Effects of Combined High and Low Temperature Deformation Processing of β III Titanium

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The structure and properties of β III titanium alloy (nominal composition: 11.5 pct Mo, 6 pct Zr, 4.5 pct Sn, bal Ti) were studied as a function of combined high and low temperature thermomechanical processing. A water quenched extrusion was deformed various amounts by swaging at room temperature prior to the aging treatment. No re-solution heat treatment was employed. The swaging introduced mechanical twinning and a small amount of stress induced orthorhombic martensite. Following a 900° F, 8 hr aging treatment, substantial increases in yield and tensile strength were observed, combined with a severe decrease in tensile ductility in samples with small amounts of swaging. The orientation and morphology of the deformation products have a critical influence on tensile ductility. A decrease in the plane strain fracture toughness accompanied the large increase in tensile strength.

THE β III titanium alloy (nominal composition: 11.5 pct Mo, 6 pct Zr, 4.5 pct Sn) is cold formable in the solution-treated condition and in the overaged condition. The formability of this alloy has been described in detail by Guernsey, Peterson, and Dulis¹ and by Peterson, Guernsey, and Johnson.² High temperature thermomechanical processing of round to round β III extrusions has been described by Adair and Roberson,³ who found a direct influence of the hot-worked structure on the properties after aging. The effects were more pronounced when a re-solution treatment was omitted. Feeney and Blackburn,⁴ and Blackburn and Feeney⁵ have discussed the effects of cold deformation on the microstructure and properties of β III, and they have presented some quantitative information on the effects of cold deformation on aged properties.

The dependence of the aged properties on the microstructure resulting from both hot and cold deformation is of considerable importance in the utilization of the β III titanium alloy. The present investigation was undertaken to determine the properties resulting from the combined effects of hot and cold deformation without an intermediate solution treatment prior to aging.

Our previous results³ for high temperature deformation alone indicated that water quenching immediately following extrusion was beneficial to mechanical properties. Aging of samples from these extrusions without a solution heat treatment resulted in superior mechanical properties over either water quenched or air-cooled extrusions which were solution heat-treated and water quenched prior to aging. These results were interpreted to mean that at least a part of the hot-worked dislocation structure was retained by quenching, and that this microstructure promoted a more favorable aging response. Sil cock^6 has reported a similar effect in cold-worked Ti-Mo alloys.

EXPERIMENTAL PROCEDURE

A 3 in. diam by 6 in. long billet of the β III titanium alloy was extruded to a 0.97 in. round from a billet preheat temperature of 1800°F and water quenched, using the same procedures as previously described.³ This billet was from the same heat of material, and the reduction ratio was nearly the same. The reduction in area was 89.5 pct vs 90.7 pct for the previous work. The extruded bar was sectioned to appropriate lengths, and pieces exclusive of the nose and tail regions were swaged at room temperature to produce reductions in area of 5, 24, 41, and 58 pct. Following machining of fracture toughness specimens and rough machining of tensile specimens, all specimens were aged in protective envelopes for 8 hr at 900° F and air cooled. The cylindrical tensile specimens were then finish machined to a 1 in. gage length and a gage section diameter of 0.252 in. Load-strain curves were determined through the use of a clip-on extensometer. A crosshead speed of 0.05 ipm was used. Plain strain fracture toughness values (K_{IC}) were determined from slow bend testing, with three point loading, as discussed by Steigerwald.⁷ Specimens with the standard Charpy geometry were used. They were precracked by fatigue loading.

Metallographic specimens were abraded through 600 grit paper, then electropolished at -30° C in a solution of 64 pct methanol, 15 pct 2-butoxyethanol, and 21 pct perchloric acid. The polishing potential was 30 v. Specimens were etched in an aqueous solution of 1 pct HF and 50 pct $HNO₃$.

RESULTS AND DISCUSSION

Cold deformation of the 1800 $^{\circ}$ F water quenched β III extrusion prior to the 900° F, 8 hr aging treatment produced significant changes in mechanical properties, considerably greater than those attributed to the influence of the retained hot-worked structure alone. 3 The observed variation in properties is not proportional to the amount of swaging. The tensile and K_{IC} results for deformed and aged specimens are presented in Fig. 1. The data shown for zero cold defor-

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Fig. 1-Mechanical properties of the β III titanium alloy after cold swaging and aging at 900° F for 8 hr.

mation are from previous results.³ Note from Fig. 1 that the tensile ductility returns to normal levels as the amount of cold deformation increases, while *KIC* does not continue to decrease.

Since both the strength level and the K_{IC} values are reasonably constant at the greater percent reductions by swaging, the *KIC* values are less sensitive to the microstructural variations induced by this processing history than is the tensile ductility. Since plane strain fracture necessarily proceeds with a minimum of plastic flow, the factors influencing fracture toughness and tensile ductility are not always the same. Our previous results³ for fracture toughness vs ultimate tensile strength, when extrapolated to the present strength level, correspond to the present values of fracture toughness. However, we suspect that the relationship is fortuitous because when the alloy is processed by forging⁸ rather than by extrusion, the properties do not fit this relationship. For the present investigation, the low tensile ductility at the 5 and 24 pct cold reduction level is believed to result from solute segregation upon aging, which occurs at the boundaries of the deformation products which are formed during the room temperature swaging.

Previous workers^{$4,9$} have not observed a thermallyinduced martensitic transformation in the β III alloy. Rack, Kalish, and Fike⁷ have presented clear evidence that the spontaneous reaction in thin foils is a twinning process in the bcc β phase. In the present work, orthorhombic martensite was observed as a result of room temperature deformation of thick sections by swaging. However, the bulk of the deformation products were mechanical twins, similar to those observed by others^{4,5,9} following cold deformation by rolling. The minor constituent was identified by elec-

tron diffraction to be an orthorhombic martensite similar to that observed by Williams and Hickman¹⁰ and Bagariatskii, Nosova, and Tagunova 11 in Ti-Mo alloys.

The results of transmission electron microscopy on an as-swaged specimen are shown in Fig. 2. Because of the small volume fraction of orthorhombic martensite, the analysis of the electron diffraction pattern, Fig. $2(d)$, was only weakly supported by the results of X-ray diffraction in cold-worked, nonaged specimens.

The diffraction pattern of Fig. $2(c)$ was indexed on the basis of an orthorhombic cell with $a = 3.01$, b = 4.82, and $c = 4.54\text{\AA}$. The a and b values correspond to the values reported by Bagariatskii $et al.¹¹$ for Ti-Mo alloys when extrapolated to the present molybdenum content. The value for c reported here is about 2 pct less than the extrapolated value. In view of the differences in alloy content, the agreement seems quite reasonable. Attempts to index this pattern on various other unit cells were unsuccessful. Blackburn and Feeney⁵ reported the observation of orthorhombic martensite in β III with lattice parameters of $a = 3.12$, $b = 4.86$, and $c = 4.71\text{\AA}$.

The loss of tensile ductility which was observed to accompany small amounts of cold deformation prior to aging, Fig. 1, is believed to be an indirect result of the stress-induced transformations. When the 900°F aging treatment is performed, the heavily-deformed areas age more rapidly than the matrix. During this process they reject large quantities of molybdenum which should stabilize the adjacent β phase. A similar reaction occurs in non-cold deformed β III during aging, but the α particles in this case are much smaller. Since the β phase is much weaker than the normal two phase mixture, a fairly long, narrow path of easy fracture may be created between the twins, which have an abnormally high concentration of large α particles. The orientation of the twins should, therefore, have a pronounced effect on the tensile ductility of coldworked and aged material. The amount and morphology of α within the twins are a result of an increased aging rate, either by heterogeneous nucleation or by pipe diffusion in the heavily dislocated regions. Either process would allow the mechanical twins and orthorhombic martensite to approach equilibrium composition much more rapidly than the surrounding matrix.¹²

The variation in the orientation and morphology of the twins in cold deformed and aged specimens is shown in Fig. 3. The first twins which are formed seem to be preferentially aligned at an angle of about 60 deg to the tensile (or swaging) axis; as deformation continues, they seem to rotate to become preferentially aligned parallel to the tensile axis. Thus, if a plane of easy fracture does exist, as postulated earlier, its effect on tensile ductility should decrease with larger amounts of prior cold work as shown in Fig. 1. It may also be noted that the twins become twisted, bent, branched, and intertwined at larger amounts of deformation. These observations indicate that the first twins which form are actually reoriented and deformed as swaging proceeds. Also, it is expected that significant amounts of slip in the matrix would accompany the larger amounts of deformation.

The results of inverse pole figure determinations on transverse sections of these specimens are presented in Table I.

Fig. $2(a)$ -Transmission electron micrograph, longitudinal section from specimen swaged to 5 pct RA, no aging treatment. Bright field; magnification 17,050 times. (b) Same area as above, dark field, magnification 17,050 times. Operating reflection indicated in $2(d)$ as M002. (c) Selected area diffraction pattern of second platelet from the right, 1 in. from the top. Operating reflection for $2(b)$ indicated by spot with halo. (d) Schematic analysis of $2(c)$.

In this table, the integrated intensity for any reflection is shown as the ratio of *Ihkl* to the total integrated intensity for the specimen. Since each specimen was scanned over the same angular range, the results are directly comparable. This technique has been described by Harris. 13 If we restrict our attention to the reflections from the α planes alone, then the data may be transformed as follows: $Ihhl/\Sigma I_{\alpha} = 0.23, 0.27,$ 0.28, 0.35 for 010, and 0.56, 0.50, 0.46, 0.47 for α 011 and for A, B, C, and D, respectively. These results show that as deformation proceeds, the prism planes of the α formed on aging are more preferentially oriented in the transverse direction. As this occurs, the basal planes will favor the longitudinal direction.

Since these variations in texture were accompanied by variations in grain shape and stress induced transformation morphology, it is not possible to isolate the influence of crystallographic texture on mechanical pr operties.

The results of transmission electron microscopy on an aged specimen with a 5 pct reduction by swaging are shown in Fig. 4. Basically, two different morphologies of α within the twins are shown. In the bright field micrograph, Fig. $4(a)$, several different orientations and sizes of α are seen within the twin. This structure of α was more frequently observed than the finer, more uniform α shown in the dark field images of Figs. $4(b)$ and $4(c)$. The diffraction pattern associated with these dark field images and a schematic of it are shown in Figs. $4(d)$ and $4(e)$ respectively. The analysis of the diffraction pattern, Fig. $4(e)$, shows three α orientations. Two of these have zone axes

Fig. 3--Photomicrographs of longitudinal sections of deformed and aged specimens, magnification 261 times. Bar axis and tensile axis is from left to right in each case. Amount of cold swaging prior to aging at 900°F for 8 hr: (a)-5 pct, (b)-24 pct, (c) -41 pct, (d) -58 pct.

 (c) (*d*)

[121] and are twin-related in the matrix. The third orientation of α is present in the twin. The explanation for the twin relation of α in the matrix is unknown at this time. The dark field image showing the twin in bright reflection, Fig. $4(b)$, is taken from the spot indicated as (002) in the schematic. No other spot except (002) produced bright reflections in the twin. All of the other spots are, therefore, associated with α in the matrix. The other dark field image, Fig. $4(c)$, is taken from the spot indicated as (101) in the schematic. These results show a high volume fraction of α within the twins. This α seems continuous when compared with the smaller α particles in the matrix.

It seems unlikely that the low tensile ductility associated with the 5 and 24 pct cold swaged and aged specimens can be attributed to the formation of ω during aging. Previous work³ has shown that the presence of the retained hot-worked structure tends to retard ω formation and promote α formation upon aging at 900° F.

 ω is a hexagonal phase commonly present in several titanium binary alloys. It was first reported in Ti-Mo alloys by Silcock.⁶ It is precipitated during quenching, and it has the orientation $[0001]_{\omega}$ || $[111]_{\beta}$,

[1120] $_{\omega}$ || [110] $_{\beta}$. The lattice spacing of ω is related to that of β by $a_{\omega} = \sqrt{2} a_{\beta}$, $c_{\omega} = \sqrt{3}/2 a_{\beta}$. The occurrence of ω in β III has been reported by previous ${\rm workers.}^{3-5,9}$

Since the overall structure becomes more uniformly strained as the amount of room temperature swaging increases, it is expected that the aging would be more uniform also. This is in addition to the reorientation of the twins formed earlier. The combined effect could account for the recovery of tensile ductility even though the strength level remains quite high.

Observations of the fracture behavior of the tensile specimens were made. Polished and etched surfaces whose plane is parallel to the tensile axis are shown

(a)

(b)

Fig. 4-Transmission electron micrographs, magnification 18,700 times. (a) Bright field. (b) Dark field image, operating reflection indicated as (002) in $4(e)$. (c) Dark field image, operating reflection indicated as (101) in $4(e)$. (d) Diffraction pattern. (e) Analysis of $4(d)$.

in Fig. 5 for specimens having 5 and 58 pct cold reduction by swaging prior to the aging treatment. The fracture path is from left to right on the upper surfaces shown. Figs. $5(a)$ and $5(b)$ are from a 5 pct reduction specimen, and Fig. $5(c)$ is from a 58 pct reduction specimen. It may be seen that a large amount of void nucleation and growth has occurred in the area adjacent to the fracture surface in the specimen with the unfavorable twin morphology, Figs. $5(a)$ and $5(b)$. Furthermore, much of the void area is straightsided and parallel to the twins. It is reasonable to believe that the void nucleation and growth was much more intense in the fracture path. In the specimen with the more favorable twin morphology, Fig. $5(c)$, some void nucleation still occurred. In this case, however, the orientation of the tensile stress was such that only minor amounts of void growth occurred. Also, there is no direct evidence in Fig. $5(c)$ that the voids are associated with the boundaries of the twins. These observations are considered to substantiate the previous discussion on the role of the morphology of the stress-induced transformation products on the tensile ductility. It should be noted that these twins do not promote low ductility in unaged specimens.

(b)

Fig. 5-Photomicrographs of broken tensile specimens, $5(a)$ and *5(b),* 5 pct reduction prior to aging, magnification 260 times; (c) 58 pct reduction prior to aging, magnification 260 times.

Feeney and Blackburn⁴ and Williams, Hickman, and Marcus¹⁴ have reported that void nucleation and growth is in fact the dominant fracture mode in β III. This sort of process was first suggested by Silcock⁶ as a fracture mode in Ti-Mo alloys containing the intermediate ω phase. The process has been subsequently investigated both theoretically and experimentally by others, and it has been found to be a common occurrence in ductile materials containing hard particles.¹⁵

SUMMARY AND CONCLUSIONS

1) The response of the β III titanium alloy to a 900°F 8 hr aging treatment after combined high and low temperature thermomechanical processing results in exceptionally high strength levels, somewhat at the expense of tensile ductility and fracture toughness.

2) Room temperature swaging of a β III titanium

extrusion which had been water quenched immediately following the 1800° F extrusion operation caused much mechanical twinning and some stress-induced orthorhombic martensite. The quantity and orientation of the stress-induced transformation products was influenced by the amount of reduction by swaging.

3) The orientation of the stress-induced transformation products with respect to the tensile axis coupled with solute segregation during aging is believed to be responsible for the low tensile ductility observed in specimens with small amounts of swaging prior to the aging treatment.

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REFERENCES

- 1. J. B. Guernsey, V. C. Peterson, and E. J. Dulis: *Metals Progr.,* 1969, vol. 96, pp. 121-24.
- 2. V. C. Peterson, J. B. Guernsey, and H. A. Johnson: AIAA Paper No. 68-976, 1968.
- 3. A. M. Adair and J. A. Roberson: *Proc. Second Int. Conf. on the Strength of Metals and Alloys,* Vol. I11, p. 932, American Society for Metals, Metals Park, Ohio, 1970.
- 4. J. A. Feeney and M. J. Blackburn: *Met. Trans.,* 1970, vol. 1, pp, 3309-23.
- 5. M. J. Blackburn and J. A. Feeney: *J. Inst. Metals*, 1971, vol. 99, pp. 132-34.
- 6. J. M. Silcock: *Acta Met.,* 1958, vol. 6, pp. 481-93.
- 7. E. A. Steigerwald: *MetalsProgr.,* 1967, vol. 92, pp. 96-101.
- 8. A. M. Adair: Air Force Materials Laboratory, W-PAFB, Ohio, unpublished research, 1971.
- 9. H. J. Rack, D. Kalish, and K. D. Fike: *Mater. Sci. Eng.,* 1970, vol. 6, pp. 181- 98.
- 10. J. C. Williams and B. S. Hickman: *Met. Trans.*, 1970, vol. 1, pp. 2648-50.
- 11. lu. A. Bagariatskii, G. 1. Nosova, and T. V. Tagunova: *Soy. Phys.-Dokl.,* 1959, rot. 3, pp. 1014-18.
- 12. J. C. Williams: Science Center, North American Rockwell Corp, Thousands Oaks, Calif., private communication, 1971.
- 13. G. B. Harris: *Phil. Mag.,* 1952, vol. 43, pp. 113-23.
- 14. J. C. Williams, B. S. Hickman, and H. L. Marcus: *Met. Trans.,* 1971, vol. 2, pp. 1913-19.
- 15. J. R. Low, Jr.: *Eng. Fract. Mech.,* 1968, vol. 1, pp. 47-53.