On Variant Distribution and Coarsening Behavior of the α Phase in a Metastable β
Titanium Alloy

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Abstract

The stereology, variant distribution and coarsening behavior of semicoherent α (hcp) precipitates in a β (bcc) matrix of a Ti5553 alloy has been analyzed, and a dominant 3-variant cluster has been observed in which the variants are related to each other by an axis-angle pair $\langle 11\bar{2}0 \rangle 60^\circ$. Shape and spatial distribution independent elastic self and interaction energies for all pairwise and triplet combinations of α have been calculated and it is found that the 3-cluster combination that is experimentally observed most frequently has the lowest energy for the semicoherent state. The coarsening behavior of the delta distribution follows LSW kinetics after an initial transient, and has been modeled by phase field methods.

Keywords

Titanium alloys, variant selection, self-accommodation, coarsening

1. Introduction

The aging of metastable β titanium alloys below a certain critical temperature in a two-phase $\alpha+\beta$ region has been shown to result in a distribution of fine, intragranular α precipitates with a characteristic triangular arrangement of α plates, shown in Figure 1a. We call this distribution a delta distribution. The

1 thermal paths that lead to these distributions have been discussed in earlier work
2 [1,2]. The morphology and crystallography of the delta distribution has been
3 examined in the past [3]. These distributions have also been shown to be
4 associated with superior mechanical properties[4]. The α plates precipitate in a
5 Burgers relationship (0001)//(110): $\langle 11\bar{2}0 \rangle // \langle 111 \rangle$ to the parent β with 12
6 crystallographic variants of which 4 sets of 3 variants taken together share a
7 common $\langle 111 \rangle // \langle 11\bar{2}0 \rangle$ zone axis, as shown in Figure 1b. The misorientations
8 between all possible pairs of these 12 variants of α laths in terms of reduced axis-
9 angle pairs reduces to 6 different types as described in [5] and is reproduced in
10 Table I. Any deviations from random distribution of these 12 variants can be
11 described in the terms of the frequency of occurrence of these pairs. Figure 1a
12 suggests that a preferred, non-random distribution does indeed exist with local,
13 spatial correlations between certain types of variants.
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26 The mechanical behavior of alloys with these α distributions will be affected by
27 elastic interactions and slip transfer processes between α and β that are strongly
28 determined by the relative orientations of slip planes and directions in the two
29 phases determined by the Burgers relationship [6]. The presence of multiple
30 variants of the α phase within a single β grain will therefore restrict slip as
31 discussed in [7]. The α phase variant distribution and its coarsening with heat
32 treatment are therefore important parameters that will determine mechanical
33 response. It is against this background that we have examined the stereology and
34 coarsening of α plates in delta distributions in a variant of the Ti5553 alloy widely
35 used in undercarriage applications in aircraft.
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46 2. Experimental

47 The alloy used in this study was melted by non-consumable vacuum arc melting
48 into a pancake that is typically 15 cm in diameter and 1 cm in thickness to the
49 nominal composition of the Ti5553 alloy (without Fe). Samples from the alloy
50 were heat treated from the as-cast condition for 0.2 hr, 0.83 hr, 2 hr, 4.5 hr, 16 hr
51 and 144 hr at 765C. Figure 2 shows the starting microstructure preceding heat
52 treatment in the as cast condition that indicates that the delta distribution had
53 already formed during cooling after melting. The microstructure was characterized
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1 by back-scattered electron (BSE) imaging, and by electron back-scattered
2 diffraction (EBSD) to identify the crystallographic orientations of the α variants.
3 The EBSD data was used to derive 'non-correlated' misorientation information
4 between pairs of α variants using the proprietary TSL software. The 'non-
5 correlated misorientation measures the misorientation between any single variant
6 of the α phase and all other variants in a pair wise-manner, and repeats this
7 process for every α phase plate in the area that has been scanned in the EBSD
8 map. This provides the frequency distribution of the reduced axis-angle
9 misorientation between any two variants of α plates in the area of the scan. Such a
10 measurement should yield frequencies of pairwise misorientations that approach a
11 value expected from a random distribution of 12 variants as given in Table I, if all
12 12 variants are present in equal numbers in the area of the scan. However the
13 proprietary TSL software is unable to obtain misorientations between pairs of α
14 variants in a near-neighbor configuration, since these α plates are not, in general,
15 in contact with each other (a requirement for measurement of misorientations
16 between grains in a near-neighbor configuration in the current TSL software). A
17 spatially correlated near- neighbor misorientation frequency distribution was
18 therefore obtained from software developed for this purpose in the MATLAB
19 environment based upon the frequency of pair-wise misorientations between
20 variants that lie within a given nearest neighbor distance of every plate, as a
21 function of aging time. The nearest neighbor distance was assumed to be the
22 inverse square root of the number of α plates per unit area and double counting
23 was eliminated.

24 EBSD images were also used to obtain data (using TSL software) on the major
25 and minor axes of the representative ellipses used to approximate the 2d sections
26 of α plates (grains), as function of aging time. These analyses were conducted on
27 typically about 1000 α laths per scan area in a given β grain. In some cases scans
28 were taken from several grains and data from all scans has been merged into a
29 single data set for each ageing time. Orientation imaging microscopy was also
30 carried out by precession electron diffraction in transmission electron microscopy
31 using NanoMEGAS instrumentation. 3d images of the α variants were obtained
32 by conventional serial sectioning techniques and reconstruction using proprietary
33 Amira software. Greater detail of the serial sectioning and reconstruction

techniques is provided in [8].

3. Results

3.1 Experimentally Observed Variant Distributions

Figure 3 shows BSE images of α laths as a function of aging time at 765°C, while Figure 4 shows corresponding EBSD images. Representative 3d images of individual α plates are shown in Figure 5 for two different aging times. The plates are disc shaped with elliptical faces.

It is convenient to analyze the spatial distribution of the 12 variants in terms of the probability of occurrence of pairwise misorientations to assess whether the spatial distribution of the 12 variants is as expected from their random occurrence, or whether preferred distributions exist. Thus a ‘non-correlated’ analysis of the α lath misorientations should approach the random pair-wise misorientation distribution shown in Table I while the spatially correlated distribution as defined in section 2 should indicate the existence any preferred variant distributions.

Figure 6a shows the non-correlated misorientation distribution of α laths after different aging times. The observed frequencies of misorientation approach the values expected for random distributions given in Table I. This data suggests that all variants are approximately present in proportions expected from a random probability of occurrence of pair wise misorientations in the area of the scans. However a spatially correlated misorientation analysis restricted to the nearest neighbor distribution (Figure 6b) shows a different frequency of occurrence with the Type B frequency being much higher than the random value corresponding to the other types. The effect of aging is to reduce the frequency of the Type A pairs while there is an increase of the Type C pairs. This change occurs within the first few hours aging, and thereafter the frequency of all pair combinations does not vary with time. The values of observed frequencies are given in Table II.

Figure 1 strongly suggests that there is a tendency of clusters of 3 variants to form (see encircled clusters), giving the distribution its characteristic delta appearance.

EBSD analysis indicates that these clusters of 3 variants have a common $\langle 1120 \rangle$ α axis parallel to $\langle 111 \rangle$ of the parent β , as shown in Figure 7. Thus the delta distribution is most evident when the plane of metallographic section lies normal to a $\langle 111 \rangle$ direction of the β phase so that the triangular arrangement for a cluster of 3 such variants is revealed. Figure 8 shows a phase partitioned image obtained using orientation image mapping using precession electron diffraction. The 3 variants share interfaces that are parallel to a $\{10\bar{1}1\}$ plane common to each pair of variants. The 3d reconstructed image of one such cluster shows that the variants form a hollow pyramid and meet at the apex of the pyramid with the crystallography shown in schematically (see also [3] and Figure 1).

As in the case of pair-wise frequencies, we have computed the expected frequencies in the random case of various combinations of clusters of 3 variants as shown in Table III. 364 such combinations are possible which reduces to 19 possible clusters (as explained in detail in the supplementary information SII). These nineteen 3-variant clusters are defined in Table III in terms of the reduced axis-angle misorientation of each pair taken at a time in each cluster. The expected random frequency is obtained by examining the number of combinations of the 12 possible variants that will yield a specific cluster type, and dividing by the total of such combinations. We have examined the frequencies of clusters of 3 variants using the same experimental methods used for determining pair-wise interactions and the results are shown in Figure 9 (and Table III) for a particular aging condition. The occurrence of several clusters of 3 variants exceeds the frequencies expected from random distributions.

3.2 Analysis of Spatially Correlated Variant Distributions

3.2.1 Pair wise distributions

The α phase can exist in the coherent or semi-coherent state, with the former more likely at the nucleation stage [7]. Previous papers [9,10] have developed a quantitative 3d model incorporating the crystallography of the β to α transformation and α/β interface defect structure to calculate a stress free transformation strain (SFTS) of each of 12 α variants in both the coherent and semi-coherent state. These SFTS are

reproduced in the supplementary section (SI) of this paper. Since there is no external bias such as pre-strain, external stress or stress-carrying defects, the occurrence of variant selection could be ascribed to the elastic interaction between variants to achieve the largest degree of self-accommodation. According to Khachaturyan's microscopic elasticity theory of solid solution [11], the total elastic strain energy can be separated into two physically distinct parts: The configuration independent, E_0 and configuration dependent, E_1 parts [12]. According to Khachaturyan's microscopic elasticity theory [11], the total elastic strain energy can be separated into two physically distinct parts: the configuration-independent part, E_0 , and the configuration-dependent part, E_1 [12]. The former is given by:

$$E_0^{el} = \frac{1}{2} \sum_{p,q=1}^N \int \frac{d\mathbf{g}}{(2\pi)^3} \langle B_{pq}(\hat{\mathbf{n}}) \rangle \tilde{\phi}_p(\mathbf{g}) \tilde{\phi}_q^*(\mathbf{g}) \quad (1)$$

where $\langle B_{pq}(\hat{\mathbf{n}}) \rangle$ characterizes the density of the configurationally independent part of the total elastic strain energy and $\tilde{\phi}_p(\mathbf{g})$ is the Fourier transformation of $\phi_p(\mathbf{r})$ that describes the spatial distribution of the p^{th} variant of α phase and $\tilde{\phi}_p^*(\mathbf{g})$ is the complex conjugate of $\tilde{\phi}_p(\mathbf{g})$. The details of Eq. (1) and definition of $\langle B_{pq}(\hat{\mathbf{n}}) \rangle$ can be found in the Appendix. E_0 is configuration-independent and thus is not able to offer information such as morphology and spatial arrangement of different variants of α precipitates in the frequently observed clusters, for which one should consider the configuration-dependent part of the elastic energy, E_1 . Since the focus of the present work is on the frequency of occurrence of different variant pairs or triplets, only E_0 is considered.

Figure 10 illustrates how $\langle B_{pq}(\hat{\mathbf{n}}) \rangle$ vary with coherency state and the different misorientation types listed in Table II. In the coherent state, as highlighted by arrows in Figure 10 a, the pair with the Type D misorientation (Table II) has the smallest (and also negative) value among all possible combinations between a pair of variants. In the semi-coherent state as shown in Figure 10b, the pair with the Type B misorientation has the most negative value, followed by Type C, among all possible combinations between a pair of variants.

An alternative way to analyze the elastic interaction between different variants is to calculate the effective Von-Mises strain, ϵ^e , between them, which is:

$$\epsilon^e = \frac{\sqrt{2}}{3} [(\epsilon_1 - \epsilon_2)^2 + (\epsilon_2 - \epsilon_3)^2 + (\epsilon_3 - \epsilon_1)^2]^{1/2}$$

where ϵ_1 , ϵ_2 and ϵ_3 are the principal strains of average transformation strains of a pair of any two variants. Figure 11 illustrates how ϵ^e varies with different misorientation types and the coherency state listed in Table I. In the coherent state, the Type D combination has the smallest ϵ^e , which is followed by the Type C pair. In the semi-coherent state, the Type B combination has the smallest ϵ^e , followed by the Type C pair. The Type A combination has the largest ϵ^e irrespective of coherency state.

It is interesting to find that both the Von-Mises strain analysis and $\langle B_{pq}(\hat{n}) \rangle$ analysis offer the same results, while noting that the latter analysis also accounts for the elastic modulus. We note that the effect of elastic modulus can also be incorporated in to the Von-Mises analysis using the approach of [13]. The analyses indicates that the preferred variant distribution observed experimentally can only be explained by the residual elastic interaction strain of semi-coherent particles, in that the Type B pair frequency is shown to be dominant both experimentally as well as by preceding analyses for the semi-coherent state. The analysis also explains the increase in the frequency of the Type C pairs at the expense of the Type A pairs with coarsening time.

3.2.2 Clusters of 3 variants

Since both the Von-Mises strain analysis and $\langle B_{pq}(\hat{n}) \rangle$ analysis offer the same results we have only calculated the Von-Mises strain for semi-coherent plates for the 19 possible 3-variant clusters and the results are provided in Table III. Figure 12 compares the Von-Mises strain with the degree of variant selection (DVS) observed experimentally normalized as

$$DVS = [P_{exp}(i) - P_{random}(i)]/P_{random}(i)$$

where $P_{exp}(i)$ is the experimentally observed frequency for a given variant cluster and $P_{random}(i)$ is the frequency expected from a random distribution. In this formulation, a positive value would indicate a frequency of occurrence higher than the random value, and vice versa. Figure 12 indicates a) that 3-variant clusters with low values of ϵ^e are observed at frequencies greater than the random value, and also b) that the Type 2 cluster, which shares a common $\langle 111 \rangle // \langle 11 \bar{2} 0 \rangle$ axis corresponding to the delta arrangement of plates described above, has the lowest ϵ^e and shows a

frequency of occurrence that is significantly higher than other triplet clusters in relation to its ϵ^e value.

3.3.Coarsening Behavior

3.3.1 Experimental data

There appears to be no previous work that has examined the coarsening behavior of assembly of precipitates whose distribution is determined by elastic energy interactions. The coarsening behavior of the precipitate assembly discussed in the preceding sections has therefore been examined. Figure 13 plots the mean major axis and minor axis of the precipitates measured from the OIM scans against coarsening time. Since it is possible that the data averaged from all variants represents a mean value of clusters that were coarsening at different rates because of their different elastic energies (as discussed in the previous section) the mean value of dimensions of all plates constituting each misorientation pair (Table I) was separately examined. This was achieved by identifying all variant pairs with a given misorientation within the nearest neighbor distance, and obtaining the dimensions of all such plates by obtaining major and minor axes of the representative ellipses fitted to these plates using TSL software. It was found that these values show the same time dependence as that of the average of all the plates taken together. An example of the pairs related by the Type B misorientation, that is the most energetically favored cluster, is included in Figure 13.

It would be inappropriate to carry out an analysis of coarsening from the dimensions obtained from such 2d sections since these dimensions do not represent the true dimensions of 3d plates from which such sections are obtained. The shape of the particles as deduced from our 3d reconstructions (Fig. 5) suggests that they can be represented as triaxial ellipsoids of variable size and eccentricity. Measurement on 2D sections of such triaxial ellipsoids with variable size and shape does not allow the reconstruction of the true 3D distributions [14]. However Orive [14,15] offers a solution for oblate or prolate **spheroids** of variable size and shape for volume fractions less than 10%. Based upon Fig. 5, the α particles could be approximated to oblate spheroids since the aspect ratio of the disc shaped particles lie between 1.5 and 1.3 at short and long ageing times. Our attempt to use this method was not successful at our volume fraction of about 30% α since we obtained physically unrealistic values

for some of the derived parameters of this method. An alternative solution is offered by DeHoff [16] without the restriction on volume fraction, based upon the assumption that the eccentricity is invariant, that is the ratio of thickness to the diameter of the disc shaped particles or oblate spheroids is invariant, in the distribution of sizes of any data set. Figure 14 shows the distribution of major axis and eccentricity, as measured in these 2d sections for a short and long coarsening time. An examination of this figure suggests that the eccentricity is tightly distributed about a mean value, and that such an assumption may be valid in our case. With this second assumption, we have therefore used the Dehoff method to derive the ellipsoidal distribution from our 2D measurements, and show the results as a function of ageing time in Figure 15. We have examined the fit of the data to various power law fits against time ranging from a dependence of $t^{1/2}$ (interface controlled coarsening) to $t^{1/3}$ (matrix diffusion controlled coarsening) and find that the data can best fitted to two regions of coarsening behavior. The major axis coarsens rapidly at first and then at slower rate linearly with $t^{1/3}$. The minor axis remains constant in an initial short duration (during the period of rapid coarsening of the major axis) and then increases linearly with $t^{1/3}$. The aspect ratio increases rapidly during the initial transient and then decreases slowly with time. The change in the experimental observed frequencies of Type A and Type C variants with time seem in Figure 6c corresponds to this initial transient. We note that the mean minor axis is not directly derived in the Dehoff method. It is obtained from an aspect ratio of the most unequiaxed particle in the data set corresponding to the largest measured major axis. It is assumed that the statistics of section sampling is good enough that the largest major axis measured in the data set is obtained from a section in which the major axis of the oblate spheroid lies in that section.

3.3.2 Phase field analysis

The problem of understanding the coarsening behavior of large volume fractions anisotropic precipitates has several aspects [17–19] that are not taken into account in the classical LSW theory. The first of these relates to the extensively discussed effect of volume fraction (see as examples [20,21]) At the high volume fractions of precipitates considered here, with significant amounts of precipitate contact (Figure 4), the diffusional fields will be completely different from that considered in the LSW

formulation and indeed there is no analytical solution that will capture the varying diffusional field in such a situation. Phase field models for coarsening may be used to explore this effect, but have primarily addressed coarsening in the γ/γ' system and explored the evolution of γ' morphology and distribution based on elastic effects with isotropic surface energy. We explore in this section a modified version of a 2-dimensional phase field model [22] in which the anisotropic surface energy form is initially configured to produce an aspect ratio in 2 dimensions that approximates experimental cross-sectional images. 12 orientation variants, represented by multiple phase-fields (produced by rotating each plate by 30°), are randomly distributed in the simulation. The volume fraction is also representative of that experimentally investigated. The model does not consider varying equilibrium sizes, elastic interactions or kinetic anisotropy, and assesses solely the effect of shape anisotropy at large volume fractions on the coarsening process in order to examine whether the trends in the experimental results can be reproduced under these conditions. Figure 16 shows the results of the model. The major and minor axes both vary linearly as a function of $t^{1/3}$. Figure 17 shows corresponding images.

4. Discussion

We have investigated variant distribution and morphology of the α phase that precipitates with the Burgers orientation relationship with the β matrix in the Ti5553 alloy for the characteristic ‘delta’ distributions that are commonly realized in all β rich alloys for relatively low temperature thermal paths to α precipitation. As Figure 1 shows, these delta distributions are characterized by triangular shaped arrangements of α plates that suggest intervariant interactions that lead to preferred distributions. The experimental data indicates a strong preference for variants that are related by intervariant misorientation characterized by the axis angle pair $\langle 11\bar{2}0 \rangle 60^\circ$ (see Table 1). Triplets of α phase with a common axis $\langle 11\bar{2}0 \rangle$ (arising from the $\langle 111 \rangle$ direction in parent β) form the delta pattern and are present at frequencies higher than expected from random values both in pair-wise distributions and as clusters of 3 variants (Figure 10-12). Our analysis based on shape and spatial orientation independent (non-configurational) elastic interaction energies shows, somewhat counterintuitively, that this distribution is preferred over others only from the

intervariant interaction arising from the residual elastic energy of semicoherent α , and not from coherent α phase. Figure 18 shows that the α phase in our investigations is indeed semicoherent and indeed coherent α has been observed only rarely [7] suggesting that the α nucleus loses coherency at a very early stage in its growth process. Our results then suggest that these characteristic distributions are formed due to preferred variant dissolution during the simultaneous growth and coarsening of semicoherent precipitates following nucleation. Indeed, the increase in the frequency of Type C variants at the expense of Type A pairs during extended coarsening supports this hypothesis. However alternative viewpoints must also be considered. It is possible that a configuration dependent energy calculation would show a lower energy in the coherent case for the Type B related 3-variant cluster, or these distributions could be formed due to autocatalytic nucleation processes that align new nuclei with existing semicoherent nuclei, that is due to elastic interactions between between semicoherent precipitates and new coherent nuclei. Alternatively, it is possible that defect mediated nucleation [23] may also create such 3-variant clusters. Finally, Figure 8 suggests the possibility that the three variants may nucleate simultaneously in an autocatalytic manner (rather than grow and meet during coarsening) to form the pyramid that is seen from the 3d image reconstruction (see for example [24]). These 3 variants would share a common $\{10\bar{1}1\}$ plane as described in the figure. In this case the energy calculations should include the reduction in surface energy due to the low energy α/α interfaces in a configuration dependent calculation. We are at present not able to distinguish between these possibilities.

The literature contains relatively few examples of the coarsening behavior of anisotropic precipitates at large volume fractions [17–19]. The coarsening behavior of the delta distribution (Figure 13) indicates that the self- accommodated structures analyzed in this work show no difference in the time dependence of coarsening of specific variant clusters. Figure 15 shows that after an initial transient, both the major axis and the minor axis of the disc shaped plates coarsen in accordance with conventional coarsening laws. We note that the samples were aged starting from a preexisting α distribution shown in Figure 2. It is suggested that the initial transient is associated with an adjustment of volume fraction to the significantly lower values associated with the coarsening temperature examined. The subsequent time

dependence is in accordance with the 2d phase field simulations shown in Figure 16. Thus the significant anisotropy of shape, and high volume fractions of precipitate in a network with a high degree of interparticle contact, has no effect on the conventional dependence on time in diffusion controlled coarsening. Clearly also, any anisotropy associated with the kinetics of growth of differently oriented precipitate/matrix interfaces [18,19] has not influenced coarsening behavior. However the aspect ratio is observed to decrease with time with a tendency to saturate at long ageing times. For precipitates with non spherical shapes arising from elastic or interface energy anisotropy, the equilibrium shape can change with increasing size as elastic energy effects begin to dominate over the interface energy [11,17]. A second effect that can arise from significant inter-precipitate contact is that the effective surface energy driving the evolution of shape must include interfaces between precipitates. The effect of these two factors on the aspect ratio is yet to be understood. Clearly however the observed variation of aspect ratio does not translate to a strong dependence of the coarsening rate constant with time such that the conventional relationship of precipitate dimensions with $t^{1/3}$ is still retained

5 Conclusions

1. The variant distribution of α in a Ti5553 alloy aged to form characteristic delta distributions has been analyzed, and a dominant 3-variant cluster in which pairs are related to each other by an axis-angle pair $\langle 11 \bar{2} 0 \rangle 60^\circ$ has been observed.
2. Shape and spatial orientation independent elastic self and interaction energies for all pairwise and triplet combinations of α precipitates been calculated for both the coherent and semicoherent state. It is found that the pairwise combination and 3-cluster combination that is experimentally observed most frequently has the lowest energy for the semicoherent state.
3. The delta distribution follows classical coarsening kinetics after an initial transient

Acknowledgements

DB of the Indian Institute of Science acknowledges funding by Boeing Company, USA. The support of K.K. Sankaran, Don Shih and Jim Cotton is gratefully acknowledged. These authors also acknowledge with gratitude the supply of the alloy by the Defence Metallurgical Research Laboratory. YW of OSU is grateful to the Department of Materials Engineering of the Indian Institute of Science for the Brahm Prakash Chair professorship during which several aspects of this work were carried out. RS and YW also would like to thank financial support from the National Science Foundation under the DMREF program with Grant No. DMR-1435483. DB is grateful to Prof. Arun Gokhale of the Georgia Institute of Technology for bringing to his attention publications dealing with the derivations of 3d distributions of ellipsoids. SB and DB thank Prof. Abinandanan of IISc for several suggestions on analysis. Several suggestions from the reviewer were also incorporated into the paper. We are grateful for these comments.

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Figure 2: The starting structure for the aging treatments.

Figure 3: The effect of aging time at 765C on α size and distribution, BSE images.

Figure 4: The effect of aging time at 765°C on α size and distribution, phase partitioned EBSD images.

Figure 5: The effect of aging time at 765°C on the typical 3d morphology of α plates a) 4 hours b) 144 hours.

Figure 6: a) The frequency of occurrence of different variant pairs based on a ‘non-correlated analysis’ of the scanned area using TSL software for different aging times at 765°C . The expected frequencies for random distributions are indicated by horizontal lines b) The frequency of occurrence of different variant pairs as a function of aging time at 765°C , based on a spatially correlated analysis using a nearest neighbor coordination of α plates based on the mean α spacing at each aging temperature.

Figure 7: Phase partitioned EBSD images of two 3-variant clusters using a common $\langle 11\bar{2}0 \rangle$ direction in each case as shown by the arrows.

Figure 9: The frequency of occurrence of various types of 3 variant clusters in comparison with values expected from random distributions.

Figure 10: The density of the configurationally independent part of the total elastic strain energy, $\langle B_{pq}(\hat{n}) \rangle$, for the 6 types of pairs in a) the coherent α case. and b) the semicoherent α case.

Figure 11: The Von-Mises strain for the 6 types of pairs in a) the coherent α case and b) the semicoherent α case.

Figure 12: A comparison of the Von-Mises strain (SFTS) for the 19 3-variant cluster types in the semicoherent α case with the degree of variant selection (DVS) as defined in the text.

Figure 13: The average major and minor axis and aspect ratio from 2d metallographic section plotted against coarsening time. The major and minor axes were determined from these metallographic sections by fitting ellipses to the phase partitioned ebsd images of all variants of the α phase. The plots show the mean dimensions of all variants as well as the mean dimensions of the variant pairs that are most frequently observed experimentally, that is variants related by the Type B misorientation.

a

b

Figure 14: Plots of major axis and eccentricity determined from metallographic sections after (a) 4.5 hours and (b) 144 hours.

Figure 15: The estimated mean major and minor axes and aspect ratio (major axis/minor axis) of oblate spheroids derived from the data of Figure 13. The major axis was obtained employing [16], and minor axis from the aspect ratio of the most unequiaxed particle at each ageing time.

Figure 16: The average major and minor axes from 2d phase field simulations. The arrows indicate the time units for which images are given in Figure 17.

Figure 17: Images from 2d phase field simulations corresponding to (a) starting structure (b) 60000 time units (c) 990000 time units in Figure 15. The color coding refers to 12 orientation variants.

Figure 18: The semicoherent interface between the α and β phases.

Type	Axis/angle pair	Number of possible combinations	Probability of occurrence
Type A	0	12	15.38%
Type B	[11-20]/60°	12	15.38%
Type C	[-1.377,-1, 2.377, 0.359]/60.83°	24	30.77%
Type D	[-10 5 5 3]/63.26°	12	15.38%
Type E	[1, -2.38, 1.38, 0]/90°	12	15.38%
Type F	[0001]/10.53°	6	7.69%

Table I: The 6 types of pair-wise misorientation between α plates in the Burgers orientation relationship and their probability of occurrence in the random case. There are 78 possible pair-wise combinations (allowing repetition) of the 12 variants that are distributed as shown in these 6 types of pairs.

Aging Time (hours)	Frequency of 2-variant clusters					
	Type A	Type B	Type C	Type D	Type E	Type F
.2	0.15	0.42	0.13	0.09	0.08	0.05
4.5	0.08	0.44	0.21	0.06	0.08	0.04
144	0.09	0.43	0.18	0.09	0.07	0.05

Table II: The experimentally observed spatially correlated frequencies of the 6 pair-wise combinations.

Type of triplet	Misorientation			Number of combinations	Frequency		SFTS for semicoherent plate combinations
					(Random)	(Measured)	
	V1V2	V2V3	V1V3				
1	Type A	Type A	Type A	12	0.033	0.021	.0674
2	Type B	Type B	Type B	4	0.011	0.05	.0195
3	Type C	Type C	Type C	8	0.022	0.025	.0224
4	Type D	Type D	Type D	4	0.011	0.001	.0253
5	Type A	Type B	Type B	24	0.066	0.097	.042
6	Type A	Type C	Type C	48	0.132	0.062	.043
7	Type A	Type D	Type D	24	0.066	0.008	.044
8	Type A	Type E	Type E	24	0.066	0.02	.0565
9	Type A	Type F	Type F	12	0.033	0.017	.0673
10	Type B	Type C	Type C	12	0.033	0.052	.0215
11	Type B	Type C	Type D	24	0.066	0.097	.0225
12	Type B	Type C	Type E	24	0.066	0.12	.0337
13	Type B	Type C	Type F	24	0.066	0.119	.0424
14	Type B	Type D	Type E	24	0.066	0.089	.0343
15	Type C	Type C	Type D	12	0.033	0.025	.0234
16	Type C	Type C	Type E	24	0.066	0.075	.0343
17	Type C	Type D	Type E	24	0.066	0.046	.0349
18	Type C	Type D	Type F	24	0.066	0.048	.0435
19	Type E	Type E	Type F	12	0.033	0.03	.0564
TOTAL				364			

Table III: The 19 3-variant clusters. The second column shows the misorientation type between each pair of variants in these clusters. The 364 possible combinations of 3-variant clusters of the 12 α variants are distributed as shown in these 19 types. The measured frequencies of each type are compared with the random frequency of occurrence and the Von-Mises strain in the semicoherent case.

Von Mises strain and frequency of observation of α variant clusters