

The effect of step-quenching on the tensile properties and microstructure of the titanium alloy Ti-4Al-4Mo-2Sn-0.5Si (IMI 550)

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Step-quenching of the titanium alloy Titanium alloy Ti-4Al-4Mo-2Sn-0.5Si (IMI 550) from 900°C into salt at temperatures in the range 445 to 600°C produced tensile strength increases of up to 15% compared with the conventional heat treatment. Material quenched to 500°C or below had a different microstructure from that produced either by quenching to higher temperatures or by solution treatment, air cooling to room temperature and ageing at 500°C. This was related to the formation of martensite with M_s (Martensite start) temperature in the range 500 to 525°C. The orthorhombic martensite reported in the bulk material was not found in thin foils since reversion of the orthorhombic phase to bcc β occurred during the foil thinning process.

1. Introduction

A common application of step-quenching (SQ) is during the austempering or martempering of steels. These treatments involve a quench to a temperature above the martensite start temperature M_s rapid enough to avoid the ferrite formation reaction, and then either an isothermal transformation to lower bainite or a slow cool through the M_s to M_F range to avoid the formation of martensite. In this way a fine, deep-hardening microstructure is produced without quench cracking or embrittlement.

The formation of martensite by quenching titanium alloys is often associated with some degradation of tensile properties; in particular with a loss in ductility. The behaviour of titanium alloys is somewhat similar to that of steels in this respect, and SQ of several high-strength titanium alloys has been investigated [1-3] which showed that for one alloy, IMI 550, tensile strength could be increased by 12 to 19% by SQ instead of solution treating, air cooling and ageing which is the standard heat treatment

for this alloy. This paper describes the effect of SQ on the tensile properties and microstructure of IMI 550.

2. Experimental procedure

100 mm diameter round bar was forged to 50 mm thick plate at an initial forging temperature of 920°C. Test-piece blanks 3 mm × 13 mm × 75 mm were cut from the plate with the long axis parallel to the original axis of the bar. Two of the blanks were then given the standard STA treatment i.e. solution treated at 900°C for 0.5 h, air cooled to room temperature and aged at 500°C for 24 h. The remaining blanks were solution treated at 900°C for 0.5 h and step-quenched into salt at 445, 475, 500, 525, 550, 575 or 600°C, soaked for 4 h and air cooled. (It had previously been shown [2] that a 4 h ageing treatment was sufficient to develop the full properties.) After heat treatment the test-piece blanks were machined into 3/8 in. (9.5 mm) BSF round tensile test-pieces, which were tested at a strain rate of 0.003 min⁻¹ (two test-pieces per heat-

TABLE I The effect of quench temperature on the tensile properties of IMI 550 (mean of two tests)

| | Quench temperature (°C) | | | | | | | STA |
|------------------------------|-------------------------|------|------|------|------|------|------|------|
| | 445 | 475 | 500 | 525 | 550 | 575 | 600 | |
| Tensile strength (MPa) | 1260 | 1294 | 1256 | 1236 | 1218 | 1192 | 1152 | 1126 |
| 0.5% proof stress (MPa) | 1149 | 1189 | 1164 | 1156 | 1139 | 1127 | 1104 | 1058 |
| 0.2% proof stress (MPa) | 1062 | 1097 | 1089 | 1092 | 1081 | 1079 | 1066 | 1026 |
| 0.1% proof stress (MPa) | 1011 | 1050 | 1048 | 1060 | 1054 | 1061 | 1048 | 1006 |
| Young's modulus (GPa) | 113 | 113 | 114 | 115 | 116 | 114 | 115 | 113 |
| Total elongation (%) | 11.5 | 10.0 | 10.5 | 11.0 | 10.5 | 11.5 | 13.5 | 18.7 |
| Uniform elongation (%) | 3.3 | 3.3 | 3.5 | 3.3 | 3.7 | 4.0 | 5.5 | 6.5 |
| Reduction in area (%) | 45 | 37 | 47 | 44 | 43 | 47 | 36 | 50 |
| $d\sigma/d\varepsilon$ (GPa) | 51 | 47 | 41 | 32 | 27 | 18 | 18 | 20 |
| $\Delta\sigma$ (MPa) | 291 | 287 | 252 | 217 | 209 | 179 | 167 | 193 |
| Vickers Hardness (H_v) | 394 | 407 | 407 | 396 | 388 | 376 | 369 | 376 |

treated condition). The test-piece heads were used for Vickers hardness measurements with a 20 kg indenter load and the microstructures were studied optically and in the scanning electron microscope (SEM) after polishing and etching with Kroll's reagent (1% HF, 12% HNO₃, 87% H₂O). Thin foils were prepared for the transmission electron microscope (TEM) using a jet technique with an electropolish solution of 500 ml methanol, 350 ml butoxyethanol and 60 ml perchloric acid at -20°C and 9 V.

3. Results

3.1. Tensile tests

The tensile properties after heat treatment are given in Table I. The work hardening rate $d\sigma/d\varepsilon$ and the total strain hardening $\Delta\sigma$ were calculated from the tensile results using

$$\frac{d\sigma}{d\varepsilon} = \frac{\sigma_{0.2} - \sigma_{0.1}}{0.001}$$

and

$$\Delta\sigma = \sigma_N(1 + \varepsilon_u) - \sigma_{0.1}$$

where σ_N is the nominal tensile strength based on the original test-piece cross-section and the maximum load in the tensile test, ε_u is uniform elongation to maximum load so that $\sigma_N(1 + \varepsilon_u)$ is the true stress at maximum load, and $\sigma_{0.1}$ is the 0.1% proof stress. The proof and tensile strengths and $\Delta\sigma$ are plotted in Fig. 1.

All the SQ treatments investigated produced a strength increase over the STA condition. This was accompanied by some loss in ductility, but with little effect on modulus. Fig. 1 shows that maxima in the 0.2 and 0.5% proof stresses (PS) and in tensile strength (TS) occurred for a

quench temperature of 475°C. The hardness values also followed this trend although the 0.1% PS varied little for quench temperatures above 475°C. The maxima in PS and TS were not reflected by a reverse trend in ductility; reduction in area remained roughly constant for the range of step-quench temperatures investigated, and both uniform and total elongation showed a slight increase for high quench temperatures. Both $d\sigma/d\varepsilon$ and $\Delta\sigma$ increased with decreasing quench temperature.

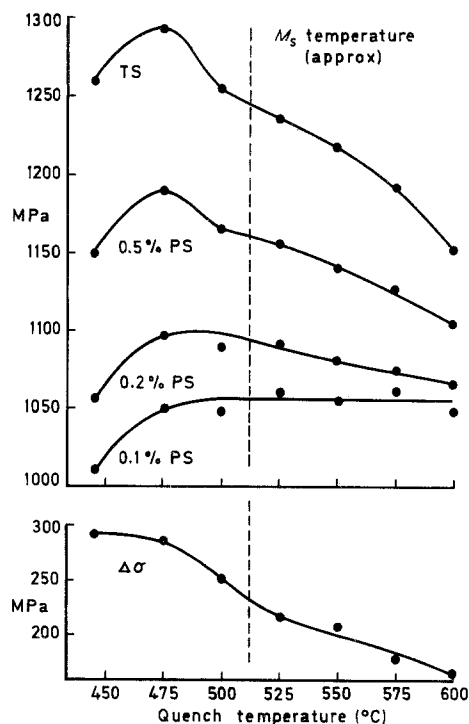


Figure 1 The effect of quench temperature on the tensile properties of IMI 550.

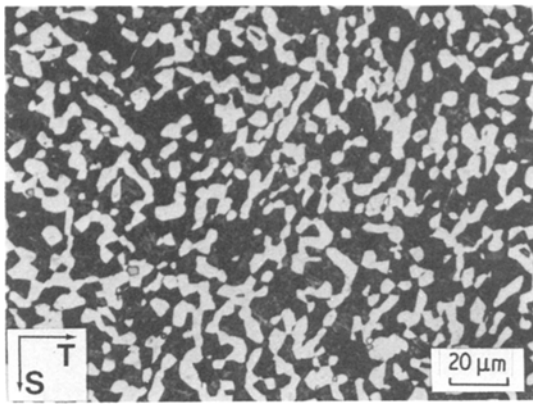


Figure 2 Microstructure of STA material.

3.2. Microstructure

The microstructure of the STA material (Fig. 2) showed approximately 50% primary α and 50% transformed β (light and dark regions respectively) with the substructure within the transformed β regions just visible. The material quenched to 445 or 475°C etched more slowly than the STA material under the same conditions, and the transformed β regions appeared relatively featureless, as in Fig. 3 (the orientations given in Figs. 2 and 3 refer to the original plate). For quench temperatures above 500°C the microstructures appeared similar to that of the STA material except that all of the step-quenched microstructures appeared to contain slightly less primary α than the STA microstructure and the substructure within the transformed β regions was not distinguishable optically in any of the step-quenched conditions.

The microstructures were then studied in the SEM. Fig. 4 is an electron micrograph of the

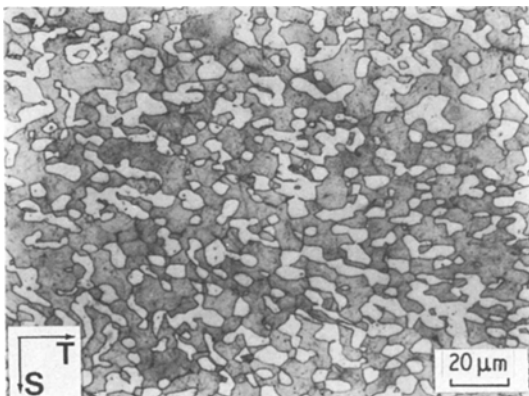


Figure 3 Microstructure of material step-quenched to 445°C.

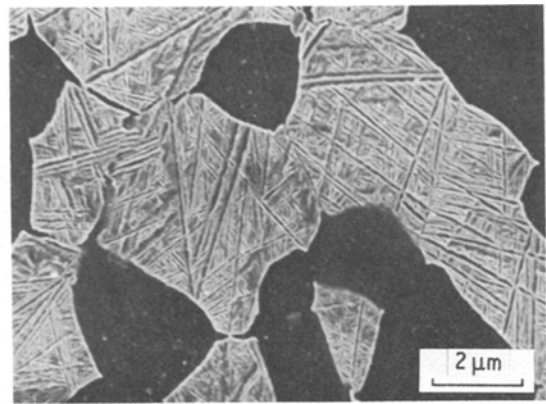


Figure 4 STA microstructure: scanning electron micrograph.

STA microstructure which showed thin secondary α plates within the transformed β regions. The transformed regions of the SQ microstructures appeared relatively featureless in the SEM, though traces of a plate-like microstructure similar to that found in the STA material were faintly visible.

3.3. Thin foil studies

Examination of the transformed β regions of the STA material revealed a fine interwoven α plate structure with β phase between the plates (Fig. 5). The corresponding areas in the material step-quenched to 525°C and above were very similar to the STA structure and no effect of quench temperature above 500°C on the transformation product within the prior β grains was apparent. However, material quenched to 500°C or below showed fine striations within the secondary α plates. These striations were resolved as narrow bands ~ 20 nm wide running approximately perpendicular to the long axis of the plates (Fig. 6).

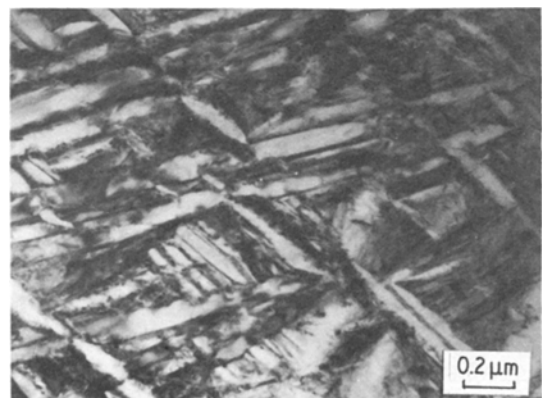


Figure 5 STA microstructure: transformed β region.

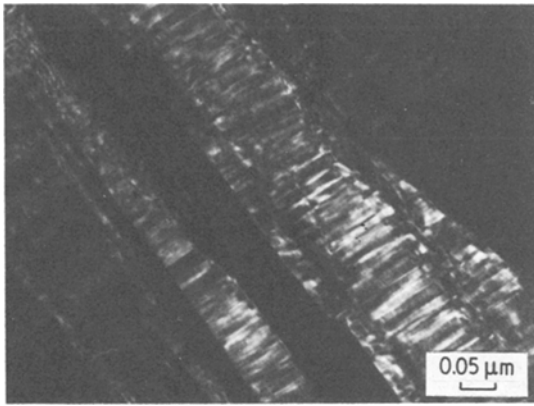


Figure 6 Step-quenched to 500° C: dark-field α phase diffraction.

The minimum size of selected-area aperture available was too large for α plates to be studied individually, so a focused electron beam was used to examine diffraction from small regions inside the striated plates. The beam diameter was ~ 50 nm, i.e. the diffracting area was several striations wide. This consistently produced α diffraction patterns with no evidence of any other phase or α phase twinning.

A trace analysis indicated that the striations lay in the basal planes of the α phase. This analysis was carried out using diffraction patterns from α plates in several different areas of the foil, and so was not restricted to one set of striations.

The Burgers orientation relationship between α and β phases was generally obeyed except that for material step-quenched to 500° C or below some twin-related α plate packets were found (Fig. 7) coexistent with the Burgers-related α .

4. Discussion

4.1. Mechanical properties

The purpose of the ageing process [4] in the STA treatment of IMI 550 is to produce a further fine precipitation of α (not easily visible in thin foils) which strengthens the alloy. Previous work on SQ [2] has shown that this ageing response is not enhanced by SQ. Furthermore, for quench temperatures of 525° C or higher the strength increase resulting from SQ rather than STA did not appear to be related to any refinement of the microstructure. Thus it is concluded that in these cases the strength increases were related to alloy partitioning effects alone [2]. For quench temperatures of 500° C or below the microstructure contained striations within the secondary α plates and 0.5% PS, TS and $\Delta\sigma$ appeared to rise more sharply due to this modification of the structure as the quench temperature was decreased below 500° C.

The formation of the striated substructure was related to a martensite reaction [2] that began at about 500° C. The most noticeable effect of the presence of the martensite was that for a quench temperature of 445° C the yield strength of the alloy was reduced (Fig. 1) with a corresponding loss in tensile strength. It is assumed that for quench temperatures just below the M_s temperature (i.e. 475 to 500° C) the martensitic phase was not present in sufficient quantity to have a noticeable effect.

For quench temperatures lower than 475° C the strengthening effects of the SQ would be progressively offset for lower quench temperatures by formation of larger fractions of the relatively soft martensite; a water quench to room temperature followed by ageing [4] results

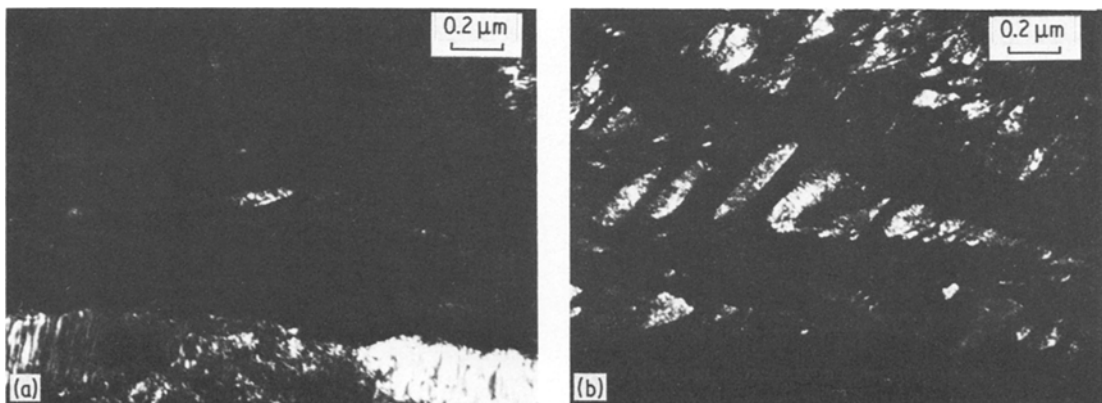


Figure 7 Step-quenched to 500° C: (0111) twinned α phase, (a) Twin 1, (b) Twin 2.

in strength similar to or less than that achieved by solution treatment air cooling and ageing, and is accompanied by relatively low ductility. Thus for maximum strength and moderate ductility the optimum SQ temperature is just below the M_s temperature.

The parameters $d\sigma/d\varepsilon$ and $\Delta\sigma$ showed that as the quench temperature was reduced the rate or work-hardening and the total work-hardening increased. It was also noticed (Fig. 1) that 0.1% PS was relatively insensitive to quench temperature (above 445°C). Thus, while SQ to a temperature in the range 475 to 600°C did increase the yield stress compared with the STA treatment, the most significant contribution to tensile strength was made by strain-hardening beyond the yield point. This was achieved largely by alloy partitioning effects, i.e. solution hardening, but with some restriction of slip by the substructure within the secondary α plates for quench temperatures of 500°C or below.

4.2. Structure

In an X-ray analysis Clark [5] found strong evidence that the structure of the martensite occurring in IMI 550 step-quenched to 500°C or below was orthorhombic with lattice parameters $a = 0.297$ nm, $b = 0.500$ nm, $c = 0.466$ nm; the orthorhombic structure arises from distortion of the hexagonal cell such that $b \neq a^{3/2}$. Clark [5] also noticed that when the orthorhombic martensite α'' was present the β phase was either just detectable or completely absent which was consistent with the normal $\beta \rightarrow$ martensite reaction in titanium.

These results are reproduced in Table II and indicate that the critical quench temperature for the formation of orthorhombic martensite is between 500 and 600°C. The striated structure detected in the present work occurred only for quench temperatures of 500°C or below, and suggests a M_s temperature between 500 and 525°C. However, the orthorhombic phase was not detected in the foils and β phase was found to be present in all cases. The probable explanation for this is that a structural alteration occurred during the thinning process. There is some evidence [6, 7] that orthorhombic martensite in titanium alloys can revert to β phase during the preparation of thin foils.

Another interesting feature of Clark's [5] results (see Table II) was that solution treatment

TABLE II X-ray structure analysis after Clark [5]

| Heat treatment | Phases present | | |
|----------------------------------|----------------|----------|------------|
| | α | β | α'' |
| 900°C 0.5 h, AC*, 600°C 4 h, AC | ✓ | ✓ | — |
| 900°C 0.5 h, SQ†, 600°C 4 h, AC | ✓ | ✓ | — |
| 900°C 0.5 h, AC | ✓ | ✓ | — |
| 900°C 0.5 h, AC, 500°C 24 h, AC | ✓ | ✓ | — |
| 900°C 0.5 h, SQ, 500°C 1 min, AC | ✓ | — | ✓ |
| 900°C 0.5 h, SQ, 500°C 24 h, AC | ✓ | — | ✓ |
| 900°C 0.5 h, AC, 400°C 4 h, AC | ✓ | a little | ✓ |
| 900°C 0.5 h, SQ, 400°C 4 h, AC | ✓ | — | ✓ |

*AC = air cooled.

†SQ = step-quenched.

followed by air cooling produced α and β phases, but subsequent ageing at 400°C resulted in α , α'' and some β phase. This implied that the orthorhombic phase was produced during the ageing process. This would mean that the α'' so formed was not the result of a diffusionless shear reaction, and therefore the transformation was not strictly martensitic.

The exact nature of the decomposition of the α'' during the thinning process and the significance of the striated α plates is not clear. The foil thinning process reportedly [6] causes reversion of α'' to β . The β phase detected in the foils of step-quenched IMI 550 was found to be between the striated α plates. Thus the α plates probably formed during the quench with the orthorhombic phase confined to the regions between the plates and transforming to bcc β during thinning without affecting the α plate structure.

The soaking treatment following the SQ does not appear to affect the α'' phase. The X-ray analysis summarized in Table II does not indicate any decomposition of the orthorhombic phase during soaking, and TEM examinations of specimens from previous work [2] shows that the duration of soaking has no discernible effect on the microstructure.

The striations within the plates lay in the α basal plane, but the (0001) plane is not a twinning plane for titanium [8] and there was no diffraction evidence of twins within the plates. No second-phase diffraction spots were observed, but this may have been due to the close spacing and lack of definition of the striations (Fig. 6). One possibility is that basal dislocations were generated to accommodate the martensite formation and these dissociated to leave stacking faults on the basal plane.

Striated α plates similar to those found in IMI 550 have been observed in other titanium alloys. Young *et al.* [9], working on Ti-6Al-2Sn-4Zr-6Mo, found striations aligned parallel to $[0001]_z$, i.e. perpendicular to those observed in step-quenched IMI 550. They found that the striated plates contained α and β phases, the latter both as narrow bands and small particles, and these striated plates coexisted with internally twinned martensitic laths. Williams [10] also found striated plates and suggested an α , α'' structure. Hammond [11], studying the α binary alloy Ti-5Mn, also observed coexisting internally twinned plates and striated plates. The traces of the striation lines lay in α basal planes and were attributed to stacking faults. Thus the striated substructure observed by Hammond corresponds to that in step-quenched IMI 550, although the internally twinned laths were not found in IMI 550. Basal plane stacking faults have also been found in pure titanium [12] and in Ti-8Al-1Mo-1V [13] after quenching.

Any study of the structure of orthorhombic martensite by TEM is severely hampered by the phase transformation that occurs during electropolishing, although it has been reported [6] that the use of an ion-milling technique to prepare thin foils does not produce this transformation.

For material quenched to 500°C or below there were some instances of a twin relationship between the α plates themselves (rather than between the striations within the plates) as shown in Fig. 7. Twin-related plates similar to this were observed by Blackburn [13] in quenched Ti-8Al-1Mo-1V. He decided that the plates form "co-operatively" to minimize the distortion associated with the formation of martensite.

The TEM work on the step-quenched material revealed that α plates in the transformed regions were not appreciably smaller for the lower quench temperatures. It is therefore concluded that the etching differences noticed in these areas of the microstructure (Fig. 3) were due to the less rapid etching rate of the orthorhombic martensite that existed in material quenched to 500°C or below, rather than simply a result of the scale of the microstructure.

It appeared that the STA microstructure contained a slightly higher primary α phase proportion than the step-quenched microstructures

(compare Figs. 2 and 3). This was probably a result of precipitation of α phase on to the existing α grains during the air cool in the STA heat treatment. This precipitation would have been minimal during a step-quench.

5. Conclusions

(a) The strength of solution treated, air cooled and aged (500°C) Ti-4Al-4Mo-2Sn-0.5Si alloy (IMI 550) can be increased by SQ from 900°C to a temperature in the range 445 to 600°C. The greatest strength increase (7% increase in 0.2% PS and 15% increase in TS) were obtained by SQ to 475°C.

(b) The strength increases obtained by SQ were accompanied by some loss in ductility.

(c) The strength increases produced by SQ were obtained largely by increases in work hardening due to alloy partitioning effects.

(d) The strengthening effect of SQ was reduced for quench temperatures below 475°C by the presence of orthorhombic martensite.

(e) The M_s temperature for orthorhombic martensite in IMI 550 is in the range 500 to 525°C.

(f) Fine basal plane striations within the secondary α plates were associated with the occurrence of orthorhombic martensite. Preparation of thin foils by electropolishing caused a reversion of the orthorhombic phase to bcc β titanium.

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