# **Strength enhancement in a metastable 13-titanium alloy: Ti-15Mo**

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The tensile properties of a metastable  $\beta$  alloy, Ti-15Mo<sup>\*</sup>, in a number of different aged conditions have been correlated with the microstructures observed by TEM. Ageing at a temperature, which is approximately the upper limit of  $\omega$  phase stability, allowed the  $\alpha$ and  $\omega$  phases to coexist, the  $\alpha$  phase apparently having been nucleated on the  $\omega$  particles. An extremely fine distribution of the  $\alpha$  phase was achieved in this way, resulting in increased strength, limited ductility and good toughness. This method of precipitate refinement is then compared with the other treatments which have been applied to metastable  $\beta$ -titanium alloys. Changes in strength due to variations in heating rate to the ageing temperature, to simulate the effect of section size, are also reported and discussed in terms of the refinement in  $\alpha$  precipitate size. It is shown that these refinements in precipitate size are reflected in higher values of the ratio 0.2% proof stress/Young's modulus.

### **1. Introduction**

Metastable  $\beta$ -titanium alloys offer a wide range of strength and ductility. In the solution treated condition (e.g.  $\beta$  quenched) the alloys are usually single phase and possess low strength and high ductility. Ageing at relatively low temperatures (250 to  $450^{\circ}$  C, depending on composition) to precipitate the  $\omega$  phase increases the strength and decreases the ductility, often drastically. Ageing at higher temperatures  $(>500^{\circ} C)$  results in the direct precipitation of the  $\alpha$  phase, with intermediate strengths and limited ductilities. Typical tensile properties for such an alloy, Ti-15Mo, are shown in Table I. Similar trends can be observed for other metastable  $\beta$ -titanium alloys, both commercial  $[3-12]$  and non-commercial  $[4, 5, 11, 13-16]$ .

Because of the problems of brittleness due to the  $\omega$  phase and its stability, these alloys are normally used in the  $\beta + \alpha$  condition, and the  $\alpha$ phase is usually coarse (Fig. 1). Recently, attempts have been made to refine the  $\alpha$  needle size by thermo-meehanical treatments [5, 11], multi-stage heat treatments  $[5, 7, 11, 12]$ , and by combinations of these two processes  $[5, 11]$ . This paper reports a further method of  $\alpha$  phase refinement, observed during an investigation of the fracture  $^*$ i.e. Ti $-15$  wt  $\%$  Mo.

toughness of  $Ti-15Mo$   $[1]$ .

# **2. Experimental**

Tensile tests were carried out on a forged bar prepared from a 9kg laboratory melt of Ti-15Mo (composition in wt %: 15.57 Mo, 0.04 Fe, 0.02 C, 0.0015  $H_2$ , 0.125  $O_2$ , 0.011  $N_2$ ). Test piece



*Figure 1* TEM of Ti-15Mo, solution treated at 800°C (1h), AC and aged at  $560^{\circ}$  C (4h), AC.

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TABLE I Mechanical properties of Ti-15Mo bar: Effect of ageing temperature [1]

Heat Treatment	0.2% Proof Stress $\sigma_{\mathbf{y}}$	Tensile Strength $\sigma_{\bf u}$	$E_{\rm{c}}$ (GPa)	$\sigma_{\bf v}/E$ $(X 10^2)$	Elong.	Uniform Red in $K_{\text{IC}}$ Elong.	Area	$(MPa m^{1/2})$
	(MPa)				(%)			
$800^{\circ}$ C air $\text{cool AC}^*$	515	799	77	0.68	29.5	24	58	$-1$
$800^{\circ}$ C (1h) AC $+250^{\circ}$ C (100h) AC		1124	115	$0.98^{+}$	7.5	$\mathbf{0}$	33	42.1
$800^{\circ}$ C (1h) AC $+350^{\circ}$ C (18h) AC		1214	123	$0.99^+$	$\overline{0}$	$\Omega$	$\mathbf 0$	22.2 <sup>§</sup>
$725^{\circ}$ C (1h) AC $+475$ °C (16h) AC	1279	1300	111.5	1.15	0.3	0.2	5	52.6
$800^{\circ}$ C (1h) AC $+525^{\circ}$ C (16h) AC	1059	1123	106	1.00	2.5	0.7	11	72.0
800° C (1h) AC $+560^{\circ}$ C (4h) AC	1026	1080	104.5	0.98	7.5	3.7	16	85.5

large grain size in sheet  $[2]$ 

t very high toughness – Charpy impact value ~ 153 J [2]

- $\sigma_{\rm u}/E$
- $\int x_Q$

blanks were solution treated in the  $\beta$  (800°C) and  $\alpha + \beta$  (725<sup>°</sup>C) fields in a flowing argon atmosphere, air cooled, and subsequently aged in air ovens or salt baths at temperatures between 250 and  $560^{\circ}$  C. Electropolished test pieces,  $4 \text{ mm}$  in diameter and 20 mm gauge length, were pulled to fracture in an Instron machine at a cross-head rate of  $1.7 \times 10^{-3}$  sec<sup>-1</sup> at 20°C. Load-elongation curves were obtained on an  $X-Y$  plotting table which was linked to the load cell of the Instron and to an extensometer clamped to the test piece. Foils for TEM, prepared from strip milled from the heat treated blanks, were thinned at  $-25^{\circ}$  C and examined in a JEOL 200 A electron microscope at 200 kV.

# **3. Results**

#### 3.1 Microstructures

The  $\alpha$  phase distribution in  $\beta + \alpha$  alloys, aged at 560°C in the present case, is shown in Fig. 1. Some refinement in  $\alpha$  needle size can be achieved by decreasing the ageing temperature to  $525^{\circ}$ C (Fig. 2), but this precipitate is still relatively coarse. In the present work, however, it has been found that ageing at a temperature which approximately corresponds with the upper temperature limit of  $\omega$  phase stability (in this case 475°C) produces very considerable  $\alpha$  phase refinement (Fig. 3). This is more clearly seen in dark field (Fig. 4). At higher magnifications (Fig. 5) it can be seen that the  $\omega$  and  $\alpha$  phases coexist, both in an

ellipsoidal form (which is the usual shape for  $\omega$ particles in this alloy system  $[17-19]$ ). The important feature of this figure is the apparent nucleation of  $\alpha$  on the  $\omega$  particles (e.g. A Fig. 5).

#### 3.2. Tensile results

The strength and modulus values resulting from the above heat treatments, and for low temperature ageing treatments where  $\beta + \omega$  exist, are shown in Fig. 6. This figure shows clearly that ageing at  $475^{\circ}$ C improves the strength and the  $\sigma_{\rm v}/E$  ratio. The ductility values associated with this aged condition are given in Table I. Although these are low, the  $K_{IC}$  value for this condition was 52 MPa  $m^{1/2}$  (Table I). This is similar to values reported by Rogers for this alloy [20], and to values for other metastable  $\beta$ -titanium alloys  $[1, 21 - 26]$ .

#### **4. Discussion**

The important observations made during this investigation were the reduction in  $\alpha$  precipitate size, which resulted from ageing near the upper temperature limit of  $\omega$  phase stability, and the consequent increase in strength.

The ratio  $\sigma_{\mathbf{v}}/E$  has been found to be useful in assessing strengthening effects due to precipitation in Ti- 15Mo, but, with the exception of the work of Sargent *et al.* [9, 10], this ratio has not been used in other investigations on metastable  $\beta$ -titanium alloys. If the ratio remains constant with ageing



*Figure 2* TEM of Ti-15Mo, solution treated at 800° *C Figure 3* TEM of Ti-15Mo, solution treated at 725° C (1b), AC and aged at 475° C (16h), AC.



(1h), AC, and aged at  $475^{\circ}$  C (16h), AC.



*Figure 4* Dark Field electron micrograph of Fig. 3;  $\alpha$ phase operating reflection.

temperature and time, then it can be concluded that precipitation is contributing equally to both parameters; if there is a change in the ratio, one parameter is affected more than the other. E will be affected mainly through the volume fraction of precipitate(s), but  $\sigma_{\mathbf{y}}$  through the volume fraction and precipitate(s) size and spacing. Thus if the volume fractions of  $\omega$ ,  $\omega + \alpha$  or  $\alpha$  are not very different (in the present case they are all  $\sim 0.5$ [1]), then the reduced size of the  $\alpha$  phase, as shown in Fig. 5, must be the cause of the en-



*Figure 5* TEM of Ti-15Mo, solution treated at 725 $^{\circ}$  C (1h), AC, and aged at  $475^{\circ}$  C (16h), AC. Apparent nucleation of  $\alpha$  phase on  $\omega$  particles, e.g. A.

hanced  $\sigma_{\mathbf{v}}$  and  $\sigma_{\mathbf{v}}/E$  values shown in Fig. 6. Since this is the first report of strengthening metastable  $\beta$ -titanium alloys by this method, it is pertinent to relate this observation to other published work. Sargent *et al.* [9, 10] noted a similar change in  $\sigma_{\mathbf{v}}/E$  for  $\beta$  solution treated and aged  $\beta$ -III titanium (Ti-11.5 Mo-5.5 *Zr-4.5* Sn); *ay/E* was constant at  $0.9 \times 10^{-2}$  for ageing temperatures  $\leq 427$  °C, but increased to  $\sim 1.1 \times 10^{-2}$  for an ageing temperature of  $\sim$  480 $\degree$  C. The authors however did not attach any significance to it. Unfortunately, no



*Figure 6* Effect of ageing temperature on the 0.2% proof stress, Young's modulus and proof stress/modulus ratio of a solution treated and aged Ti-15Mo bar (strength values for the  $250$  and  $350^{\circ}$  C aged conditions are tensile not proof stresses: see Table I).

relevant microstructural evidence was presented, but other work on  $\beta$ -III titanium suggests that the  $\omega$  and  $\alpha$  phases do coexist at about 480°C for  $\beta$ solution treated material (Feeney and Blackburn [3] report it as low as  $430^{\circ}$  C and Kalish and Rack [6] at  $480^{\circ}$  C). It is reasonable to conclude, therefore, that a fine distribution of precipitates also resulted in the increase in  $\sigma_{\mathbf{v}}/E$  ratio recorded by Sargent *etal.* [9, 10].

Williams [19], in a recent review, has commented upon the type of precipitate for ageing temperatures near the upper limit of  $\omega$  phase stability, and on the possibility of this increasing strength. The present work, and that of Sargent *et al.* [9, 10], would appear to offer experimental proof that such improvements in strength can be achieved.

Other published work is more difficult to discuss because most results do not include modulus values. The few results which can be considered include those for  $\beta$  solution treated and aged  $\beta$ -III, where Kalish and Rack  $[6]$  observed a large decrease in vield strength on ageing above  $480^{\circ}$ C (the temperature at which they observed  $\omega$  and  $\alpha$ ), while Feeney and Blackburn [3] found maximum strength between 370 and 480 $^{\circ}$  C and  $\omega$  and  $\alpha$  present at 430° C. For  $\alpha + \beta$  solution treated and aged  $\beta$ -III Sommer, Ono *et al.* [5, 11] found  $\omega$ and  $\alpha$  coexisting as low as 370°C, with a plateau in tensile strength near this temperature. Although it is difficult to compare the above data, it is apparent that good combinations of strength and ductility can be achieved in metastable  $\beta$ -titanium alloys, particularly when  $\omega$  and  $\alpha$  coexist. It is also apparent that the optimum solution treatment and ageing temperatures (and times) have not yet been achieved in these alloys; hence further improvements in strength, coupled with adequate ductility and toughness, should be possible when the kinetics of precipitation are studied more fully.

Some comments are now made regarding the relative merits of strengthening by the present method, and those of other processes which have been tried to date. These include:

(a) Multiple Ageing: It was suggested earlier [13] that the degree of improvement in properties, by duplex ageing to initially precipitate the  $\omega$  phase prior to  $\alpha$  phase formation, depended on the shape of the  $\omega$  particles. Thus systems containing cubic  $\omega$  particles, because of their higher misfit energy  $[17-19, 27, 28]$ , were expected to show a refined  $\alpha$  phase distribution and higher strength (e.g.  $Ti-V$  alloys  $[15]$ ), while those containing ellipsoidal particles were not (e.g. Ti-Mo alloys [13]). The present work has shown, however, that the earlier conclusion resulted from work carried out on material aged below the maximum temperature for  $\omega$  phase stability (i.e.  $\lesssim$  $450^{\circ}$  C). It is now clear that near this maximum temperature, refinement of the  $\alpha$  phase will occur *irrespective* of the shape of the  $\omega$  particles, and therefore single ageing treatments can result in similar properties to those of duplex treatments. The work of Sommer, Ono *et al.* [5, 11] is of interest in this respect: they found that the advantage of the initial ageing treatment, in a duplex ageing treatment, was a much reduced time at the second (higher) ageing temperature. (No increases in strength were observed except for a slight increase on a second ageing treatment at  $430^{\circ}$  C). Unfortunately, the second ageing treatments were not carried out as low as  $370^{\circ}$  C, where they showed  $\omega$  and  $\alpha$  to coexist. The

Heat Treatment	0.2% Proof Stress $\sigma_{\mathbf{y}}$	Tensile Strength $\sigma_{\bf u}$	Е (GPa)	$\sigma_{\rm v}/E$ $(X 10^2)$	Elong.	Uniform Elong.	Red in Area	Time to ageing temp
	(MPa)			$(\%)$			(min)	
$800^{\circ}$ C (20m) AC $+560^{\circ}$ C (4h) AC	940	1006	100	0.94	10	5	32	1
$800^{\circ}$ C (20m) AC $+560^{\circ}$ C (4h) AC	1001	1071	104	0.97	13	5	22	375
$800^{\circ}$ C (20m) AC $+525^{\circ}$ C (16h) AC	1044	1045	102	1.02	6.5	$\mathbf{2}$	20	1
$800^{\circ}$ C (20m) AC $+525^{\circ}$ C (16h) AC	1062	1135	107	0.99	9.1	5	24	315
$725^{\circ}$ C (40m) AC $+475^{\circ}$ C (16h) AC	1209	1215	104	1.16	2.9	$\mathbf{2}$	15	1
$725^{\circ}$ C (40m) AC $+475$ °C (16h) AC	1333	1340	116	1.15	4.2	$\mathbf{2}$	20	270

TABLE II Tensile properties of  $Ti-15M\sigma$  sheet: effect of heating rate to the ageing temperature\*

Experimental details in  $[2]$  and  $[13]$ 

properties of such a treatment appear very attractive, since they could be achieved in a very short time.

(b) Reversion Treatments: These have been carried out by Boyer *et al.* [7, 12]. Here the  $\omega$ phase was allowed to form at  $\sim$ 350°C and the temperature subsequently raised to  $\sim$  500 $^{\circ}$  C for short times to allow partial reversion of the  $\omega$ phase. No  $\alpha$  phase was observed. This is a useful technique but unfortunately involves very long ageing times at  $350^{\circ}$  C and very short ageing times at 500°C. Phase stability may therefore be a problem.

(c) Thermo-Mechanical Treatments: Very high strengths can be achieved in this way, particularly if the treatments are combined with multistage ageing  $[5, 11]$ . Deformation prior to reversion has also been studied, which also further improved strength [7, 12]. However, such treatments have the disadvantage of being readily applicable only to fiat plate or sheet.

Thus although the maximum strength may not be achievable by the single ageing treatment suggested here, it has the distinct advantages of simplicity and independence of component shape. Clearly, a rigorous comparison of these different methods is needed on a single alloy before definitive conclusions can be drawn.

It should also be remembered that the time to reach the ageing temperature can also influence the strength levels obtained in metastable  $\beta$ -titanium alloys. For instance, Table II contains some data for three aged conditions of Ti-15Mo sheet. In the first set the test pieces were placed in the salt bath or oven at the ageing temperature,

while in the second set the test pieces were placed in a cold oven and then heated to the ageing temperature. The times to the ageing temperature are given in the table. It can be seen clearly that the slow heating rate, which allowed precipitate nucleation during the time taken to reach the ageing temperature, resulted in higher strengths with little or no effect on ductility. Table II also shows that the  $\sigma_{\rm v}/E$  ratio is increased only for high ageing temperatures (where  $\alpha$  is precipitated directly), and where refinement of the precipitate size results from slow heating rates. Ageing at 475° C, irrespective of the heating rate, will produce a refined precipitate size. The relevance of the trend shown by this data is that it simulates the slow heating rate to the ageing temperature at the centre of very thick section components. Hence the implications of such an increase in strength, particularly if there is a decrease in ductility due to the predominance of plane strain conditions, must be considered in the use of these alloys. Furthermore, the data in Table II also suggests that a direct read across of data from thin sheet to thick section bar, for the same heat treatment, should be treated with caution.

#### **5. Conclusions**

Tensile tests on a solution treated and aged metastable  $\beta$ -titanium alloy, Ti-15Mo, have shown that:

(a) A choice of an ageing temperature near the maximum temperature limit for  $\omega$  phase stability results in the coexistence of the  $\omega$  and  $\alpha$  phases. Precipitation apparently occurs on  $\omega$  particles, resulting in a very fine distribution of the  $\alpha$  phase.

(b) Increased strength results from ageing at such a temperature, which for Ti-15Mo solution treated at  $725^{\circ}$ C was  $475^{\circ}$ C. The increased strength was particularly noticeable in the  $\sigma_{\rm v}/E$ ratio, a ratio which is thought to be useful in assessing strengthening due to precipitation in metastable  $\beta$ -titanium alloys.

(c) The increased strength was accompanied by limited ductility and good fracture toughness.

(d) Refinement in precipitate size was also achieved by slow heating rates to the ageing temperature. Since these rates are similar to those at the centre of thick section components, caution must be exercized in any comparison between thin and thick section component data for this or similar alloys.

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