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OF TITANIUM ALLOYS WITH A COLONY-ALPHA
MICROSTRUCTURE (Preprint)**

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**Processing Section
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BRANCH ELIMINATION DURING HEAT TREATMENT OF TITANIUM ALLOYS
WITH A COLONY-ALPHA MICROSTRUCTURE

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Abstract - The kinetics of the dissolution of lamellar branches in an alpha-beta titanium alloy with a colony-alpha microstructure were determined using a series of heat treatments at 900 and 955°C. The experiments revealed that the branch recession rate was constant. An approximate diffusion analysis was developed to describe the diffusion process, and corresponding model predictions showed good agreement with measurements.

The conversion of titanium-alloy ingots to produce billet products comprises a series of thermomechanical processing steps, each of which is designed to bring about a metallurgical transformation such as recrystallization or spheroidization. One of the most important operations for two-phase alpha-beta titanium alloys consists of the hot working and subsequent annealing of semi-finished products in the two-phase field used to spheroidize a colony-alpha lamellar microstructure. Such a microstructure is produced during air cooling following hot working and recrystallization in the high-temperature beta field.

Spheroidization of colony alpha may be accomplished during hot deformation (i.e., dynamically) via localized shearing of lamellar platelets or during static heat treatment following deformation via thermal grooving/termination migration. In either case, the ease of spheroidization is heavily dependent on the thickness of the alpha lamellae [1-5]. Thick lamellae require greater strains for dynamic spheroidization and longer times for static

spheroidization. Hence, an understanding of the factors which control the thickness of alpha platelets during preheating operations is important with regard to overall process design.

Thickening of alpha lamellae during static heating occurs by mass transport which results in a reduction in overall surface area and thus surface energy. The transport may occur between a lamella and so-called “branches” which are attached to it (Figure 1) as well as between adjacent lamellae due to a classical coarsening-type process. The former mechanism, i.e., branch elimination, is analogous to the phenomenon of “fault migration” treated previously in the literature [6, 7] and is relatively easy to quantify inasmuch as the entity which dissolves (a branch or lamellar fault) forms a well defined geometric relation with the lamella or lamellae onto which its mass is transferred. By contrast, classical coarsening of an aggregate of lamellae is a much more difficult problem because of the irregular plan-view shapes of typical alpha platelets, the complex spatial arrangement of the platelets within a colony relative to each other, and the irregular shape of the colonies themselves [8].

As a first effort in developing a quantitative description of platelet thickening, the kinetics of the elimination of branches during the heat treatment of the alpha/beta titanium alloy Ti-6Al-4V were determined and interpreted in terms of an approximate diffusion analysis. The program material comprised 12.5-mm-diameter hot-rolled bar with a measured composition (in weight percent) of 6.33 aluminum, 4.07 vanadium, 0.19 iron, 0.16 oxygen, 0.01 carbon, 0.01 nitrogen, 0.005 hydrogen, balance titanium. The beta-transus temperature (temperature at

which $\alpha + \beta \rightarrow \beta$) for this material was 995°C. The bar was given a heat treatment consisting of 940°C/10 min. + 1065°C/15 min. + furnace cool to develop a colony-alpha microstructure.

To establish the branch-recession rate, samples measuring 63-mm in length were cut from the bar, encapsulated in individual quartz tubes that were evacuated and backfilled with argon, and heat treated at a temperature of 900 or 955°C for times ranging from 0.25 to 20 hours followed by water quenching. After heat treatment, each sample was sectioned, prepared using standard metallographic procedures, and examined using backscattered-electron (BSE) imaging in a scanning electron microscope. The length (L_b), thickness (T_b), and spacing (Y_b) (Figure 1) of at least twenty branches for each heat-treatment condition were taken from digital micrographs. Because of the irregular shape of the lamellar platelets and associated branches, the distribution of the values of L_b was likely not very sensitive to the sectioning plane. On the other hand, the use of perpendicular distances on the micrographs for T_b and Y_b yielded *overestimates* of the actual values because of stereological considerations, and are discussed further below.

Figure 2 shows a typical branch-elimination observation. A thickening of the lamella “upstream” from the receding branch which is attached to it is apparent. Measurements of average branch dimensions for the heat treatments at 955°C are summarized in Table I and Figure 3. As expected, the *average* length of the branches decreased with increasing time (Table I). However, the rate of decrease in length did not appear to be constant. This anomaly can be

ascribed to the fact that short branches are totally eliminated during short times, and hence care must be exercised in comparing length measurements taken at different times. For example, the measured distribution for ~20 branches after heat treatment at 955°C for 0.25 or 2 h is shown in Figure 3a. There appears to be a decrease in length of ~13.2 μm between 0.25 and 2 h, yielding a recession rate of $13.2/1.75 \approx 7.5 \mu\text{m/h}$. When comparing the data for 0.25 and 2 h, therefore, branches whose length was less than ~15-20 μm after 0.25 h were removed from the distribution for the shorter time. When this was done, a true recession rate of $17.3/1.75 \approx 10 \mu\text{m/h}$ was obtained (Figure 3b). The validity of this value was confirmed by noting that the longest branches at 0.25 h were ~60 μm (Figure 3b), thus requiring $\sim 60/10 = 6$ h to be eliminated. This time lies between 4 h and 8 h for which branches were or were not observed, respectively (Table I). For the heat treatments at 900°C, similar data analysis yielded a measured branch recession rate of ~4 $\mu\text{m/h}$.

The observations were rationalized on the basis of an approximate one-dimensional diffusion analysis analogous to that used by Courtney and Malzahn Kampe [9] and Semiatin, et al. [5] for various spheroidization problems. For the present problem, it was assumed that mass transport occurs from the tip of the alpha-phase branch onto the adjacent lamellae due to the concentration gradient associated with the difference in curvature, i.e., the Gibbs-Thompson effect [10]. Mass transport between the lateral faces of the branch and lamella to the saddle (located where the branch is connected to the lamella) was neglected because of

the substantially longer diffusion distances required to maintain the essentially zero curvature of these faces.

The branch and lamellae are considered to be semi-infinite in the direction perpendicular to the plane of Figure 1. Applying the Gibbs-Thompson equation for the concentration difference between the branch and adjacent (approximately flat) lamella and taking the diffusion distance as $Y_b + T_b/2$, the concentration gradient $\partial c/\partial x$ is given by the following approximate expression:

$$\frac{\partial c}{\partial x} = \frac{C_\beta V_M \gamma_{\alpha\beta}}{RT(Y_b + T_b/2)} \times \left(\frac{1}{T_b/2} \right) \quad (1)$$

in which $C_\beta \equiv$ concentration of the rate-limiting solute, $V_M \equiv$ molar volume, $\gamma_{\alpha\beta} \equiv$ alpha/beta interface energy, $R \equiv$ gas constant, $T \equiv$ absolute temperature. The flux per unit breadth of the branch/lamella is obtained from Fick's Law and the circumference of the semi-circle that forms the tip of the branch. This volume transport rate is then equivalent to the rate of decrease of the volume (per unit breadth) of the branch, i.e.,

$$\text{Flux/breadth} = D \frac{\partial c}{\partial x} \times \frac{\pi T_b}{2} = - \frac{d}{dt} (L_b \times T_b) \quad (2)$$

in which $D \equiv$ diffusivity of rate-limiting solute.

Strictly speaking, Equations (1) and (2) are valid only when the matrix phase and second phase are terminal solid solutions. For Ti-6Al-4V, neither the alpha nor the beta phase is a terminal solid solution. In this case, two corrections are needed; one is required for the Gibbs-Thomson equation used in deriving Equations (1) and the other to account for the actual amount of solute needed for the dissolution of the branch [10]. As described in Reference 10, these two

corrections are respectively (1) $\{(1-C_\beta) / [(C_\alpha-C_\beta)(1 + \partial \ln r / \partial \ln C_\beta)]\}$, in which the second parenthetical term in the denominator is a thermodynamic factor ($r \equiv$ activity coefficient of the solute in the beta phase) and (2) $(C_\alpha-C_\beta)$. In both terms, C_α denotes the concentration of the rate-limiting solute in the alpha phase. The term C_β in Equation (1) is thus multiplied by the *quotient* of these two terms, i.e.,

$$\text{Composition Factor, } C_F = \frac{C_\beta(1-C_\beta)}{(C_\alpha-C_\beta)^2 [1 + \partial \ln r / \partial \ln C_\beta]} \quad (3)$$

Corrections such as that given in Equation (3) are particularly important for alloys such as Ti-6Al-4V in which there is only a small composition difference, $C_\beta - C_\alpha$, between the phases, thus leading to a large acceleration of the interfacial-energy-driven process [10].

Assuming T_b is constant, the final relation for the rate of branch recession obtained by combining Equations (1) – (3) is the following:

$$\frac{dL_b}{dt} = \frac{\pi D C_F V_M \gamma_{\alpha\beta}}{RT T_b (Y_b + T_b/2)} \quad (4)$$

The observed rate of recession of branches at 955°C was interpreted in the context of Equation (4). For this purpose, average values from Table I were used for T_b and Y_b , viz., 1.66 and 1.86 μm , respectively. The diffusion-limiting solute was taken to be vanadium per previous work [11], and the value of D was selected accordingly, i.e., 0.052 $\mu\text{m}^2/\text{s}$. The values of C_F (= 61.3), V_M ($=1.044 \times 10^{-5} \text{ m}^3/\text{mol}$), and $\gamma_{\alpha\beta}$ ($= 0.4 \text{ J/m}^2$) were also taken from previous work [11]. These

inputs for the material/geometric parameters yielded a predicted recession rate of 3.3 $\mu\text{m/h}$.

The predicted value of the branch recession rate at 955°C, 3.3 $\mu\text{m/h}$, is substantially less than the measurement of ~ 10 $\mu\text{m/h}$. There are two main sources for this discrepancy, the neglect of the motion of the saddle in a direction opposite to that of the tip of the branch and stereological considerations. The first source is probably small as noted above. The second, or stereological source, is probably more important. In particular, the actual branch thickness T_b and spacing Y_b are actually *smaller* than the values in Table I due to a section-plane effect. The analysis in Reference 12 suggests that the true thickness/spacing are each smaller by a factor of approximately 1.5. (Similar, simple estimates of the average of $\sec\theta$ for θ between 10 and 80 degrees suggest that the factor is of the order of 2.) Inserting a correction of 1/1.5 for both T_b and Y_b in Equation (4) increases the predicted recession rate to 7.4 $\mu\text{m/h}$, a value comparable to that measured.

Performing a similar analysis, the recession rate at 900°C was predicted to be 3.8 $\mu\text{m/h}$, in excellent agreement with the measured value of ~ 4 $\mu\text{m/h}$.

In conclusion, the kinetics of the elimination of lamellar branches in Ti-6Al-4V with a colony-alpha microstructure have been quantified and modeled to a reasonable accuracy using a relatively simple diffusion approach. The importance of the elimination of the shortest branches when estimating the branch-recession rate and stereological effects in quantifying branch geometry have been underscored.

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References

1. I. Weiss, F.H. Froes, D. Eylon, and G.E. Welsch: *Metall. Trans. A*, 1986, vol. 17A, pp. 1935-1947.
2. E.B. Shell and S.L. Semiatin: *Metall. and Mater. Trans. A*, 1999, vol. 30A, pp. 3219-3229.
3. N. Stefansson, S.L. Semiatin, and D. Eylon: *Metall. and Mater. Trans. A*, 2002, vol. 33A, pp. 3527-3534.
4. N. Stefansson and S.L. Semiatin: *Metall. and Mater. Trans. A*, 2003, vol. 34A, pp. 691-698.
5. S.L. Semiatin, N. Stefansson, and R.D. Doherty: *Metall. and Mater. Trans. A*, 2005, vol. 36A, pp. 1372-1376.
6. L.D. Graham and R.W. Kraft: *Trans. TMS-AIME*, 1966, vol. 236, pp. 94-102.
7. H.E. Cline: *Acta Metall.*, 1971, vol. 19, pp. 481-490.
8. N. Vanderesse, E. Maire, M. Darrieulat, F. Montheillet, M. Moreaud, and D. Jeulin: *Scripta Mater.*, 2008, vol. 58, pp. 512-515.
9. T. H. Courtney and J.C. Malzahn Kampe: *Acta Metall.*, 1989, vol. 37, pp. 1747-1758.
10. J.W. Martin, R.D. Doherty, and B. Cantor: *Stability of Microstructure in Metallic Systems*, Cambridge University Press, Cambridge, UK, 1997.

11. S.L. Semiatin, B.C. Kirby, and G.A. Salishchev: *Metall. Mater. Trans. A*, 2004, vol. 35A, 2809-2819.
12. H.J.G. Gundersen, T.B. Jensen, and R. Osterby: *J. of Microscopy*, May 1978, vol. 113, part 1, pp. 27-43.

Table I. Measurements of the average branch thickness (T_b), branch length (L_b), and spacing between the branch and the lamella to which it is attached (Y_b)

Temp (°C)	Time (h)	Y_b (μm)	T_b (μm)	L_b (μm)
955	0.25	1.92	1.50	29.7
955	1	2.49	2.08	22.2
955	2	1.39	1.33	19.2
955	4	1.62	1.73	17.5
955	8	--	--	--

Figure Captions

- Figure 1. Schematic illustration of the branch elimination mechanism and geometry in a material with a lamellar microstructure.
- Figure 2. BSE micrograph of the microstructure in a Ti-6Al-4V sample heat treated at 955°C for 0.25 h and water quenched. The dark (platelet) phase is alpha, and the light-gray matrix was beta at the heat-treatment temperature. A typical branch is circled in the micrograph.
- Figure 3. Branch lengths in Ti-6Al-4V samples heat treated at 955°C for 0.25 h or 2 h: (a) Measured distribution and (b) distributions after removing the four shortest branch lengths from the measured distribution for 0.25 h.

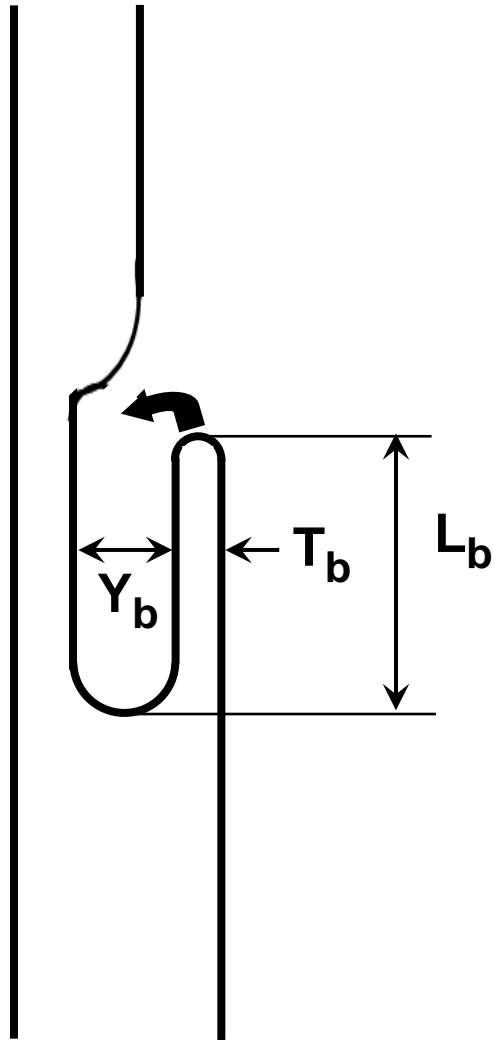


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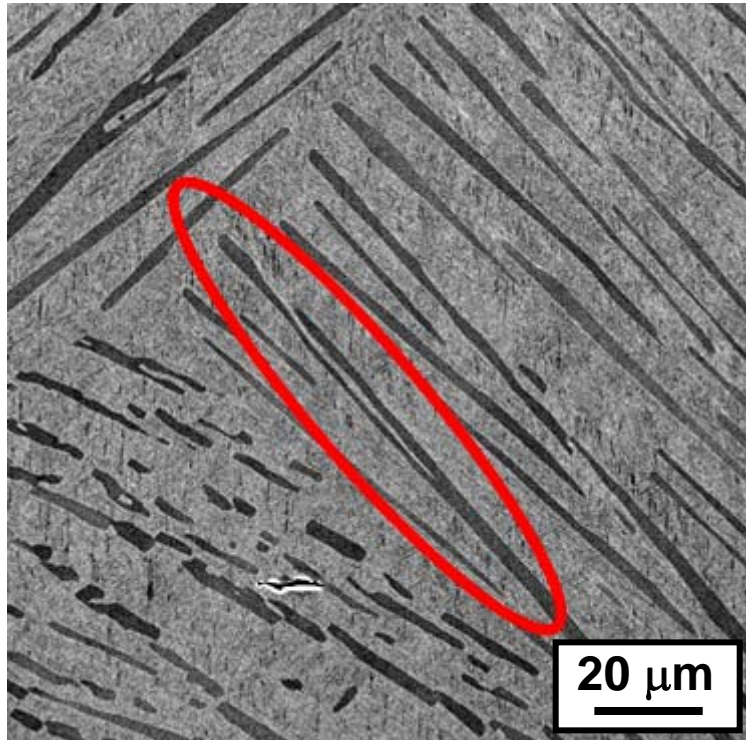


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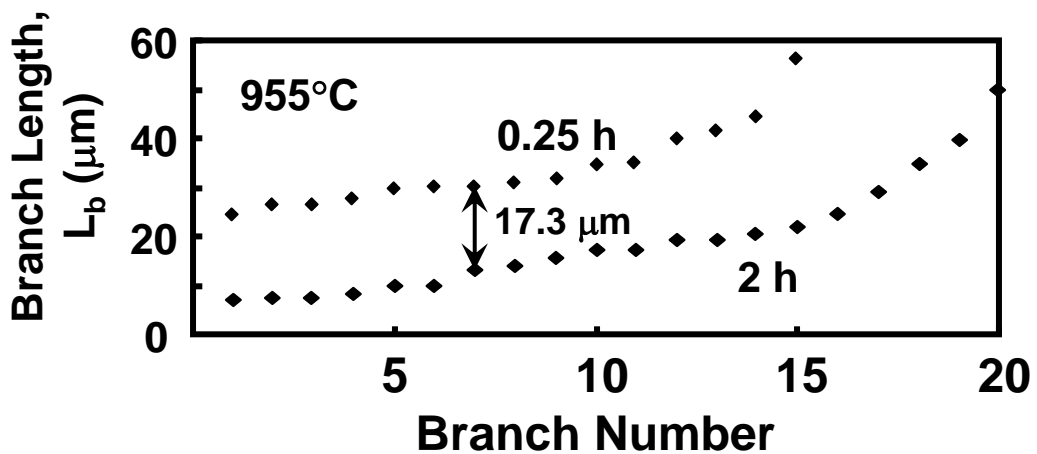
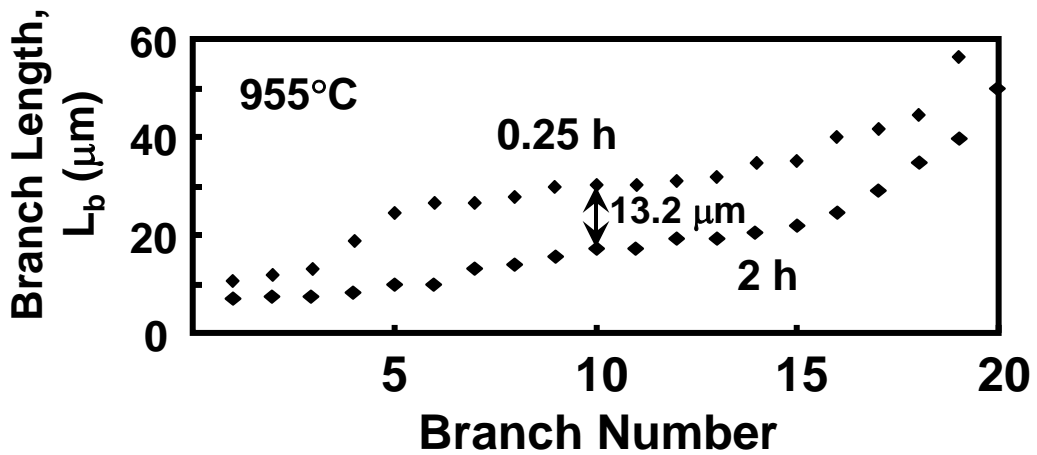


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