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Effects of ageing on the temperature coefficient of resistivity of a β titanium alloy

C.H. CHUN, D.H. POLONIS
Department of Materials Science and Engineering, University of Washington, Seattle, WA 98195, USA

The temperature coefficient of electrical resistivity (TCR) of the commercial Ti–15–3 alloy is negative, if the alloy is in the 100% β condition. The TCR increases from negative to positive values as a result of precipitation of the ω and α phases during ageing. An incubation or transient period, during which the value of TCR remains essentially unchanged, precedes the formation of the ω phase; this transient period decreases with increasing ageing temperature over the range 250–400 °C. Changes in the value of TCR signal the initiation of beta decomposition before the ω phase can be detected by conventional TEM and diffraction techniques. Reversion of the ω phase by up-quenching after ageing restores the original negative value of TCR. If both α and ω phases are present in the aged condition, only partial recovery of the quenched TCR value is possible, indicating that the alpha phase is not reverted by up-quenching. The results point to the potential value of TCR determinations for monitoring the initiation of β decomposition in titanium alloys exposed to temperatures above room temperature.

1. Introduction

Negative values of the temperature coefficient of electrical resistivity (TCR or $d\rho/dT$) have been reported for β stabilized alloys in several titanium-based systems [1–5]. Explanations for this behaviour vary, with some investigators [5] proposing it is due to structural transformation and others [5–7] attributing the effect to the complex electronic band structure of alloys based on titanium and zirconium. A major point of controversy relates to the suggestion that the negative $d\rho/dT$ is linked to the thermally reversible formation of the ω phase in β stabilized alloys [5, 8]. While the results for Zr–Nb alloys support this argument, evidence for a variety of titanium-based alloys [4, 9–12] indicates the negative $d\rho/dT$ occurs in the absence of detectable ω phase and is, therefore, an intrinsic characteristic of the β phase over limited compositions.

Alloys of titanium containing low concentrations of the transition elements (e.g. Ti–5Cr, Ti–10V) generally exhibit mixtures of the α (or α′), β and ω phases in the quenched condition. At intermediate concentrations, α or α′ formation is suppressed and a mixture of the β and ω phases exists in the quenched condition. The amount of retained β increases with solute content until 100% retained β phase occurs. It has been reported [4] that the value of $d\rho/dT$ may be negative in quenched alloys containing 100% retained β, or even in alloys exhibiting a mixture of the β and athermal ω phases. The influence of solute content on $d\rho/dT$ is illustrated by results shown in Fig. 1 for the Ti–Cr system [4]. The value of $d\rho/dT$ decreases (i.e. becomes increasingly negative) when the chromium content is increased from 10 at % to 13 at %, followed by a small increase from 13 at % to 16 at %; however, a sharp rise in $d\rho/dT$ occurs when the composition is increased above 16 at %. The as-quenched constitution of these alloys changed from a β + ω mixture in the 10 at % composition to marginal, but complete β retention at 13 at %, and increasing β stability at higher chromium concentrations. The evidence therefore points to a reversal of the negative $d\rho/dT$ phenomenon with increasing stability of the β phase. In view of the sensitivity of $d\rho/dT$ to variations in the composition and constitution of titanium alloys, it is a potentially useful parameter for monitoring minor microstructural changes accompanying β decomposition during ageing or isothermal transformation. For example, previous work [4, 13] linked the changes in $d\rho/dT$ during ageing to a phase separation process in the β phase of Ti–V and Ti–Cr alloys; however, only a few investigations [4, 14–16] have employed $d\rho/dT$ values to monitor phase changes. The present project was undertaken to assess the value of $d\rho/dT$ measurements for monitoring the kinetics of microstructural changes due to ageing in the commercial all β Ti–15–3 alloy. This alloy was chosen to represent compositions showing strong β stabilization and good microstructural stability below 250 °C.

2. Experimental procedure

The Ti–15–3 all β alloy was provided in sheet form by the Timet Corporation. The nominal composition of the alloy is given in Table I; interstitial requirements are C < 0.05%, N < 0.05%, O < 0.15%, H

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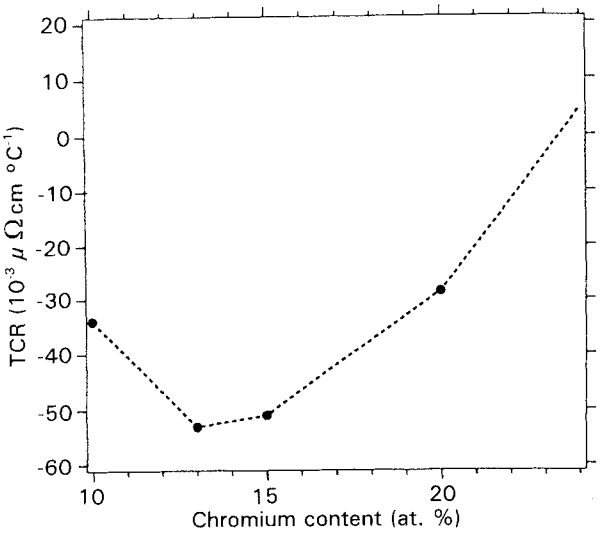


Figure 1 Influence of chromium content on the temperature coefficient of resistivity (TCR) of quenched binary Ti–Cr alloys [4].

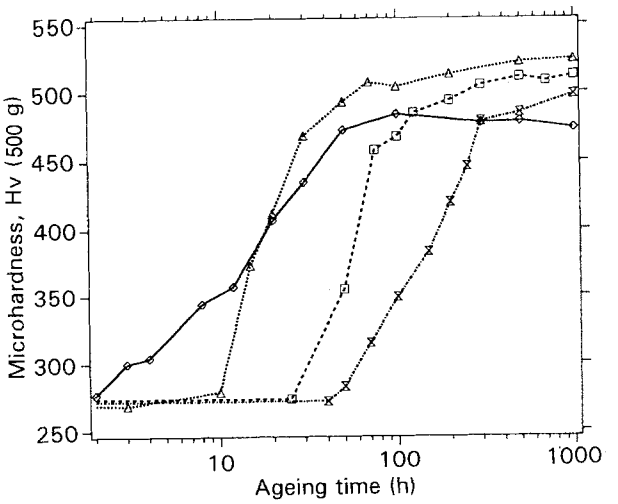


Figure 2 Effect of ageing time and temperature on the microhardness of the Ti–15–3 alloy. (⧖) 250 °C, (□) 275 °C, (△) 300 °C, (◇) 350 °C.

TABLE I Composition specifications Ti–15–3 alloy

Element	wt %
V	14–16
Cr	2.3–3.5
Al	2.5–3.5
Sn	2.5–3.5
C	0.05 max.
N	0.05 max.
O	0.15 max.
H	0.015 max.

< 0.015%. Solution treatments were carried out at 900 °C, using specimens that were wrapped in tantalum foil and sealed in evacuated silica capsules; quenching was accomplished by breaking the capsules under water. Pure titanium specimens were also used to monitor potential contamination. The ageing treatments were carried out at temperatures ranging from 200–500 °C using neutral salt baths controlled to ± 2 °C.

The electrical resistance measurements were made by means of a standard four-point balance technique, employing a Leeds and Northrup K-5 potentiometer and a Keithly 148 Nanovoltmeter as a null detector; measurements could be made to an accuracy of ± 1 × 10^{-3}Ω. Four copper leads, 0.4 mm diameter, were spot-welded to the ends of resistance specimens which were thin strips 6.4 mm wide, 50 mm long and 0.51 mm thick; the specimens were slit at both ends to connect the leads, two of which were connected to the current supply and two were used for EMF measurements. Five different baths were employed to measure the electrical resistance at various temperatures, as follows: liquid nitrogen (77 K), methanol and dry ice (273 K), methanol and ice (273 K), methanol at room temperature (291–295 K), and boiling water (373 K). By reversing the current through the specimen and averaging the voltage drop in both directions, the effects of thermal EMF could be eliminated during the measurement of electrical resistance. The reproducibility of a measurement for any particular condition was better than ± 0.01%. The values of the temperature coefficient of resistivity (TCR), were calculated on the basis of the following relationship

$$\mathrm{TCR} = \frac{\mathrm{d}\rho}{\mathrm{d}t} = \frac{\Delta\rho}{\rho_{77\mathrm{K}}} \Big/ \Delta T \qquad (1)$$

By placing ρ_{77K} in the denominator, any contributions due to variations in the temperature or geometric components of resistivity were minimized and the dimensions of TCR are K^{-1}.

During the course of this work, extensive characterization studies were carried out using transmission electron microscopy and X-ray analysis techniques. The details of this part of the work are to be reported later, so only brief reference is made in this paper to substantiate the interpretation of the resistance results.

3. Results

3.1. Hardness results

Specimens of the Ti–15–3 alloy that had been solution treated at 900 °C for 30 min and water quenched were then aged at several temperatures over the range 250–400 °C for periods up to 1000 h. Fig. 2 shows the hardness of the alloy remained essentially unchanged during an initial incubation period, following which an increase accompanied continued ageing. X-ray diffraction (XRD) analysis and transmission electron microscopy (TEM) techniques confirmed that such hardening was accompanied by the precipitation of the ω and/or the α phase from β. As ageing temperatures were increased over the range 250–350 °C the incubation time decreased from approximately 40 h at 250 °C to less than 10 h at 350 °C; the maximum hardness value increased when the ageing temperature was raised from 250–300 °C, but was reduced for ageing at 350 °C, as shown in Fig. 2. Overageing was initiated after 100 h at 350 °C, but was not detected after 1000 h at the lower temperatures.

Reversion treatments were conducted at 500 °C after various prior ageing times at 275, 300 and 350 °C. The ageing times at each temperature were selected to examine the extent of the reversion process for successive stages of ω development and for mixtures of the ω and α precipitates in the β phase. Fig. 3a–c indicate the hardness of the alloy decreased after reversion, approaching a value similar to that of the original quenched condition for prior ageing at 250 °C. The results also show that the magnitude of the hardness decrease due to reversion is less pronounced after prior ageing at 300 and 350 °C, although the drop is significant.

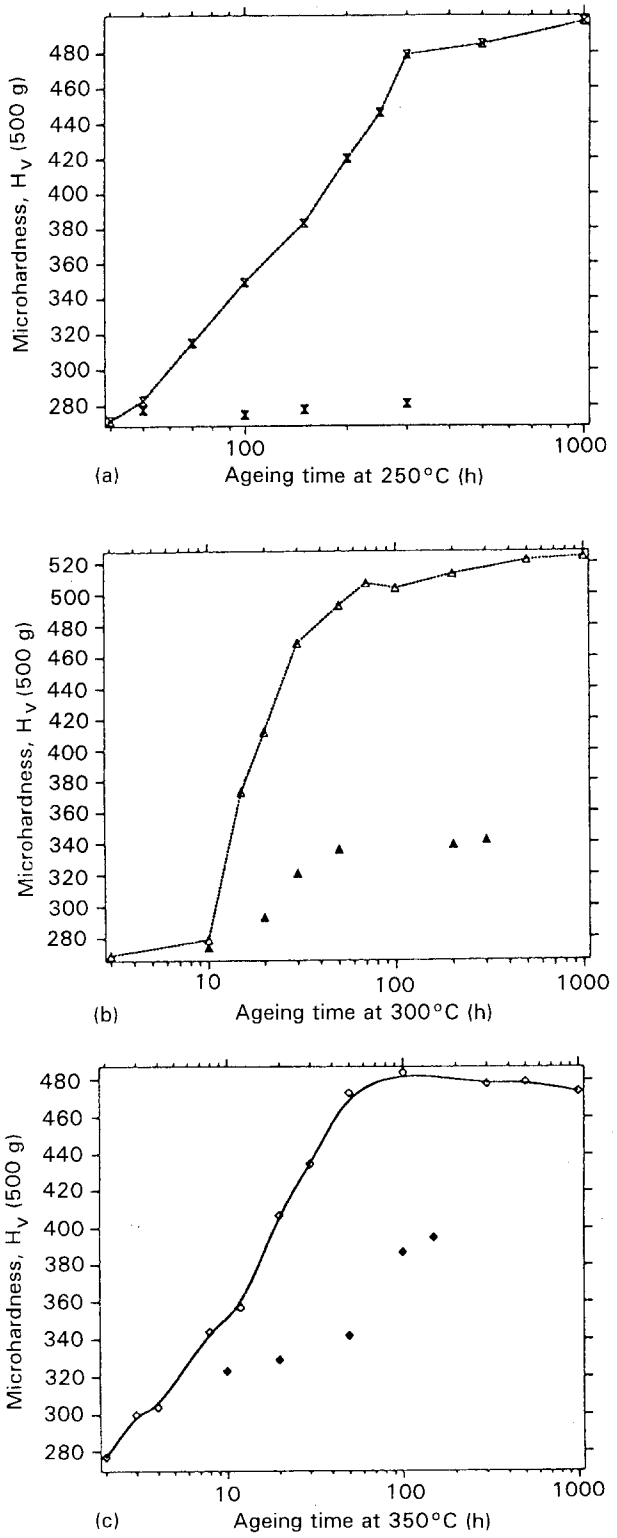


Figure 3 Comparison of the microhardness of the aged Ti–15–3 alloy (⧖, △, ◇) before and (⧗, ▲, ◆) after reversion after various ageing intervals at three ageing temperatures: (a) 250 °C, (b) 300 °C, (c) 350 °C.

X-ray diffraction analysis was used to identify the phases at various stages of ageing and after reversion; the results are summarized in Table II. The first evidence of the ω phase was an extra diffraction peak at 2θ values of approximately 66° and a broadening of the β phase peaks. During ageing at 250 °C, the ω phase was first detected after 120 h and the α phase after 250 h. At 275 °C a weak ω phase peak was first detected after 75 h and the initial evidence of the α phase was observed after 150 h ageing. Raising the ageing temperature to 300 °C produced ω after 7 h and α after 10 h. During ageing at 350 °C and higher, no evidence of ω precipitation was detected by X-ray diffraction, although the presence of α was first detected after 8 h at 350 °C, 3 h at 400 °C and 2 h at 500 °C.

3.2. Temperature coefficient of resistivity (TCR)

The temperature coefficient of resistivity (TCR) of the aged alloys, as defined previously, was determined from electrical resistance measurements conducted at several temperatures ranging from − 196–100 °C. The results for specimens aged at 250, 275, 300, 350, and 400 °C are shown in Fig. 4. The TCR values were originally negative in the quenched condition and remained relatively constant during the early stages of ageing, but changed significantly with the precipitation of the ω phase as ageing continues. With increasing ageing temperature, the incubation period decreases and the maximum TCR value increases, as shown in Fig. 4. The TCR value is still negative after 400 h at 250 °C, although it becomes progressively less negative as ageing continues. The results of X-ray diffraction analysis and transmission electron microscopy confirm that the progressive increase in TCR values reflects the precipitation of the ω and/or the α phase, depending on the ageing time and temperature.

TABLE II X-ray diffraction analysis of the phases present in the Ti–15–3 alloy aged at the indicated temperatures

Ageing temp. (°C)	Ageing time (h)	Phases present
250	120	β + ω
	150	β + ω
	200	β + ω
	250	β + ω + α
275	75	β + ω
	100	β + ω
	150	β + ω + α
300	7	β + ω
	10	β + ω + α
	16	β + α
350	8	β + α
	30	β + α
400	3	β + α
	5	β + α
500	2	β + α

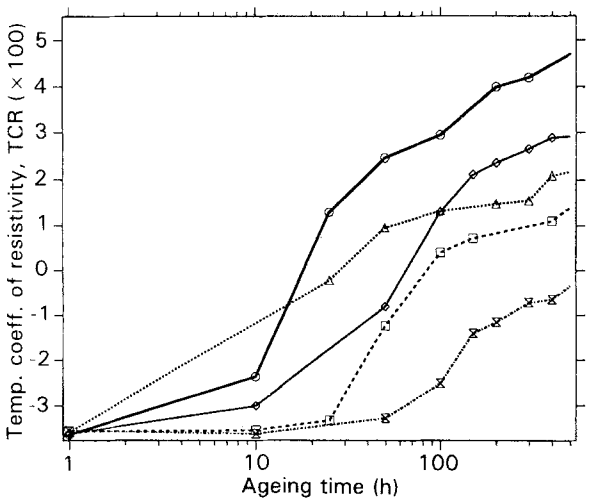


Figure 4 Variation in the temperature coefficient of electrical resistivity (TCR) of the Ti–15–3 alloy with ageing time at several treatment temperatures: (⊠) 250 °C, (□) 275 °C, (△) 300 °C, (◇) 350 °C, (○) 400 °C.

Raising the temperature of an aged alloy rapidly to a level above the ω formation range for several minutes, can cause reversion of the ω phase in a variety of metastable β alloys of titanium [4, 17–20]. The effects of such reversion treatments on the TCR value of the aged Ti–15–3 alloy are shown in Fig. 5a–e, for up-quenching and holding for 2 min at 500 °C, followed by quenching to room temperature. The results show that the TCR value of the alloy previously aged at 250 °C returned to the as-quenched value, suggesting the complete reversion of the ω phase. The original as-quenched value of TCR also was recovered after reversion in specimens aged for less than 40 h at 300 °C. Specimens aged for longer than 40 h at 300 °C showed only partial recovery of the TCR; the difference between the reverted and the as-quenched values of TCR increased with increasing ageing time beyond 40 h as shown in Figs 5c and 6. After ageing at 350 and 400 °C the reversion treatment resulted in only partial recovery of the TCR value and the difference with respect to the original quenched value increased with increased ageing time, as shown in Figs 5d, e, and 6.

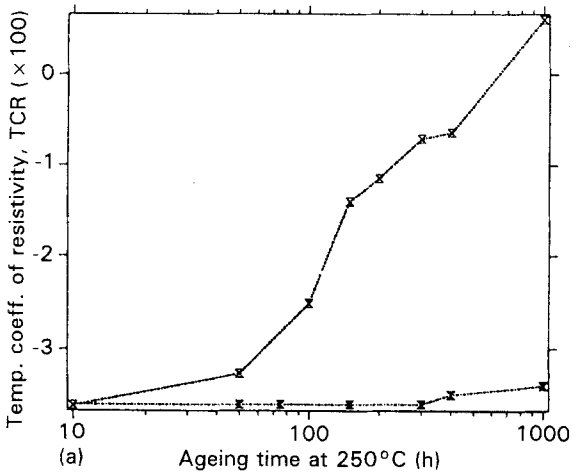
3.3. TEM observations

The Ti–15–3 alloy exhibited 100% β phase in the as-quenched condition. After ageing at 250 °C for 100 h, the SAD patterns exhibited diffuse streaking, as shown in Fig. 7. Ageing for 200 h at 250 °C produced reflections of the ω and α phases in the SAD pattern, as shown in Fig. 8a–c. Uniformly distributed ellipsoidal particles of the ω phase, ranging in size from 10–15 nm, were observed in the dark field, as shown in the micrograph of Fig. 9. The α phase could not be resolved by the dark-field technique after 200 h at 250 °C, due to the small volume fraction and the low intensity of the reflections. Ageing for 1000 h at 250 °C revealed uniformly distributed α particles 5–10 nm in size, as shown in the dark-field micrograph of Fig. 10; the ω particles showed some indication of change from ellipsoidal to cubic morphology after 1000 h, but no apparent change in the density or size of the ω particles was evident for ageing beyond 200 h. During ageing at 300 °C, or higher, the α phase was the predominant precipitate; α particles could be resolved using dark-field imaging after ageing for 20 h at 300 °C; the α phase formed as clusters of particles arranged in rows parallel to the $\{1\,1\,0\}_\beta$ planes, as shown in Fig. 11.

TEM observations revealed the complete reversion of the ω phase after up-quenching to 500 °C following ageing treatments at 250–300 °C; however, it was not possible to image the solute-lean zones. The α phase particles exhibited morphology changes due to holding at 500 °C after up-quenching. Particle coarsening and blunting of the platelets was evident after 2 min holding time (Fig. 12) and this change became more pronounced when the hold time at 500 °C was increased to 5 and 35 min (Fig. 13).

4. Discussion

The results of the present work show that the TCR of the Ti–15–3 alloy is negative after quenching from the solution temperature to retain 100% β phase at room temperature. TEM and X-ray diffraction analysis did not reveal evidence of the athermal ω phase or the martensitic α′ phase in the quenched condition, thereby confirming the negative value of TCR is a characteristic of 100% β phase. An incubation period



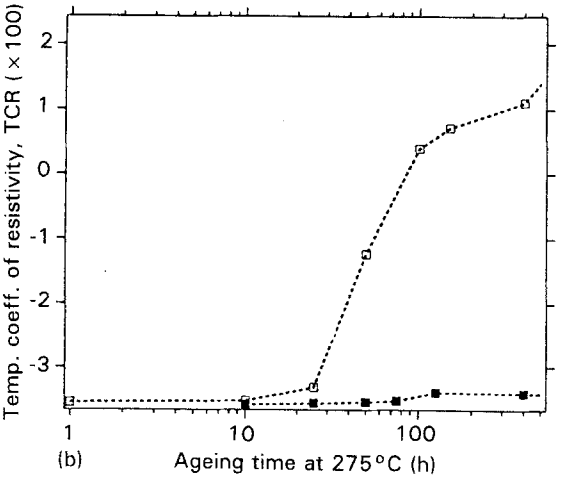


Fig. 5 a,b

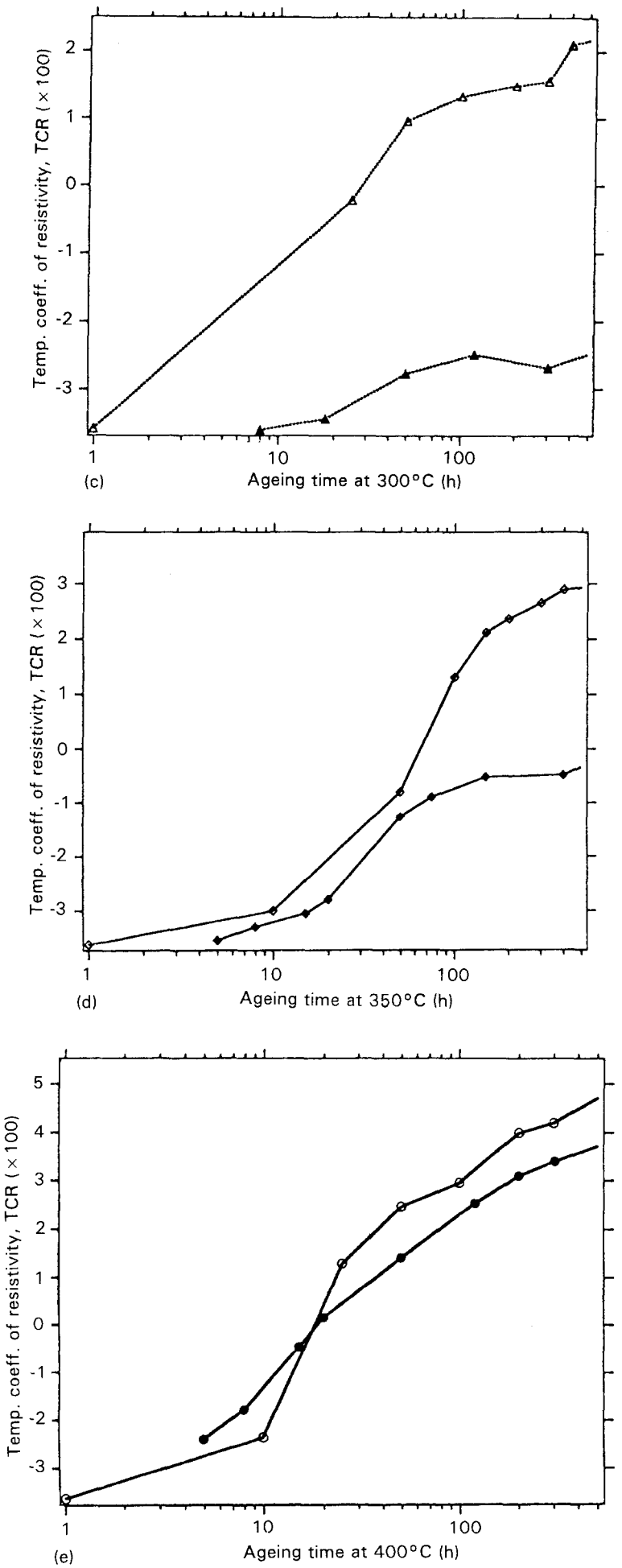


Figure 5 Comparisons of the temperature coefficient of electrical resistivity (TCR) of the Ti–15–3 alloy due to (⧗, □, △, ◇, ○) ageing, with (⧗, ■, ▲, ◆, ●) the values resulting from up-quench (reversion) treatments at 500 °C after ageing for varying times at several temperatures: (a) 250 °C, (b) 275 °C, (c) 300 °C, (d) 350 °C, (e) 400 °C.

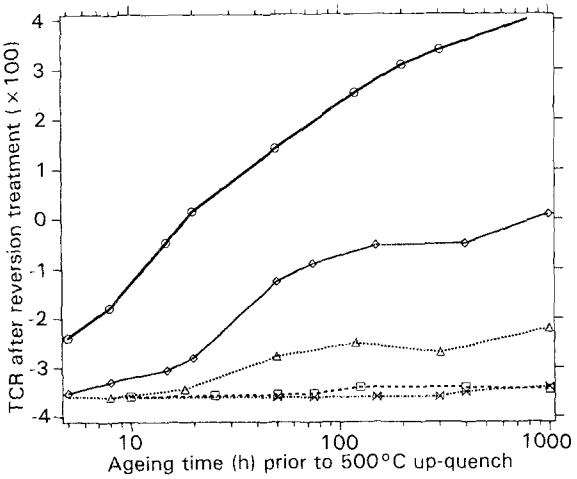


Figure 6 Comparison of the effects of different ageing temperatures and prior ageing times on the values of TCR after reversion treatment at 500 °C (up-quenching). (⋈) 250 °C, (□) 275 °C, (△) 300 °C, (◇) 350 °C, (○) 400 °C.

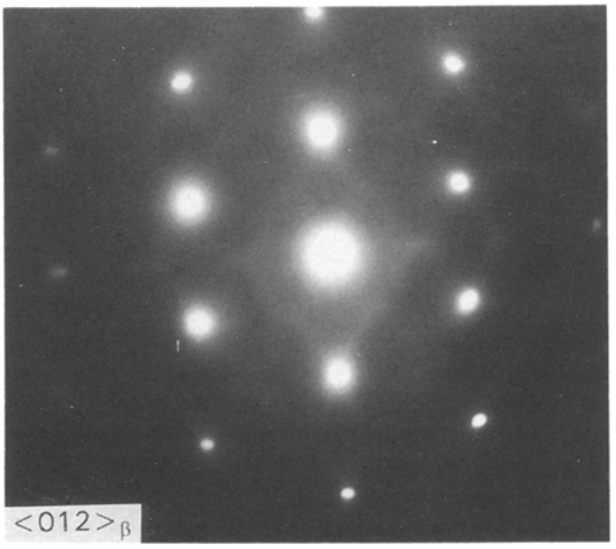


Figure 7 $\langle 0\,1\,2\rangle_\beta$ selected-area electron diffraction pattern (SAD) from the Ti–15–3 alloy after quenching from 900 °C and ageing for 100 h at 250 °C, showing diffuse streaks.

occurred during initial ageing below 400 °C; no precipitate phases were detected by X-ray analysis or TEM during the incubation period. The incubation period decreased with increasing ageing temperature and was characterized by constant hardness values, as shown in Fig. 2 and steady values of the TCR, as shown in Figs 4 and 5a–e. Continued ageing beyond the incubation period resulted in increases in both the hardness and the value of TCR, as shown in Figs 2, 4 and 5. X-ray and TEM examination confirmed the progressive formation of the ω and/or α phases, depending on the ageing time and temperature, as indicated in Table II. The ω reflections were first observed after 100 and 120 h during ageing at 275 and 250 °C, respectively; pronounced increases in the TCR values were detected after 25 h at 275 °C and 40 h at 250 °C (Figs 2, 3, 4). These results indicate the value of the TCR is sensitive to minor modifications of microstructure that occur during the initial stages of β phase decomposition in titanium alloys; furthermore, the

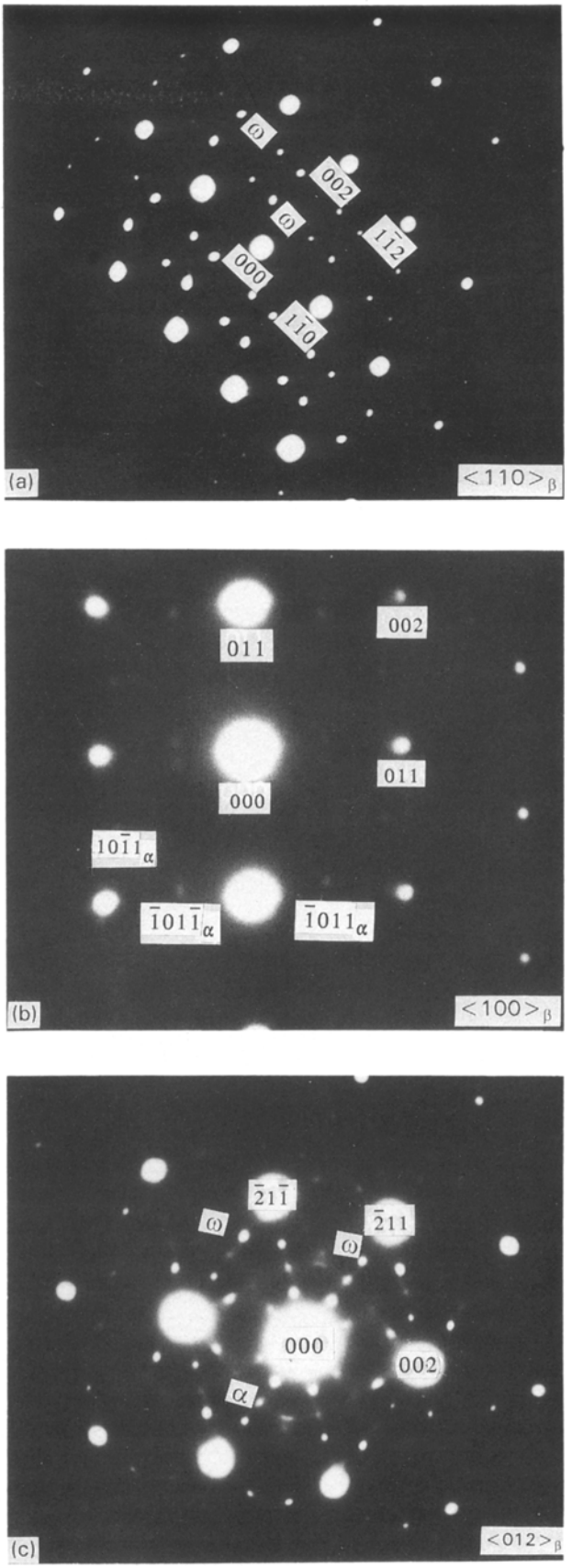


Figure 8 SAD patterns from a specimen aged 200 h at 250 °C showing reflections from the ω and α phases. (a) $\langle 1\,1\,0\rangle_\beta$ SAD pattern showing ω reflections. (b) $\langle 1\,0\,0\rangle_\beta$ SAD pattern showing α reflections from the same specimen as (a). (c) $\langle 0\,1\,2\rangle_\beta$ SAD pattern showing both α and ω reflections from the same specimen as (a).

results show that TCR measurements permit the detection of precipitation prior to confirmation by X-ray and TEM techniques.

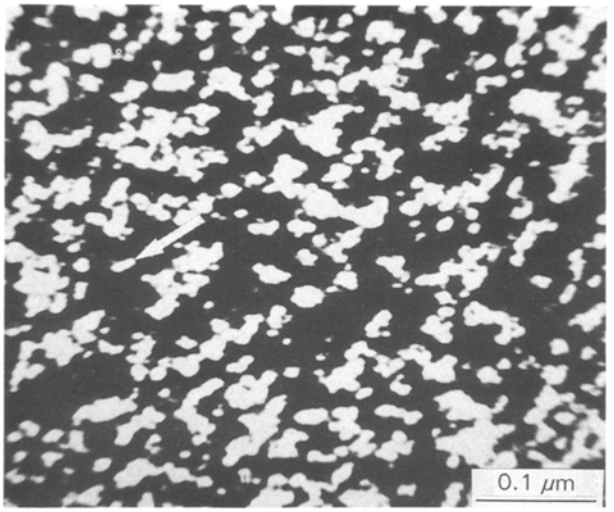


Figure 9 Dark-field micrograph from the Ti–15–3 alloy after ageing 200 h at 250 °C, showing the ellipsoidal morphology of the ω phase.

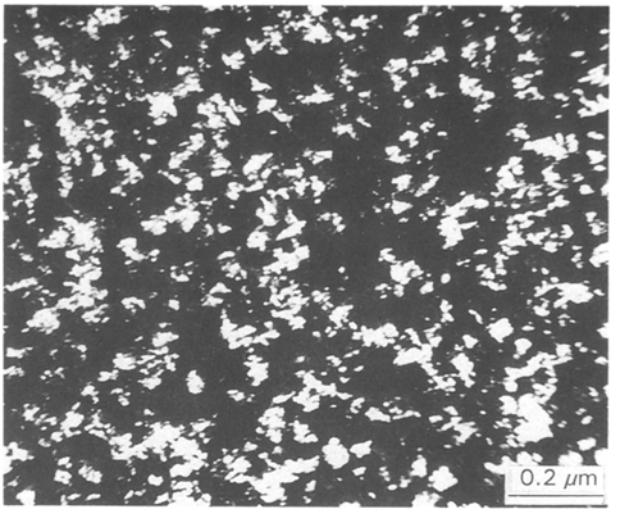
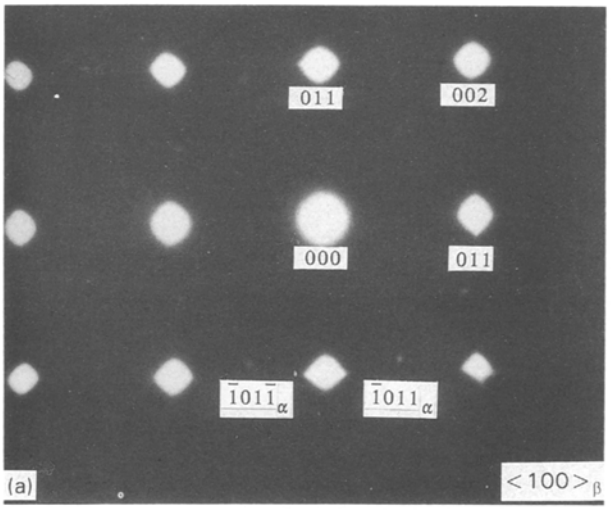


Figure 11 Dark-field micrograph showing the clusters of α platelets in the Ti–15–3 alloy after ageing at 300 °C for 20 h.



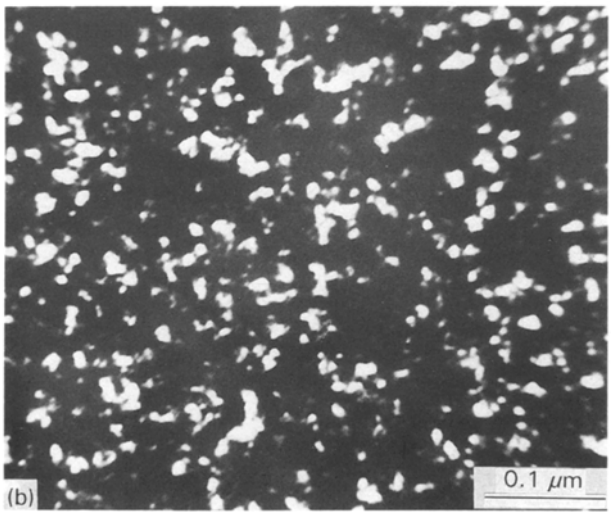


Figure 10 α phase formation in the Ti–15–3 alloy after ageing at 275 °C for 150 h. (a) $\langle 1\,0\,0\rangle_\beta$ SAD pattern exhibiting two variants of $\langle 1\,\bar{2}\,0\rangle_\alpha$. (b) Dark-field image taken from a $\{11\bar{2}0\}_\alpha$ reflection showing the morphology and distribution of the α phase.

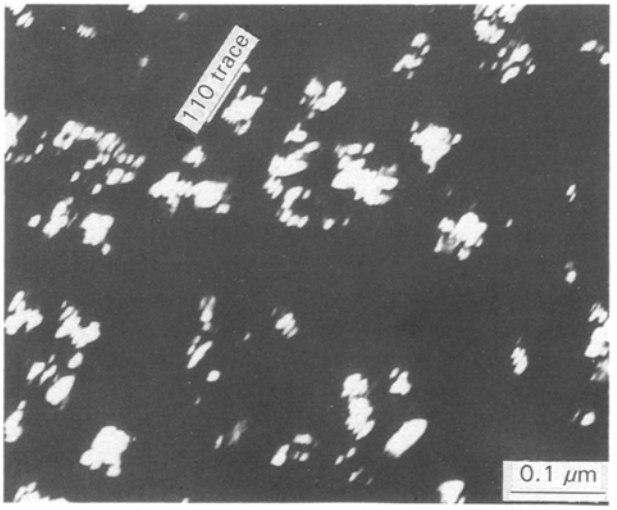


Figure 12 Dark-field micrograph obtained by imaging a $\{01\bar{1}1\}_\alpha$ reflection in a specimen aged at 300 °C for 30 h and up-quenched to 500 °C for a 2 min reversion treatment; note stacks of α platelets.

The decomposition of the β phase during ageing in the Ti–15–3 alloy proceeds by the precipitation of the ω and α phases, depending on the time and temperature of treatment, as summarized in Table II. ω phase is formed during the first stage of precipitation at 250, 275 and 300 °C. α precipitation required more than 200 h at 250 °C, whereas less than 10 h were required at 300 °C. The ω reaction was overshadowed by α precipitation within 8 h at 350 °C and ω precipitation was not detected after ageing at temperatures above 350 °C. The precipitation of the ω and α phases was accompanied by increasing values for the TCR, which changed from negative to positive, as shown in Fig. 4. The most rapid changes occurred for ageing above 300 °C where α precipitation was the predominant reaction.

Previous studies [4, 6, 17] reported that the ω phase in several titanium alloys can be reverted by up-quenching after ageing and holding briefly (e.g. 2 min) at a temperature above the ω range. This reversion reaction also occurs in the Ti–15–3 alloy, as indicated by significant decreases in both the hardness and the TCR values of aged specimens after up-quenching to 500 °C for 2 min. Complete reversion of the hardness and TCR to the original as-quenched values occurred

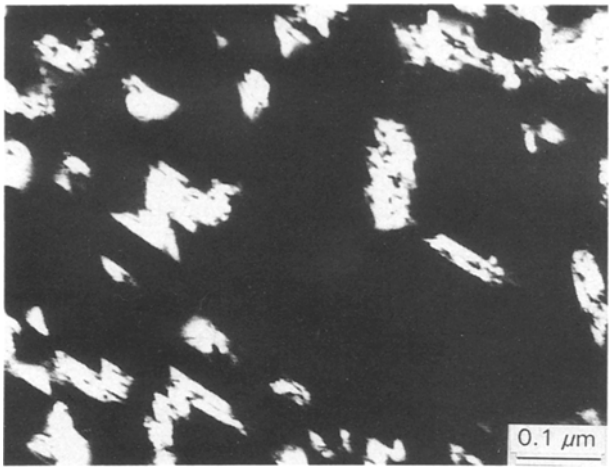


Figure 13 Dark-field micrograph obtained by imaging a $\{0\,1\,\bar{1}\,1\}_\alpha$ reflection in a specimen aged at 300 °C for 50 h and up-quenched to 500 °C for a 35 min reversion treatment; note coarsening of α plates compared to Fig. 12.

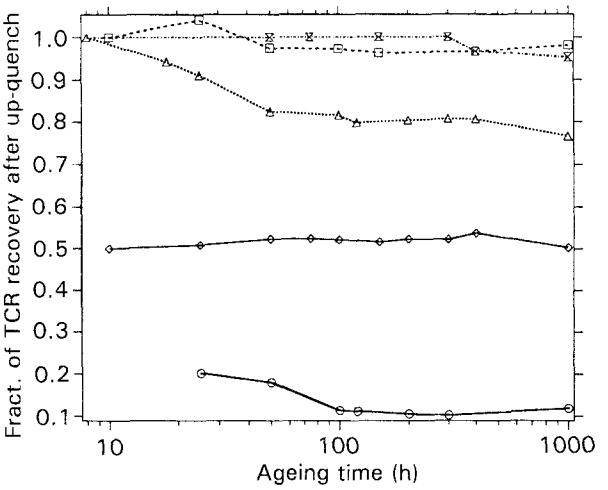


Figure 14 The fraction of the TCR increase due to prior ageing that is recovered by a 500 °C up-quench for 2 min; i.e. the fraction of TCR reversion for various ageing times and temperatures over the range 250–400 °C: (⧖) 250 °C, (□) 275 °C, (△) 300 °C, (◇) 350 °C, (○) 400 °C.

in specimens containing only the β and ω phases, such as those previously aged at 250 °C (see Figs 3a–c and 5a–e). The TCR value did not return to the quenched value in specimens that were up-quenched after ageing to an intermediate stage where α precipitation had occurred. For example, as shown in Fig. 5c, specimens aged longer than 40 h at 300 °C, which contain both the ω and α phases, showed a definite difference in the TCR value compared to the original as-quenched condition. The TCR value after reversion treatments was partially reduced, but did not recover the original quenched value for increased ageing times and temperatures, as shown in Figs 5c–e and 6. The reversion of the TCR values after up-quenching to 300 °C is consistent with the hardness changes for the same treatments shown in Figs 3a–c.

Plausible explanations for the reversion behaviour include the following.

(a) Full recovery of the as-quenched TCR value after reversion treatment requires complete dissolution of ω and α precipitates.

(b) Partial recovery of the as-quenched TCR value results when the ω and α precipitates are incompletely reverted by the up-quenching treatment.

(c) The existence of high densities of remnant solute lean zones in the β phase following ω reversion can influence the value of TCR.

Differences in the reversion behaviour of titanium alloys have been reported in previous investigations [4, 18]. Boyer *et al.* [18] reported incomplete reversion of the ω phase in the β-III alloy after up-quenching to 550 °C for 300 s. Chandrasekaran *et al.* [4] reported total reversion of the ω phase in a Ti–15% Cr alloy after 300 s at 500 °C; remnant solute-lean zones remained after the reversion process. The difference in reversion behaviour between these two alloys was attributed to a variation in the lattice misfit between the ω and β phases in the two systems.

In the present investigation, residual ω phase and solute-lean zones were not detected by either TEM or X-ray diffraction analysis in the Ti–15–3 alloy subjected to ageing and reversion treatments. The failure of this alloy to recover the original as-quenched hardness and TCR values after some reversion treatments was consistently associated with the presence of the α phase. Stable α phase precipitates are expected to grow rather than revert in this alloy at 500 °C; therefore, while metastable ω particles can revert to β, the α precipitates would remain rather than revert, if their initial sizes satisfy the critical requirement for growth at 500 °C. The stability of the α phase during reversion treatment is illustrated in Fig. 12 for a specimen aged at 300 °C for 30 h, followed by 2 min at 500 °C; the α phase remains in the form of clusters of platelets after up-quenching.

If it is assumed that negligible amounts of the α phase undergo reversion due to up-quenching, the net contribution of ω phase precipitation to the total increase in TCR during the ageing treatment will be determined by the fractional recovery of the TCR value from that of the aged alloy prior to up-quenching. The results are shown in Fig. 14, a plot of the fractional TCR recovery as a function of the prior ageing time for several ageing temperatures; this plot provides an insight into the effect of ageing time and temperature on the relative involvement of ω phase precipitation in the decomposition of the parent beta phase. Fig. 14 indicates almost complete reversibility of TCR during ageing at 250 °C, suggesting β decomposition is primarily due to the precipitation of the ω phase and α precipitation is negligible up to 400 h; however, α formation becomes increasingly important for longer ageing times at 250 °C. Fig. 14 shows ~0.5 fractional recovery of TCR during ageing at 350 °C, suggesting co-precipitation of ω phase and α phase occurs during the first 1000 h. The relative contributions of ω and α formation are unclear from the TCR data for ageing at 350 °C, because the TEM observations suggest a predominance of α phase formation beyond 30 h. The results also show that ageing at 400 °C is above the temperature range for

significant ω formation; from Fig. 14 the fractional recovery of TCR is less than 0.2 prior to 50 h ageing and decreases with prolonged ageing, thereby suggesting α precipitation is predominating even at early ageing times.

On the basis of comparing the TCR data with TEM observations, changes in the TCR appear to be less sensitive for detecting the start of α precipitation than for ω formation. For example, TEM results revealed the predominance of α precipitation after 50 h ageing at 300 °C and 30 h at 350 °C, whereas the TCR data suggest ω formation prevails for these same ageing conditions. The fact that precipitation of the ω phase is the initial stage of β decomposition below 400 °C accounts for the apparent difficulty in identifying the separate contribution of the α phase by monitoring changes in TCR. The negativity of TCR has been established as a characteristic of the metastable β phase over a limited composition range. The most dramatic changes in TCR occur at the beginning of β decomposition when perturbations leading to ω formation develop in the parent β phase at the lower ageing temperatures. Consequently, TCR is most useful as a parameter for monitoring the initiation and early stages of β phase decomposition, especially at low ageing temperatures where ω phase is the likely reaction product.

5. Conclusions

1. The TCR of the Ti–15–3 alloy is negative if the alloy is in the 100% β condition, consistent with the results of earlier studies suggesting that this anomoly is a characteristic of the β phase over limited ranges of alloy composition.

2. The value of TCR increases from negative to positive values with the precipitation of the ω and α phases during β decomposition. The value of TCR remains essentially unchanged from that of the quenched condition during the initial stages of ageing; the transient period decreases with increasing ageing temperature. Changes in the TCR were detected before any definite evidence of precipitation was obtained by diffraction techniques.

3. During ageing at temperatures below 300 °C the initial precipitation process involved ω formation, thereby pointing to the sensitivity of TCR as a parameter for detecting the initiation of instability in the β phase during prolonged exposure above room temperature. At temperatures above 300 °C changes in the TCR value also reflect the precipitation of the α phase, which becomes the predominant reaction with increasing temperature.

4. Reversion of the ω phase in aged alloys by up-quenching causes reversal of the TCR values, approaching those for the quenched condition. The fractional recovery of TCR due to reversion depends on the amount of α phase formed during the ageing treatment; in the absence of α precipitation it was possible to completely recover the quenched values for both hardness and the negative TCR.

5. TCR determinations are more sensitive than conventional TEM or diffraction techniques for monitoring the initial stages of β decomposition when ω phase is the product; however, changes in TCR are less effective in detecting the initial precipitation of α phase unless the ageing reaction is carried out at temperatures above the ω formation range.

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