The Aging Behavior of Orthorhombic Martensite in Ti-6-2-4-6

MORRIS YOUNG, ERNEST LEVINE, AND HAROLD MARGOLIN

In Ti-6-2-4-6 alloys, beta transforms to orthorhombic martensite when quenched from a temperature of 1188 K or above, X-ray analysis showed that aging at 773 or 873 K gradually reduces the degree of orthorhombicity until a hexagonal structure equivalent to alpha, but having the morphological characteristics of the prior martensite, is produced. The orthorhombicity is reduced by solute rejection to beta which forms as particles both homogeneously and heterogeneously within the martensitic structure. The structure at maximum hardness is a fine distribution of Burger's oriented beta particles in a matrix of martensite of greatly reduced orthorhombicity. Overaging appears to occur as a result of coarsening of the homogeneous beta particles. It is shown that aging at temperatures from 873 to 1083 K results in growth of one particular varient of the beta which is located at the interface between twin related regions composing a martensite lath. This beta along with similarly oriented beta at lath interfaces forms a continuous beta matrix by a gradual growth process. It is shown that this matrix has the identical orientation and shape of the original beta grain prior to quenching. A mechanism is proposed to account for this "memory effect."

SIMILAR to most other titanium alloys, a range of temperatures exists for the Ti-6Al-2Sn-4Zr-6Mo alloy (6-2-4-6 in wt. pct) where β transforms to martensite on quenching. Unlike other systems it has been reported that the martensite has an orthorhombic structure[1] rather than the usual hexagonal variety. Most aging studies have been conducted on the hexagonal form of martensite where it has been found that beta precipitates heterogeneously within the martensite plates.[2] Williams and Hickman[1] have discussed aging mechanisms in orthorhombic martensite (α''). They reported that the first stage consists of the homogeneous precipitation of alpha needles in the α''. These needles were observed to co-exist with heterogeneously precipitated particles of beta for aging times of 150 h at 873 K.

Bywater and Christian[3] have examined the aging process of orthorhombic martensite formed in the Ti-Ta system. They observed that aging occurred by precipitation of the beta phase. These studies[1-3] did not attempt in any extensive way to correlate the aged structure with mechanical properties nor did they examine the system's approach to the equilibrium α plus β structure at higher aging temperatures.

This investigation has been concerned with the aging behavior of orthorhombic martensite in the temperature range of 773 to 1083 K. In this range the structure changes from that characteristic of maximum strength to overaged and grossly overaged.

EXPERIMENTAL PROCEDURE

The principal 6-2-4-6 material investigated was in the form of flat bar stock which has been previously warm worked in the $\alpha + \beta$ field. Its chemical analysis is shown in Table I.

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The flat bar stock was sectioned, polished and analyzed in an X-ray diffractometer with a pulse height analyzer. Orthorhombic lattice parameter values were obtained by calculation from high index reflections.

Hardness was obtained from electropolished specimens using a Zwick diamond pyramid hardness tester at a load of 10 kg. Tensile specimens of 0.318 cm diam, 2.54 cm gage length were machined from the flat bar stock after various heat treatments. The tensile results were obtained using a Tinius Olsen testing machine with a microformer extensometer and strain rate pacer. The strain rate employed was in the range 0.003 to 0.005 cm/cm/min. All heat treatments were done in quartz capsules which were filled with argon so as to reach 2 atm. pressure at the heat treatment temperature. At the end of the heat treatment the capsules were quenched and broken under water. The beta transus temperature was determined to be 1211 K.

Thin foil polishing for electron microscope studies was done with a standard solution[4] but with a potentiostatic power source. This resulted in extremely large thin areas suitable for observation at 120 kv.

RESULTS AND DISCUSSION

Mechanical Properties

Isothermal aging curves obtained at 873 and 773 K for an initial orthorhombic martensite structure produced by water quenching from the beta field are shown in Fig. 1. For both aging temperatures a rapid increase in hardness was observed in the first 1 min. followed by a gradual increase to a hardness of ap-

Table I. Chemical Composition of Ti-6-2-4-6 (wt pct)

Al	Sn	Zr	Mo	Fe	N	O	Ti
5.48	1.94	4.00	6.35	0.072	0.004	0.083	bal

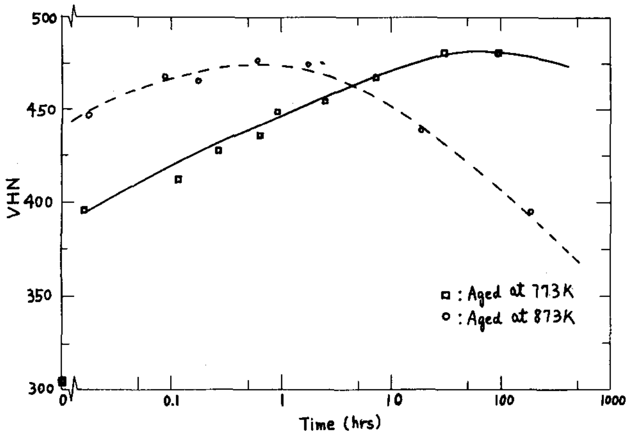


Fig. 1—Isothermal aging curve for orthorhombic martensite aged at 773 K and 873 K after quenching from the β field.

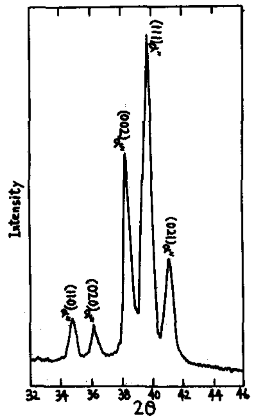


Fig. 2—Schematic of diffractometer scan between 32 and 46 deg. 2θ showing first five peaks of orthorhombic martensite. CuK_α radiation with Ni filter. Quenched from the β field.

proximately 475 VHN. Increased aging temperature (873 K) both decreased the time necessary to attain this maximum and in addition resulted in a significant overaging trend for times of 5 h or more.

Tensile tests were performed for an alloy quenched from the beta field and aged at temperatures of 873 K and above; the results are shown in Table II. It can be seen that aging resulted both in an increase in strength and a drastic reduction in ductility. This decrease in ductility has previously been reported for 6-2-4-6.[5]

The final tensile test was conducted after the quenched alloy was aged for 2 h at 1083 K. After this treatment, the strength level dropped and the ductility returned to its original as-quenched value.

Structure—As Quenched

A typical partial diffractometer scan observed in specimens quenched from the β field is shown in Fig. 2. Instead of the three peaks expected in this range of 2θ for hexagonal martensite there are five peaks characteristic of orthorhombic α''. The lattice parameters determined from the full diffraction pattern are $a = 3.02$, $b = 4.96$ and $c = 4.68$Å. The degree of orthorhombicity is conveniently given by the ratio of $b/a\sqrt{3}$ which for hexagonal symmetry is equal to unity. This ratio for specimens quenched from the β field is 0.948.

Table II. Tensile Properties of As Quenched and Aged Orthorhombic Martensite. Quenched from the Beta Field

	Yield Stress 0.2 pct (ksi)	Ultimate Tensile Strength (ksi)	Fracture Strength (ksi)	Elongation
As Quenched	55.7	155	155	6 pct
1 min Aged at 873 K			191	0
50 h Aged at 873 K			200	0
2 h Aged at 1083 K	141	148	148	6 pct

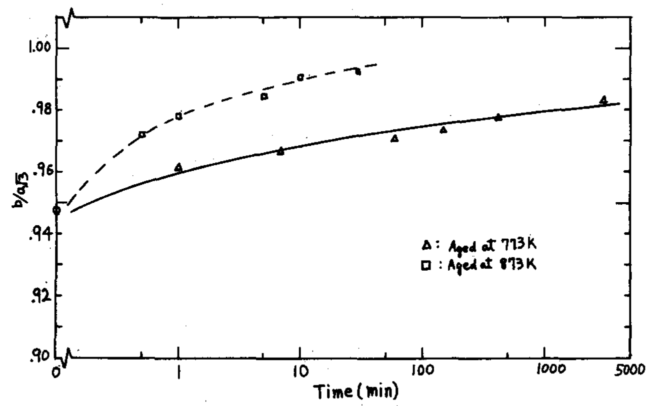


Fig. 3—Reduction in degree of orthorhombicity as a function of aging time. Quenched from the β field.

In order to ascertain the effect of increased beta solute content on the degree of orthorhombicity of the martensite, a specimen was heat treated in the $\alpha + \beta$ field (1182 K) so as to enrich the beta phase. On quenching, martensite was observed having an orthorhombicity ratio of 0.941. This was manifested by an increased peak splitting in the X-ray scan.

Optical microscopy of the as-quenched structure showed clear evidence of a martensitic transformation with no indication of retained beta. This is in agreement with the absence of β peaks in the X-ray pattern.

Due to the poor delineation of the as-quenched martensitic structure satisfactory replicas could not be obtained. In addition, spontaneous transformation of thin foils prevented verification of this structure by transmission electron microscopy.

Structural Analysis of the Low Temperature Aging Products

X-ray and electron microscopic analysis was performed on specimens aged at 773 and 873 K for various times. The X-ray analysis revealed a gradual reduction in the degree of orthorhombicity as a function of aging time as summarized in Fig. 3. No phases other than the original martensitic structure were apparent in the X-ray scan until the appearance of beta after 50 h aging at 773 K or 5 min. at 873 K. The beta peaks were very broad, suggesting fine particles, but gradually sharpened with time. By the time of initial appearance of these broad peaks the degree of orthorhombicity had been significantly reduced. It is probable that β particles were present prior to their visibility in the X-ray scan but due to small particle

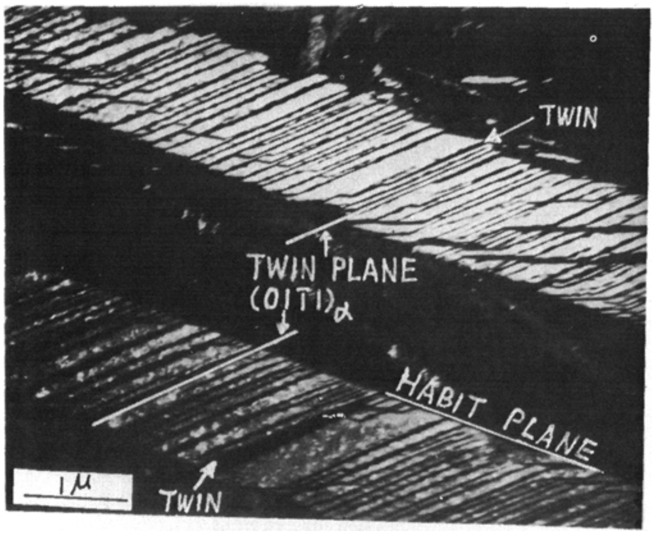


Fig. 4—Dark field micrograph on 1101 alpha reflection showing two parallel laths with identical orientation. Quenched from the β field, aged at 873 K for 50 h.

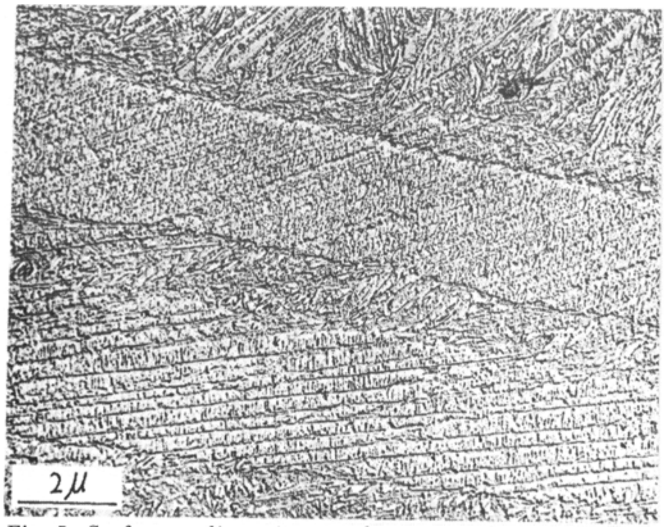


Fig. 5—Surface replica micrograph. Quenched from the β field, aged at 873 K for 50 h.

size effects they would be difficult to observe in a diffractometer scan. Subsequent aging at 773 and 873 K serves to accentuate the β peaks as growth occurs and the martensite clearly attains hexagonal symmetry and lattice parameters more characteristic of alpha.

In view of the earlier results which showed that increased solute content increases the orthorhombicity, it is clear that the solute content of the martensite is reduced by aging. Since only α, α'' and β phases have been observed, this suggests that the orthorhombic martensite is gradually converted to equilibrium alpha by the precipitation of β and concomitant solute redistribution. The following results will serve to verify and expand on this conclusion with transmission electron microscopy techniques.

Because of the spontaneous transformation mentioned previously in connection with as-quenched foils it was decided to start the thin film transmission electron microscopy investigation of the decomposition structure after 50 h aging at 873 K. In this way the nature of the decomposition products could be established and compared with replicas to assure that the structure was retained. Once this structure was established, developments at other aging times could be traced, with the view that the structure observed at any stage must be consistent with the martensite morphology observed in the fully stabilized state, and with a surface replica of the stage of aging under investigation.

Examination of thin foils of specimens aged for 50 h at 873 K showed two distinct types of aged martensitic structures, namely martensitic laths containing internal twins and a second structure which will be called the striated plate structure. Both structures were usually observed within one foil, but the internally twinned structure appeared to predominate. Indeed in some foils only the internally twinned structure was visible. In the following the aging behavior of each structure will be analyzed.

Twinned Martensite

Fig. 4 shows the general morphology of the internally twinned plates. This micrograph was taken in dark field using a $1\bar{1}01\ \alpha$ reflection.* It clearly shows two long laths each of which consists of alternating twin related regions which appear to characterize this form of martensite. In this micrograph, only one of the regions within the lath was in contrast. The finer black lines in between are twin related to the light structure. These features as well as the trace of martensite habit plane are indicated on the micrograph. In the area between these laths another martensitic system, which was twinned on a different $\{10\bar{1}1\}$ plane, was visible in bright field. This area was not in contrast under the operating dark field imaging conditions. Also it can be noted that in the lower lath, fine precipitates are apparent within the region in contrast. These will be considered in detail in a later section.

Fig. 5 shows a replica of the residual martensitic structure after 50 hours aging at 873 K. The replica contains long narrow laths similar in appearance and size to those shown in Fig. 4. From the similarity of structure observed by thin film transmission electron microscopy, replicas and optical microscopy at this and shorter aging times it was concluded that the structure under observation is truly indicative of the bulk transformation product.

It thus follows that examination of the lath structures at this aging time should permit one to deduce the morphological characteristics of the as-quenched martensite as well as the aged structure.

Fig. 6 clearly shows the characteristics of this type lath. The insert in Fig. 6 is the diffraction pattern from the lath. This micrograph taken with a $1\bar{1}01\ \alpha$ reflection shows that the bulk of the lath is of one orientation. The narrower dark bands are twin related to those in contrast. The ratio of volume fraction of these twin related regions was approximately 3:1 to 4:1. The twinning plane of this lath was also of

*The structures will be referred to as α due to the reduced orthorhombicity at this aging time. However, both the morphological and X-ray observations clearly indicate that they were originally α''.

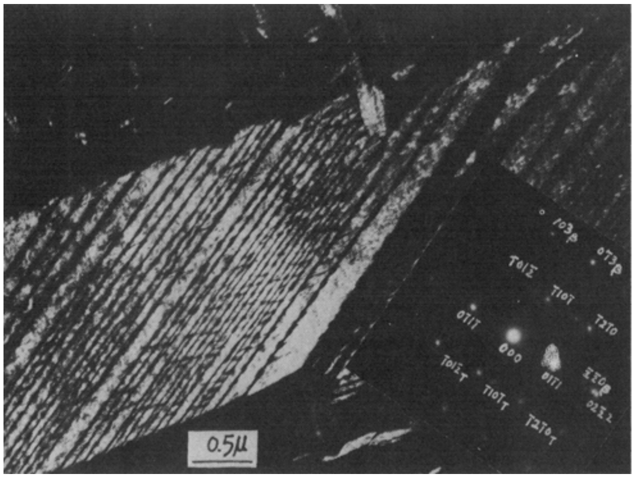


Fig. 6—Dark field micrograph on 1101 alpha reflection, quenched from the β field, aged at 873 K for 50 h.

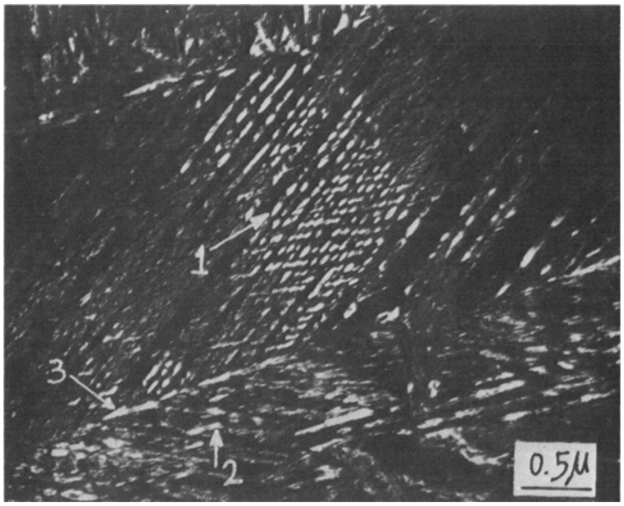


Fig. 7—Dark field micrograph on 103 beta reflection in [331] zone axis showing beta precipitating on twin and lath interfaces. Arrow 1 shows interface β on the same lath as shown in Fig. 6. Arrow 2 shows interface β in adjacent lath. Arrow 3 shows β located between laths. All β precipitates have the same orientation. Same heat treatments and location as shown in Fig. 6.

the $\{10\bar{1}1\}$ type. No twin planes other than $\{10\bar{1}1\}$ type were found in this study. These characteristics are consistent with previous investigations of both hexagonal and orthorhombic martensite plates.[1,6] Bowles and Mackenzie[7] suggested that the $\{10\bar{1}1\}$ type twinning plane should be active since it meets the requirements that a mirror plane in the parent BCC becomes a twinning plane in the product martensite. In this case the lattice correspondence for minimum atomic displacements is such that the $\{10\bar{1}1\}$ twinning plane is equivalent to a $\{110\}$ parent beta plane. This fact will become important in subsequent analysis of the beta precipitate.

It can be seen in Fig. 6 that the narrower twinned regions appear irregular. Dark field analysis using a 103 beta reflection clearly lit up particles of beta at twin interfaces (Fig. 7, Arrow 1) which were responsible for this irregularity. The complete diffraction pattern from the lath is shown schematically in Figs. 8(*a*) and (*b*), which are separated for clarity. Dark field techniques showed that the $0\bar{1}3$ and 110 reflections both belong to the same zone, *i.e.*, that of the interface beta precipitates. The interface particles were thus identified as beta in a $[\bar{3}31]$ orientation.

A comparison of the interface beta precipitate image to that of Fig. 6 showed that the beta is consuming both twin related regions equally but because of the narrowness of one region it alone appeared irregular. It was also noted that precipitates of similar orientation were in contrast in adjacent laths (arrow 2, Fig. 7) and also at lath-lath interfaces parallel to the observed habit plane trace (at arrow 3). The latter precipitates were larger than the interface beta.

It can be shown by stereographic analysis that the interface beta has a Burger's orientation relationship, ($\{0001\}_\alpha \parallel \{110\}_\beta$, $\langle 11\bar{2}0\rangle_\alpha \parallel \langle 111\rangle_\beta$) with respect to the twin related regions to either side of it. It is thus not surprising to observe that these precipitates grow equally into both regions.

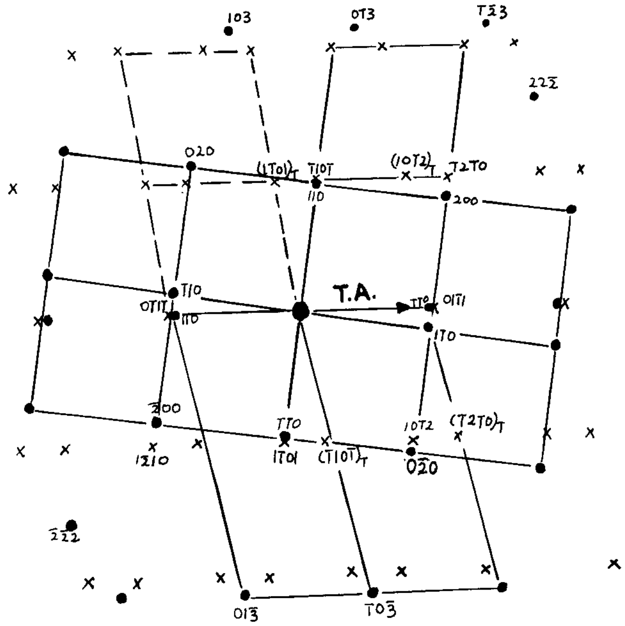
It is of interest that no other orientation of beta was observed at the twin interfaces both within a lath and within other adjacent laths in the surrounding regions. This point will be considered in more detail in a later section.

In addition to the interface beta precipitates, homogeneously distributed β particles were observed within the twin related regions comprising a lath. Unlike the heterogeneous precipitates at the twin interfaces where only one variant was present, at least five Burger's oriented variants of the precipitate were observed in each region. Fig. 9, a dark field micrograph of one variant, shows the small size of these precipitates (200Å) compared to the interface precipitates.

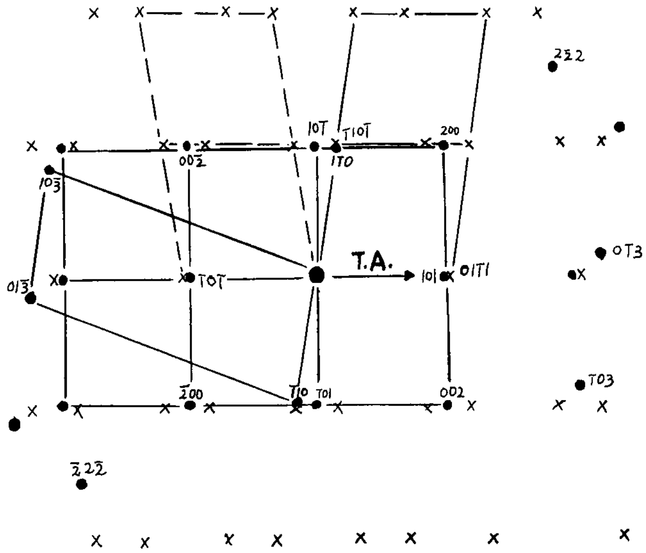
The interparticle spacing of one variant is estimated at 250Å. Since all the variants were homogeneously distributed it is probable that the actual interparticle spacing was closer to 1/4 to 1/6 of this spacing.

Long time aging (500 h) at 873 K results in a general coarsening of the structure. Thus, Fig. 10 shows that the interface β exists as either isolated sausage-like particles or long plates with a habit plane of $\{110\}_\beta \parallel \{1\bar{1}01\}_\alpha$. Dark field techniques revealed that very little of the narrower regions within a lath remained at this aging time and temperature. Also the edges of the remaining plates take on an increased irregularity as they are consumed by β. At this stage of aging the remaining portions of martensite plate are very easily distinguished from the precipitated interface β by the presence of internal β precipitates. The size and interparticle spacing of all variants of internal β precipitates had considerably increased. One variant of the β is shown in Fig. 11 with the β indicated by arrows. The spacing has increased to approximately 1000Å, however due to the intermixing of other variants which are not in contrast this spacing could be as small as 200 to 250Å.

The above described results were obtained after 50 h and 500 h aging at 873 K. Comparison to Fig. 1, the aging curve, shows that the structure at this time is characteristic of the overaged state. Similar analysis performed after 1 and 5 h of aging (maximum aging condition) showed essentially the same structural



(a)



(b)

Fig. 8—(a) and (b) separated for simplicity. Schematic diffraction pattern of zone axis [10$\bar{1}$1] alpha with all possible Burgers β variants and the twin pattern of α. T.A. refers to twin axis, twin reflections are indicated by subscript T.

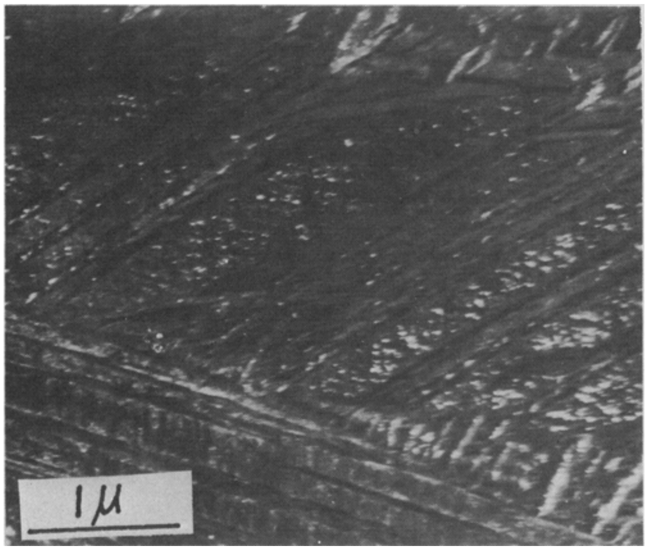


Fig. 9—Dark field micrograph on a 110 beta reflection showing internal beta precipitates. Quenched from the β field, aged at 873 K for 50 h.

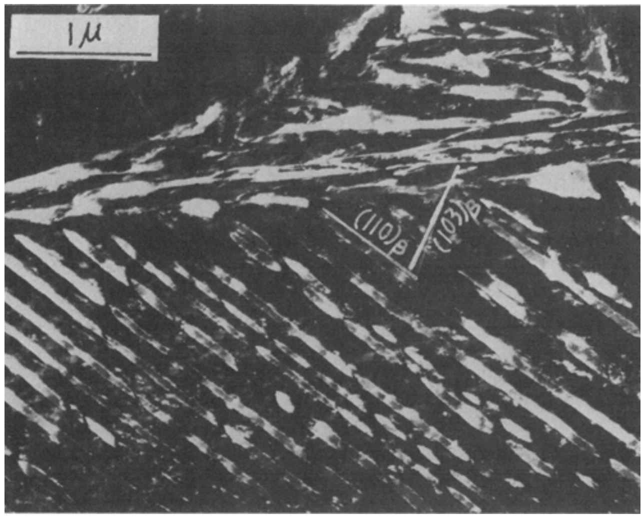


Fig. 10—Dark field micrograph taken on 103 beta reflection in [$\bar{3}$31] zone axis showing interface beta precipitates in an overaged structure. Quenched from the β field, aged at 873 K for 500 h.

characteristic except that the size of the beta precipitates was much finer.

An attempt was made to analyze specimens aged for 1 and 5 min. at 873 K, but the results were not reliable since the foils did not have the same structure as the replicas of the same specimens.

Striated Martensite

The striated martensite plate structure has a distinctly different morphology than the internally twinned lath just discussed. A bright field micrograph of this structure, aged at 873 K for 50 h is shown in Fig. 12(*a*) (note the internally twinned laths intermixed with this structure). The selected area diffraction pattern of this structure is shown in Fig. 12(*b*) and is labeled as an HCP structure in a ⟨11$\bar{2}$0⟩ zone axis. The striations are spaced about 200Å apart and are parallel to the $[0001]_\alpha$ direction as indicated on the micrograph.

Dark field analysis showed that the striations are alternate bands of alpha and beta. A dark field micrograph on an alpha reflection is shown in Fig. 13. The regions of β which are dark in this micrograph are much narrower than the alpha. No twins or other planar defect structures were observed within the structure at this stage of aging. However, in addition to the alternate striations of α and β, internal rods of β were observed which are shown in Fig. 14. The rods are 25Å by 200Å long and they have a Burger's orientation relationship with the α phase.

Unlike the martensite laths containing twin related regions, the prior structure of this type of martensite could not be ascertained. At all aging times

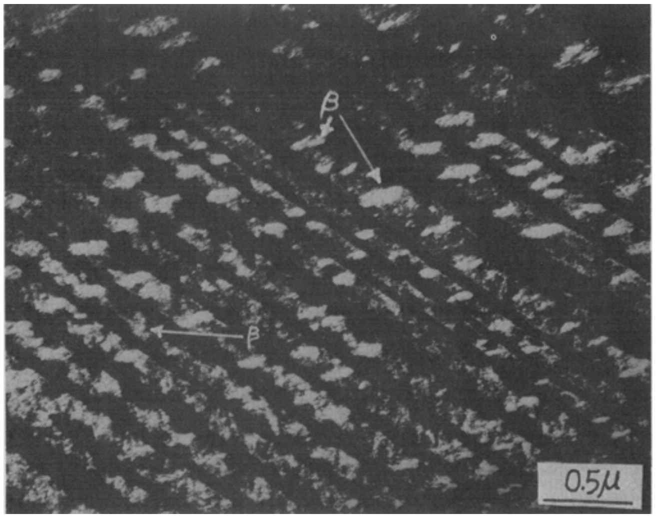
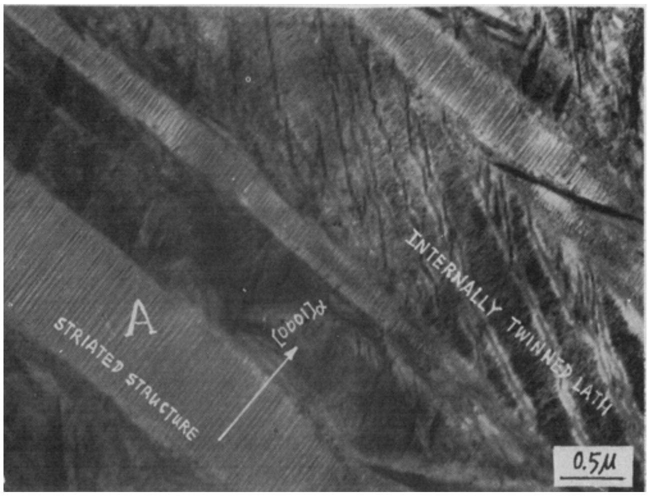
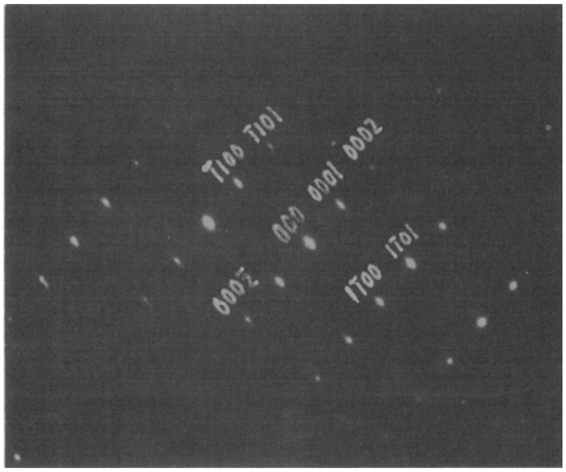


Fig. 11—Dark field micrograph on a 110 beta reflection showing one variant of Burger's beta in the twin related alpha phase. Same heat treatments as Fig. 10.



(a)



(b)

Fig. 12—(*a*) Bright field micrograph showing striated martensite plate and internally twinned structure. Quenched from the β field, aged at 873 K for 50 h. (*b*) Selected area diffraction pattern of area indicated by ''A''.

where structural analysis was considered reliable, it consisted of α and β, the latter both as particles and narrow bands. Further, the trace of the α bands were not consistent with any possible twinning plane of the orthorhombic martensite. Williams[1] has observed a similar structure in his study of orthorhombic martensite and attributed it to α and α'' coexistence. No evidence to support this contention was obtained in this study. In any case the relatively small amount of this structure suggests that its contribution to hardening would be small.

High Temperature Aging

Since a significant increase in ductility was found to occur in specimens heat treated for 2 h at 1083 K it was of interest to determine the associated struc-

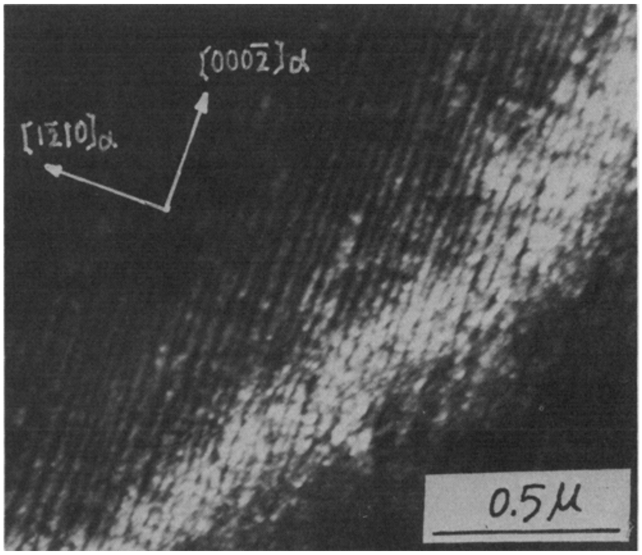


Fig. 13—Dark field micrograph on 1210 alpha reflection. Quenched from the β field, aged at 873 K for 50 h.

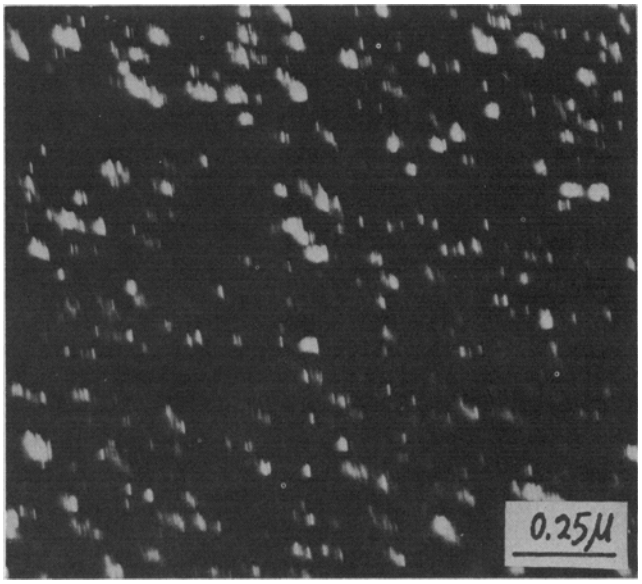


Fig. 14—Dark field micrograph showing rod-like β precipitates in the striated martensite plate. Quenched from the β field, aged at 873 K for 50 h.

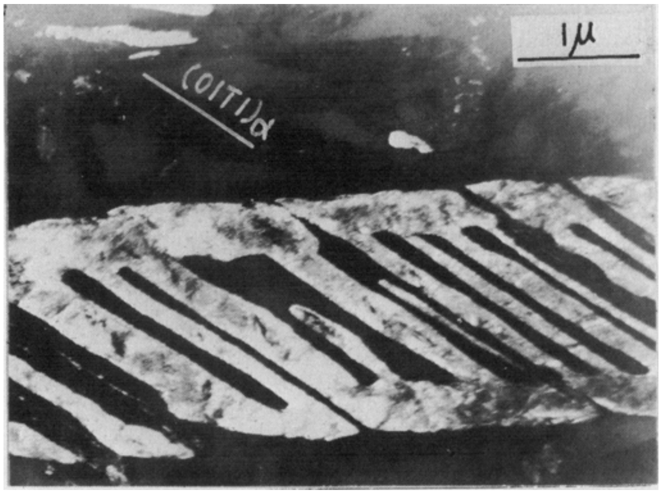


Fig. 15—Dark field micrograph on a 1101 alpha reflection. Quenched from the β field, aged for 160 h at 873 K + 12 h at 1023 K.

tural changes. At this higher aging temperature the interface β thickened and increased in volume fraction to the extent that a clear β matrix was established. Examination of large areas of the foil showed that the orientation of β was essentially the same. Some residual areas with trapped β remained as shown in the dark field micrograph, Fig. 15, which was taken with an α reflection. The β regions in this micrograph are the dark entrapped areas. In addition this micrograph clearly reveals the similarity of the present α morphology to that of the prior martensite, although its composition is identical to that of the equilibrium α.

The above described general characteristics at this aging time and the course of development at lower aging temperatures clearly suggests that the establishment of a β matrix is a consequence of the growth process of the interface β as well as the β observed at lath-lath interfaces. As stated previously, the interface β has the same orientation both within a single lath and from lath to lath. The growth of this beta would thus establish a large continuous region of β and the remaining α would still have a similar morphology to the martensite formed on quenching.

It remained to show that the β after aging at 1083 K has the same orientation and grain size as the prior β grain. Large β grains (about 0.5 cm.) were grown at a solution treatment of 1473 K for 48 h and quenched into water. The martensite was aged at 1083 K for 2 h and Laue back reflection photographs were taken of two large grains. The sample was then reheated to 1373 K for 1 h, water quenched and aged at 1083 K for 2 h. Laue photographs were taken of the same two grains which could be recognized from their visual appearance. The Laue photographs showed that after the second heat treatment at 1373 K and aging at 1083 K the same β orientations were found. It appears, therefore, that the heterogeneous β precipitation forming when orthorhombic martensite is aged in the $\alpha + \beta$ field reproduces the original beta orientation which eventually converts the entire grain to its original size and orientation.

One final point should be mentioned. Now that it is known that the interface β precipitates reflect the former β orientation, it is possible to determine the habit plane of the martensite. Such a determination yields the normal $\{334\}$ and $\{344\}$ habit planes and at once confirms the suggestion that the original β grain is reproduced by growth of these interface β precipitates.

GENERAL DISCUSSION

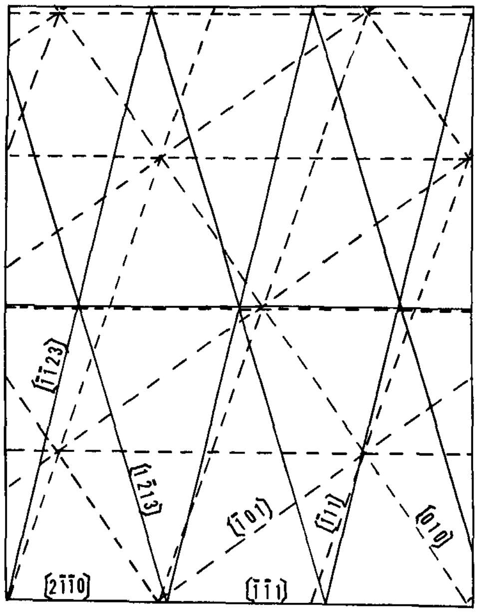
The low yield stress associated with the as-quenched martensite structure (Table II) suggests that the strain induced martensitic formation or boundary movement may be involved in the deformation process. Since no beta was revealed by optical microscopy or X-ray analysis it is probable that glissile boundary movements or the growth of favorably oriented twins within martensite is the operative deformation mode. Some support for this latter suggestion is given by the marked increase in the delineation of martensite plates in the vicinity of a hardness indentation placed within the as-quenched martensitic structure. The twinned regions of these martensite plates were found to be much larger than those seen in the as-quenched martensite not subjected to deformation. Further, the observed size of these laths was incompatible with transformation of small undetected residual regions of β.

The formation of heterogeneous boundary precipitates of β on aging would lock both twin and martensite lath boundaries and thus restrain the as-quenched martensite deformation mode from operating, thus accounting for the initial rapid increase in hardness.

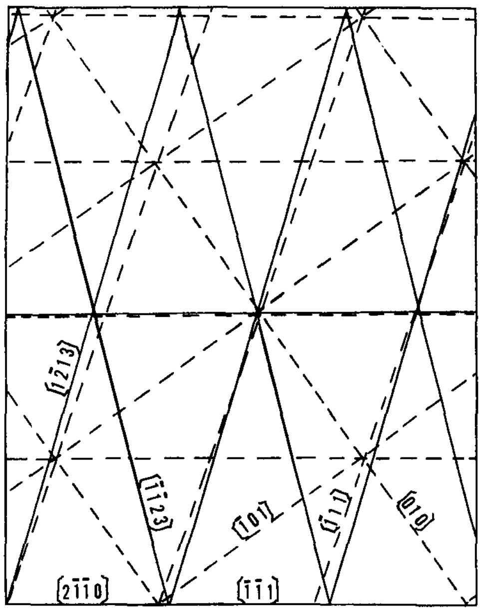
As aging continues, homogeneous β precipitates were observed within the martensite plates. The interparticle spacing of the internal β is probably less than 50Å at the maximum hardness if one considers all variants of the β. This would make Orowan bowing around these particles extremely difficult. The β is probably harder than the residual martensite, possibly because of the difficulty of nucleating slip in such small particles. The combination of these two effects would significantly strengthen the martensite plates since all deformation is now restricted to dislocation motion within the laths which necessitates shearing of the fine β particles. Aging for 500 h at 873 K results in coarsening of these β particles and the hardness of the alloy decreases. The larger interparticle spacings observed after this aging treatment could permit a by-pass mechanisms to become operative.

It is interesting to note that significant ductility was not observed until after the high temperature aging treatments. This final heat treatment at 1083 K produced a large grained β matrix structure free of homogeneous precipitation which could support a significant degree of deformation without fracture.

It is necessary to consider why the orientation of the interface β is constant both within laths and between different martensite laths for this lies at the heart of why a large grained β matrix is produced on high temperature aging. In fact it has been shown that the original grain structure is reproduced even though it is clear that a competitive growth process is occurring.



(a)



(b)

Fig. 16—Schematic plane matching showing [$\bar{1}\bar{1}1$] ∥ [$2\bar{1}\bar{1}0$]; (*a*) [$\bar{1}11$] approximately parallel to [$\bar{1}\bar{1}23$]; (*b*) [$\bar{1}11$] approximately parallel to [$1\bar{2}13$]. Solid lines $\{10\bar{1}1\}_\alpha$ plane. Dotted lines $\{110\}_\beta$ plane.

One can postulate that the initial precipitation occurs by a nucleation and growth process. In this case the twin boundaries which were found to be only of the $\{10\bar{1}1\}$ type would be a preferred heterogeneous nucleation site. Converting this $\{10\bar{1}1\}$ twin plane into a $\{110\}$ beta plane would eliminate the twin interface and also satisfy the Burgers orientation relationship. A comparison of the detailed atomic relationship associated with overlapping these two planes is shown in Fig. 16. It should be noted that two possibilities exist. Due to the orthorhombicity of the parent phase both possibilities are not equivalent. It is clear that a better lattice match is obtained in case *b* and thus only one orientation of beta would be favored assuming that minimization of lattice mismatch is important in the nucleation process.

One could also consider that the formation of interface β may be a result of a reverse martensitic shear movement. The twin interface would be a favored site for this reverse shear since the principal strains of the lattice deformation involved would partially cancel across this interface. However, if this were the case one would expect a habit plane parallel to $\{334\}$ or $\{344\}$ type plane which has a trace parallel to the side of the martensite lath. This habit plane was found for the β precipitates observed at lath-lath interfaces (Fig. 7, arrow 3) but not within a lath (Fig. 7, arrow 1). However, it is possible that the initial β nucleus formed at the twin interface could have had a habit plane parallel to $\{334\}$ or $\{344\}$ type plane, but since it would grow faster along the twin interface, at some later aging time it would appear to have a habit plane parallel to $\{110\}_\beta$.

The net result of all these processes favors a return to the original β orientation and prevents cyclic grain refinement in these alloys. Based on this information, the only possibility for grain refinement in this alloy would be by further cold work of the structure in order to disrupt these favored sites for original β orientation precipitates.

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