to the α

phase particles. The morphology of the α phase initially appears to be similar to the ω phase morphology, i.e. ellipsoidal. Fig. 7a is a $[1\,1\,0]_\beta$ diffraction pattern from a specimen aged at 900° F (482° C) for 10 h. The diffraction pattern from this specimen contains reflections from β, ω and α phases indicating that all phases co-exist at this ageing time and temperature. The dark-field micrograph of the $(0\,\bar{1}\,1\,1)_\omega$ reflection in Fig. 7b shows the morphology of the omega precipitate. A $[1\,1\,0]_\beta$ diffraction pattern of a specimen aged at 900° F (482° C) for 50 h is

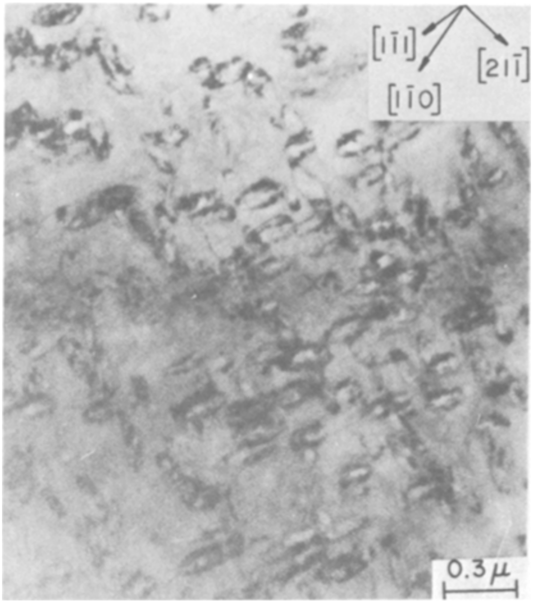
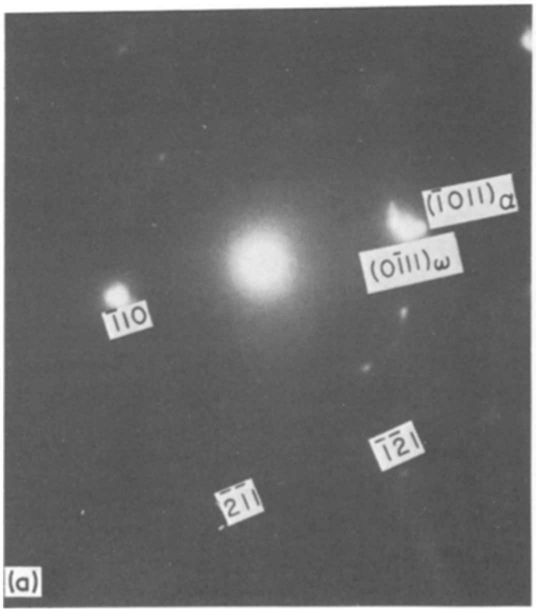
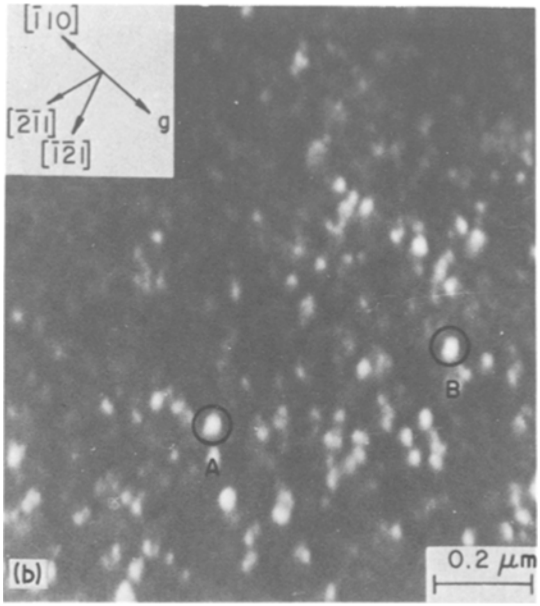
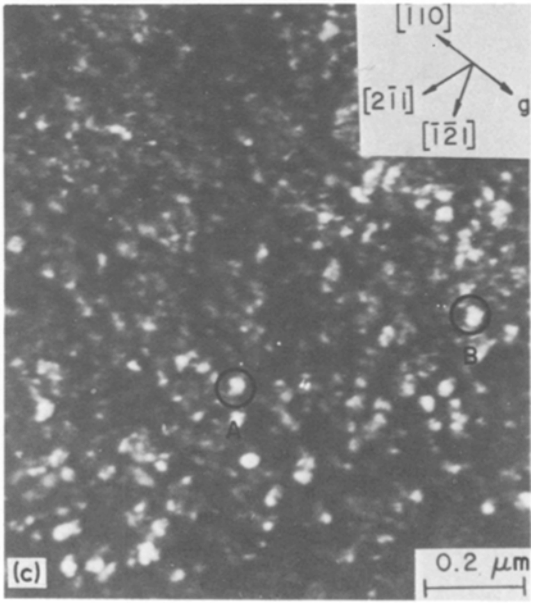


Figure 5 Bright-field electron micrograph of solution-treated β III-Ti aged at 800° F for 5 h showing dislocations at β/ω interfaces. Zone normal = $[1\,1\,3]_\beta$.

Figure 6 (a) Diffraction pattern of β III-Ti aged at 800° F for 10 h. Foil orientation is $[1\,1\,3]_\beta$. The diffraction pattern contains reflections from β, α and ω phases. (b) Ellipsoidal ω phase in β III-Ti aged 10 h at 800° F. Dark-field electron micrograph $g = (\bar{1}\,0\,1\,1)_\alpha$. (c) Dark-field electron micrographs of $(\bar{1}\,0\,1\,1)_\alpha$ and $(0\,\bar{1}\,1\,1)_\omega$ spots. Zone normal $[1\,1\,3]_\beta$.







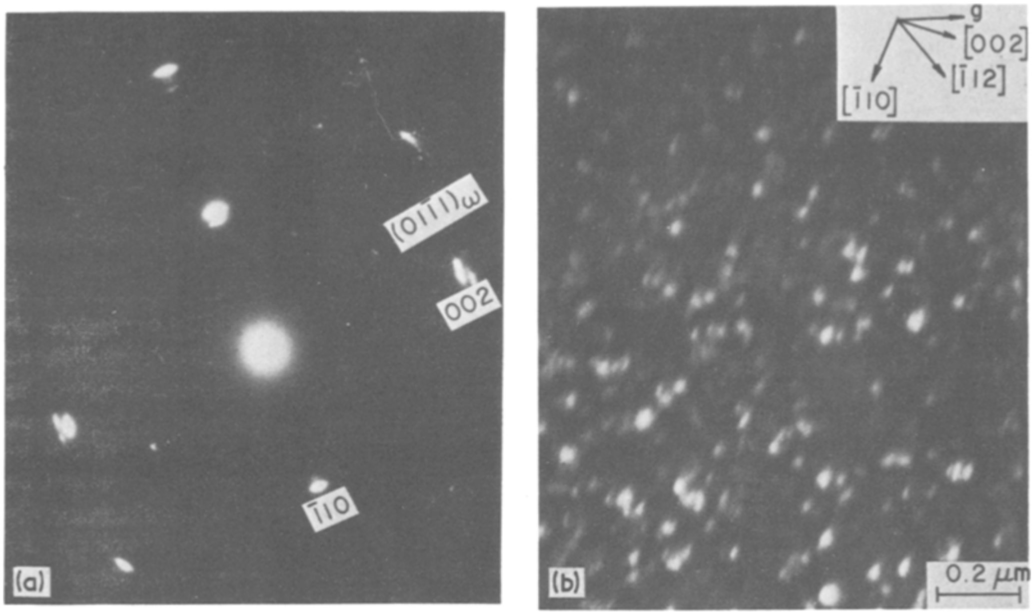


Figure 7 (a) Diffraction pattern of specimen aged at 900° F for 10 h. Foil orientation is $[1\ 1\ 0]_\beta$. Figure shows reflections from β, α and ω phases indicating that all phases co-exist at this ageing time and temperature. (b) Dark-field micrograph of $(0\ 1\ \bar{1}\ 1)_\omega$ spot showing ω precipitate.

shown in Fig. 8a. This diffraction pattern does not contain any reflections from the ω phase. The bright-field image of the same area is shown in Fig. 8b and it is now apparent that the α phase has a needle-type morphology. Thus morphology of the α phase changes from ellipsoidal, in the initial stage of formation, to needles in the later state of formation.

The microstructural observations can be summarized as follows: the structure consists of β and ω in the temperatures range 400 to 700° F (204 to 371° C). β, ω and α co-exist at 800 to 900° F (427 to 482° C) for short ageing times and the structure consists of $\beta + \alpha$ after ageing at 900° F (482° C) for longer times. This is in accord with the observations of Vigier *et al.* who studied the kinetics of

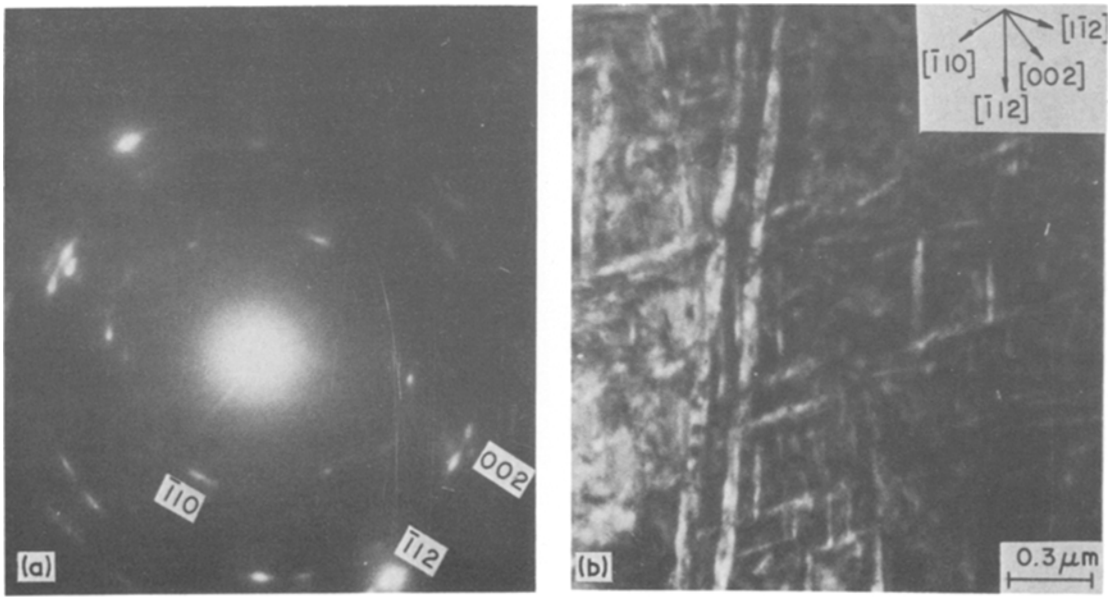


Figure 8 (a) Diffraction pattern of a specimen aged at 900° F for 50 h. Zone normal = $[1\ 1\ 0]_\beta$. (b) Bright-field electron micrograph of (a), showing needles of α phase.

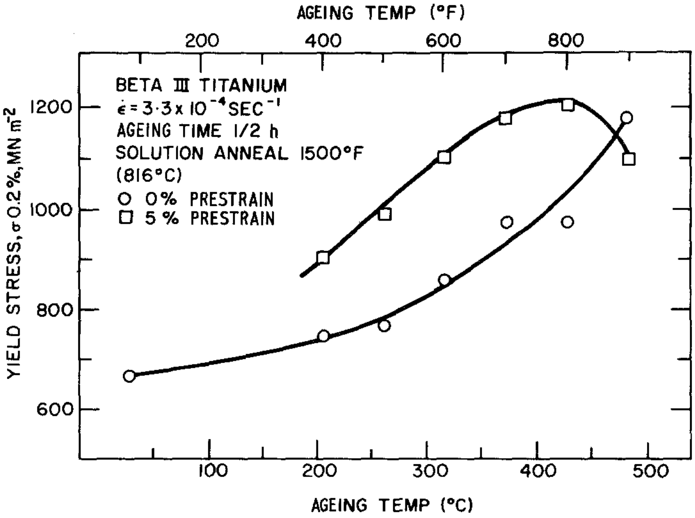


Figure 9 Yield stress of β III titanium as a function of ageing temperature.

phase transformations in β III titanium, based on the variation of the elastic modulus during the precipitation of the α and ω phases [23]. This is also shown by Feeney and Blackburn [5].

A plot of yield stress versus ageing temperature is shown in Fig. 9. The yield strength of β III is observed to increase rapidly with increasing ageing temperature. The effect of the 5% pre-strain is to increase the ageing response. The variation of the yield strength and plastic strain at ultimate strength as a function of ageing temperature for various times were reported elsewhere (Fig. 3 to 5, [16]). A good correlation was obtained between the microstructure and the mechanical properties. For example, at ageing times of 10 h and above in the temperature range 600 to 800° F (316 to 427° C) the specimens were brittle and fractured at low stresses, the brittle behaviour being associated with the formation of a large volume fraction of omega precipitate. However, at higher ageing temperatures and longer ageing times it was shown that the precipitation of alpha gives rise to a higher strength, more ductile, material. Similarly, the plastic strain at the ultimate strength decreased rapidly during ageing in the ω formation range and then increased with ageing at higher temperatures. The pre-strained material did not show any improvement in ductility even after ageing at 900° F (482° C).

A plot of hardness versus ageing temperature for different ageing times shows a classical hardening behaviour as can be seen in Fig. 10, the hardness increasing at temperatures up to ~ 700° F (371° C). The variation of hardness with ageing time for different temperatures is shown in Fig. 11. The decrease in the values of hardness at 800° F (427° C) as compared to 700° F (371° C) indicates that the strain fields around the ω particles, which tend to increase hardness, are most likely being relieved by the nucleation of α. As also can be seen from the data in Fig. 11, the alloy over-ages rapidly at 900° C (482° C); over-ageing being apparent at ageing times greater than 100 min. This, no doubt, is due to the rapid nucleation of fine α at this temperature.

3.2. Quantitative relationship between microstructure and mechanical properties

Second-phase particles can increase the strength of an alloy by acting as impenetrable barriers to dislocations moving in the matrix. Orowan [24] pointed out that in this case an additional stress will be needed to expand the dislocation between the particles and if the stress is large enough to bend the matrix dislocations into a semi-circular shape between the particles, the dislocations can by-pass them leaving encircling loops of dislocations at the particles. Consequently, Orowan [24] proposed that the initial flow stress of an aged alloy containing dispersed small crystals of a second phase could be given by an equation of the form

$$\tau = \tau_s + \frac{T}{b\lambda/2}, \tag{1}$$

where τ_s is the yield stress of the matrix without a dispersion, T, is the line tension of the dislocation, λ the planar interparticle spacing and b = Burgers vector. Since the initial equation was proposed sev-

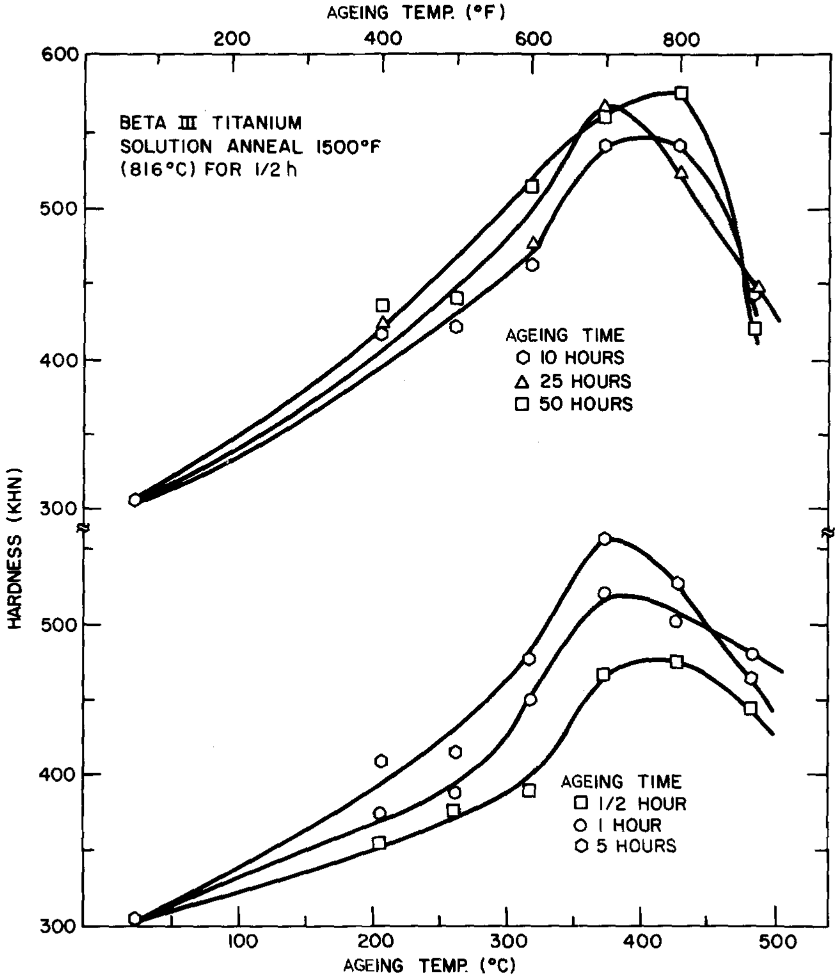


Figure 10 Effects of ageing temperature and time on the hardness of solution-treated β III titanium.

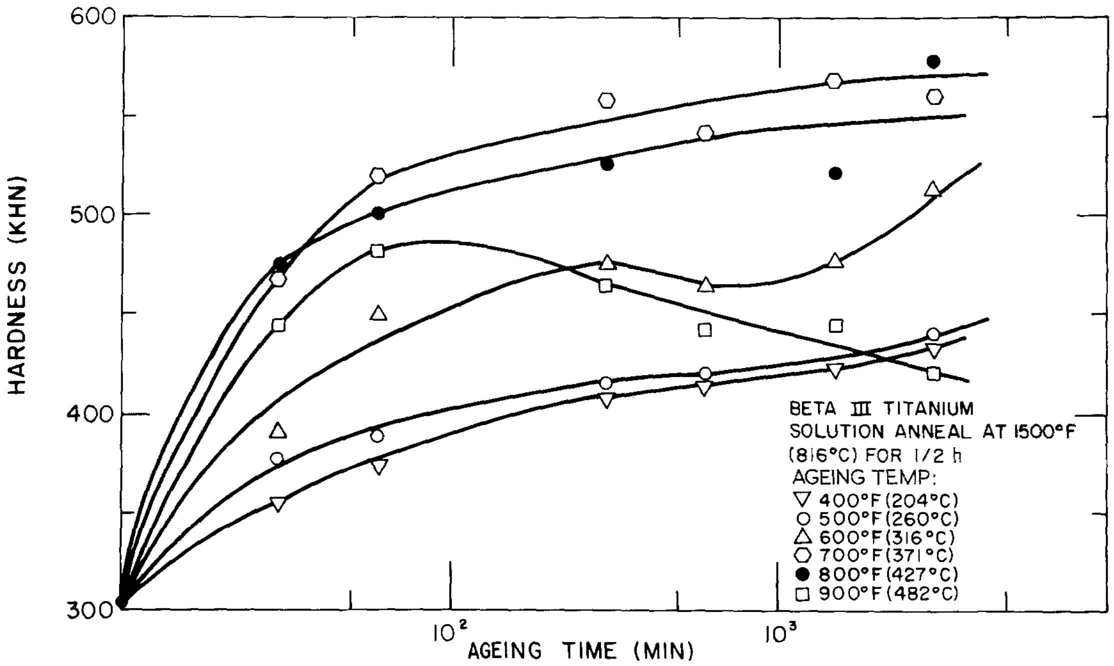


Figure 11 Hardness of β III titanium as a function of ageing time for different ageing temperatures.

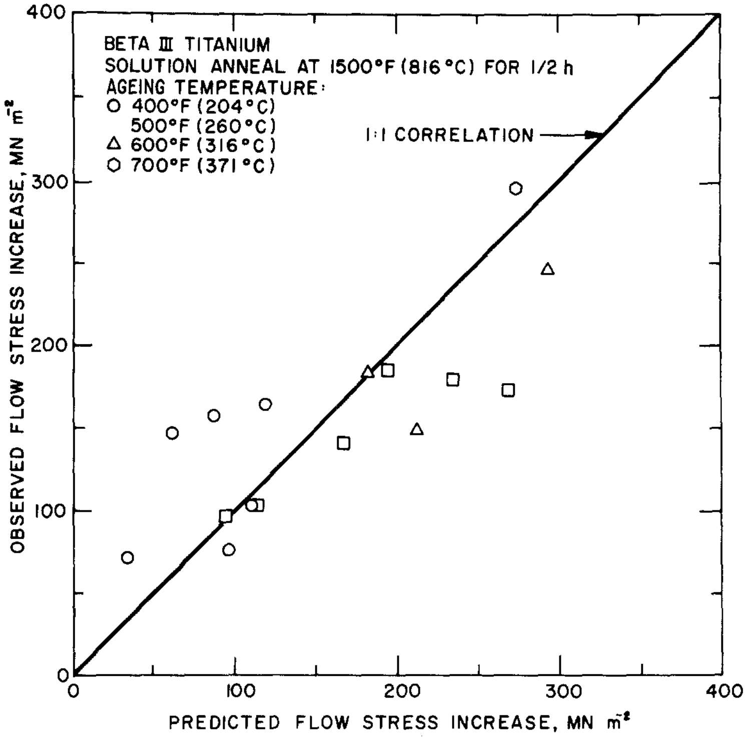


Figure 12 The observed increase in yield strength versus the increase in strength predicted by the Orawan hardening mechanism.

eral modifications of it have been made [24–29] which have resulted in the currently accepted version of the Orowan equation:

$$\sigma = \frac{0.83\,Eb}{(1.34)(2\pi)(1-\nu)^{1/2}} \; \frac{\ln\,[(\frac{\pi}{f})^{1/2}-2]\;R^1/r_0}{[(\frac{\pi}{f})^{1/2}-2]\;R^1} \qquad (2)$$

where E is Young's modulus, R^1 is the effective particle radius [26], f the volume fraction of the second phase, ν the Poisson's ration and r is the dislocation core radius. To determine if this equation agrees with the results obtained here on β III alloy, the observed increase in strength above that of the solution-treated condition has been plotted against the increase in strength as predicted by the Orowan equation, using $r_0 = 5b$ and $\nu = \frac{1}{3}$ (Fig. 12). Good agreement is obtained between the predicted and experimentally measured values. This result indicates that the strengthening observed during ageing is produced by the effective barriers to dislocation motion provided by the ω particles.

4. Discussion

The mode of formation of α phase in metastable β-Ti alloys at low ageing temperature varies between different systems. In Ti–Mo(30) and Ti–Nb (10) the $\beta + \alpha$ structure grows by a cellular reaction from the grain boundaries consuming the $\beta + \omega$ structure. In Ti–V, however, Blackburn and Williams [30] have shown that the α phase platelets are nucleated at β/ω boundaries and that these grow at the expense of the ω phase to give a very fine distribution of α phase. In the present investigation of β III titanium at no stage in the ageing of $\beta + \omega$ structures was a cellular reaction observed. Also, there was no evidence of ω phase reverting to β by dissolving in the β matrix because at no stage in the ageing process was it observed that the diffraction patterns contained only reflections from the β phase. In fact, it has been shown from quantitative free-energy considerations that conversion of β to ω can lower the free energy of the alloy [8]; hence the reversion of $\beta + \omega$ to β^1, another b c c phase having a different solute concentration, should not occur. At ageing temperatures of 800° F (427° C) and 900° F (482° C) for short ageing times the β, ω and α phases co-exist. The initial morphology of the α phase is similar to the ω phase, i.e. ellipsoidal. The observations of dislocations in β/ω interfaces and the nucleation of the α phase very close to the ω phase lead one to believe that the α phase nucleates heterogeneously on the interface dislocation structure. The precipitation of α phase in a fine dispersion form (Fig. 6b) could easily account for the large increase in the yield strength of this alloy at 800° F (727° C) (Fig. 9). From these evidences it is clear that the α phase forms directly from the ω particles producing a very fine uniform distribution of α particles whose morphology has characteristics of the prior ω phase morphology. The mechanism by which ω transforms to α is not clear at present, although it seems reasonable to conclude that the order of phase transformations in this alloy is $\beta \rightarrow \omega \rightarrow \alpha$ as opposed to $\beta \rightarrow \omega \rightarrow \beta^1 \rightarrow \alpha$.

These observations are consistent with those made by Rosales *et al.* [14] who noted that step ageing of β III titanium, first in the ω temperature range followed by ageing in the α temperature range, gives a uniform α precipitate. Bowen [13] has also demonstrated that an improvement in strength in a metastable β alloy Ti–15 Mo can be achieved by precipitation of a fine distribution of α phase at an ageing temperature close to the upper limit of ω phase stability. From the observations that the precipitaion of fine α at the β/ω interfaces gives rise to a large increase in yield strength with reasonable ductility, it may be concluded that the optimum heat treatment to produce good strength and ductility in β III titanium alloy is to age at 900° F (482° C) for times between 10 and 25 h. Ageing at 900° F (482° C) for longer than 25 h produces α phase in the form of needles (Fig. 8b). This change in the morphology of α phase may be the cause of the decrease in yield strength and ductility at longer ageing times.

The plot of yield strength versus ageing temperature indicates that the yield strength of this material increases rapidly with ageing temperatures. This is in agreement with the fact that the particle size, and hence volume fraction, of ω phase increases with ageing temperature. Since the ω phase is inherently a stronger and less ductile phase than the β matrix, its formation results in an increase in the flow stress of the alloy upon ageing. The second-phase particles can increase the strength of an alloy in one of two ways. The particles may act as an impenetrable barrier to dislocation motion and force the glide dislocations to bow out and bypass them following the Orowan mechanism [24]. Alternatively, the second-phase particles may be cut or sheared by the glide dislocations. While there is no evidence of particles being cut or sheared by dislocations, dislocation loops around particles were observed frequently throughout the investigation of this alloy. The strength level estimated by assuming the Orowan process compares very well with the observed increase in the flow stress. Also, the σ/E ratio calculated for different conditions of heat treatment remained constant [16]. This is due to the fact that the only ω-particles to grow were those nucleated initially during the quench. Hence, the distance between the particles remains constant and changes only to the extent the particle size changes. Thus all the terms in Equation 2 other that E remain more or less constant. Thus a constant σ/E ratio indicates that both the increase in modulus and yield strength have the same dependence on the volume fraction of the ω phase.

At ageing times of 10 h and above in the temperature range of 600 to 700° F (316 to 271° C), the alloy is very brittle and fractures at low stresses. Silcock [31] discusses the mechanism of embrittlement and points out that only one of the four ω variants has $[0\,0\,0\,1]_\omega$ direction parallel to $[1\,1\,1]_\beta$, which is the predominant slip direction in the b c c structure. The other variants have irrational directions parallel to this direction and hence would act as barriers to slip. The very high hardness values observed in the above temperature range is consistent with this view.

The yield strength values are higher for the prestrained material under equivalent ageing conditions (Fig. 9). The increased ageing response of the prestrained material can probably be accounted for by the fact that the plastic deformation of the solution-treated β III titanium produces a martensitic transformation [30]. Subsequent ageing produces ω in the untransformed material and α in the transformed regions and on the dislocation substructure. This morphological change of the α precipitate may be the prime cause of the accelerated ageing response for prestrained material. The morphology of the α that forms upon ageing after prestraining does not improve the ductility of the alloy.

5. Conclusions

(1) In β III titanium the ω phase nucleates during the water quench from 1500° F (816° C). On subsequent ageing, the size and hence the volume fraction of the ω phase increases.

(2) During the low-temperature ageing in the temperature range 400 to 700° F (204 to 371° C) the structure contains the two phases, β and ω. For ageing temperatures of 800 to 900° F (427 to 482° C) for short ageing periods, β, ω and α phases co-exist. For longer ageing times at 900° F (482° C) the structure contains the two phases, β and α.

(3) The precipitaion of ω phase is uniform and the morphology is ellipsoidal with the major axis

of ellipsoids parallel to $\langle 1\,1\,1\rangle_\beta$ direction. For longer ageing times the morphology tends to be more spherical.

(4) The initial morphology of the α phase is ellipsoidal. Electron microscopic evidence suggests the possibility of direct transformation of ω to α. At high temperatures for longer ageing times the α phase is in the form of needles.

(5) The yield strength of this alloy increases with increasing ageing temperature due to the precipitation of ω phase. The observed increase in the yield strength over the solution-treated condition is in good agreement with that predicted by the Orowan hardening mechanism.

(6) Prestraining the material prior to ageing increases the yield strength but does not improve the ductility.

(7) The precipitation of the α-phase as a fine dispersoid results in a large increase in yield strength with reasonable ductiluty. The optimum heat treatment to produce good strength and reasonable ductility is to age at 900° F for times between 10 and 25 h.

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