



AFRL-RX-WP-TM-2010-4168

**COLLABORATIVE RESEARCH AND DEVELOPMENT
(CR&D)**

**Delivery Order 0002: Deformation Mechanisms During Hot Working
of Titanium**

Ayman Salem

Universal Technology Corporation

S.L. Semiatin

Metals Branch

Metals, Ceramics & NDE Division

OCTOBER 2005

Final Report

Approved for public release; distribution unlimited.

See additional restrictions described on inside pages

STINFO COPY

**AIR FORCE RESEARCH LABORATORY
MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE**

NOTICE AND SIGNATURE PAGE

Using Government drawings, specifications, or other data included in this document for any purpose other than Government procurement does not in any way obligate the U.S. Government. The fact that the Government formulated or supplied the drawings, specifications, or other data does not license the holder or any other person or corporation; or convey any rights or permission to manufacture, use, or sell any patented invention that may relate to them.

This report was cleared for public release by the Air Force Research Laboratory Wright Site Public Affairs Office (PAO) and is available to the general public, including foreign nationals. Copies may be obtained from the Defense Technical Information Center (DTIC) (<http://www.dtic.mil>).

AFRL-RX-WP-TM-2010-4168 HAS BEEN REVIEWED AND IS APPROVED FOR PUBLICATION IN ACCORDANCE WITH THE ASSIGNED DISTRIBUTION STATEMENT.

*//Signature//

MARK GROFF
Program Manager
Business Operations Branch
Materials & Manufacturing Directorate

//Signature//

KENNETH A. FEESER
Branch Chief
Business Operations Branch
Materials & Manufacturing Directorate

This report is published in the interest of scientific and technical information exchange, and its publication does not constitute the Government's approval or disapproval of its ideas or findings.

*Disseminated copies will show “//Signature//” stamped or typed above the signature blocks.

REPORT DOCUMENTATION PAGE					Form Approved OMB No. 0704-0188	
The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.						
1. REPORT DATE (DD-MM-YY) October 2005		2. REPORT TYPE Final		3. DATES COVERED (From - To) 02 May 2003 – 30 October 2005		
4. TITLE AND SUBTITLE COLLABORATIVE RESEARCH AND DEVELOPMENT (CR&D) Delivery Order 0002: Deformation Mechanisms During Hot Working of Titanium					5a. CONTRACT NUMBER F33615-03-D-5801-0002	
					5b. GRANT NUMBER	
					5c. PROGRAM ELEMENT NUMBER 62102F	
6. AUTHOR(S) Ayman Salem (Universal Technology Corporation) S.L. Semiatin (AFRL/RXLM)					5d. PROJECT NUMBER 4349	
					5e. TASK NUMBER L0	
					5f. WORK UNIT NUMBER 4349L0VT	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Universal Technology Corporation 1270 North Fairfield Road Dayton, OH 45432-2600 Metals Branch (AFRL/RXOB) Metals, Ceramics & NDE Division, Air Force Research Laboratory Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command, United States Air Force					8. PERFORMING ORGANIZATION REPORT NUMBER S-531-0002	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Air Force Research Laboratory Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command United States Air Force					10. SPONSORING/MONITORING AGENCY ACRONYM(S) AFRL/RXOB	
					11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S) AFRL-RX-WP-TM-2010-4168	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.						
13. SUPPLEMENTARY NOTES PAO Case Number: AFRL/WS-06-2356; Clearance Date: 30 Sep 2006. Research for this report ended in 2005.						
14. ABSTRACT This research in support of the Air Force Research Laboratory Materials and Manufacturing Directorate was conducted at Wright-Patterson AFB, Ohio from 2 May 03 through 30 Oct 05. The research examined the stress-strain responses for individual a/B colonies of Ti-6Al 1-4V established via uniaxial compression testing at 815 °C for crystals oriented for single slip along specific slip systems. Some samples were designed to enforce the highest Schmidt factor on (a, a, and a) slip on either basal or prism planes. Others were loaded parallel to the c-axis to activate pyramidal slip in the a-phase. The high-temperature stress-strain response showed lower yield strength (soft prism) compared to slip along a (hard prism). Similar anisotropy was observed for basal slip. However, the flow stress for all basal slip systems were always higher than prismatic slip. This anisotropy is a result of the orientation relationship between the a and B phases and the associated misalignment of slip directions in both phases.						
15. SUBJECT TERMS titanium, high temperature deformation, single-colony crystals						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT: SAR	18. NUMBER OF PAGES 16	19a. NAME OF RESPONSIBLE PERSON (Monitor) Mark Groff	
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER (Include Area Code) N/A	

Deformation Mechanisms During Hot Working of Titanium

A.A. Salem* and S.L. Semiatin

Air Force Research Laboratory, Materials and Manufacturing Directorate,
AFRL/MLLM, Wright-Patterson AFB, OH 45433

1. ABSTRACT

The stress-strain responses for individual α/β colonies of Ti-6Al-4V were established via uniaxial compression testing at 815°C for crystals oriented for single slip along specific slip systems. Some samples were designed to enforce the highest Schmidt factor on (a_1 , a_2 and a_3) slip on either basal or prism planes. Others were loaded parallel to the c-axis to activate pyramidal slip in the α -phase. The high-temperature stress-strain response showed significant anisotropy for different prismatic slip systems as well as different basal slip systems. Particularly, prismatic slip along a_1 showed lower yield strength (Soft Prism) compared to slip along a_2 (Hard Prism). Similar anisotropy was observed for basal slip. However, the flow stress for all basal slip systems were always higher than prismatic slip. This anisotropy is a result of the orientation relationship between the α - and β -phases and the associated misalignment of slip directions in both phases.

2. INTRODUCTION

Dual phase α/β titanium alloy, Ti-6Al-4V, has been the workhorse for numerous aerospace applications for many decades. Hot working in the $\alpha+\beta$ field (forging, extrusion, rolling, etc.) is the main shaping technique for wrought billet or bar material. Hot working processes are effectively designed via constitutive modeling and plastic flow analysis. Successful predictions of these models are strongly dependent on accurate descriptions of the material constitutive behavior within the specified temperature regime. Although, establishing the constitutive behavior of single-phase material showed a lot of progress in recent years, there is limited information available to describe the constitutive behavior of the two-phase alloys. Upon cooling Ti-6Al-4V alloy from the β -field, the α -phase (hcp) nucleates within the β -phase (bcc) following an orientation relationship (OR) in which $(0001)_\alpha$ is parallel to $(101)_\beta$ and $[2-1-10]_\alpha$ is parallel to $[1-1-1]_\beta$.

Earlier investigations by Chan et al. [1] reported room-temperature deformation behavior of a two-phase α/β titanium alloy, Ti-8Al-1Mo-1V, with a colony microstructure. Compression tests of small samples cut from a plate material with large grain/colony sizes revealed that the critical resolved shear stresses for the prism $\langle a \rangle$ and basal $\langle a \rangle$ were similar, but lower than that for pyramidal $\langle c+a \rangle$ slip systems. Schmidt law failed for every orientation tested except for the specific case where prism slip occurred parallel

* Corresponding author (e-mail: ayman.salem@wpafb.af.mil)

Permanent address: Universal Technology Corp., Dayton, OH 45432

to the broad face the α - β interface. Recently, Suri et al [2] reported similar anisotropy during room temperature deformation of single colonies of Ti 6242.

3. EXPERIMENTAL PROCEDURE

Cylindrical bars containing single α - β colonies of Ti-6Al-4V were grown utilizing a vertical float zone technique in a Crystallox furnace utilizing 12 mm diameter rods in an inert Ar atmosphere (3-4 psi), with a pull rate of 2.00 mm/hr. Successfully grown colonies ranged from 5- 30 mm length. The chemical composition of the starting material (in weight percent) was 6.33 Al, 4.07 V, 0.19 Fe, 0.16 O, 0.01 N, 0.0048 H, Bal. Ti. Thin foils parallel to $(0001)_\alpha$ and $[2-1-10]_\alpha$ were extracted from the single colony rod for Transmission Electron Microscopy (TEM) to determine the precise orientation relationship between the α and β phases. These samples were dimpled and ion milled to inhibit the formation of hydride phases associated with TEM sample preparation using electropolishing techniques.

Single colonies, containing single variant of α in a single crystal of β , were oriented for mechanical testing using EBSD/SEM technique to identify the crystallographic orientation of the α phase simultaneously with the relative alignment of the β phase, allowing for the unambiguous identification of the three $\langle 11-20 \rangle$ slip directions (figure 1). SEM observations were conducted on a Leica-SEM operated at 20 kV and 10 nA. Samples were held in a special fixture designed to fit inside the SEM and then carried to the electrical discharge machining (EDM) with minimum misalignment (figure 2). Surfaces parallel to the basal plane were cut from the single colony using the EDM. Samples were reoriented and then cut such that the final samples have the compression axis tilted 45° from the desired Burger's vector to be activated. Final grinding and electropolishing removed surface damage and the recast layers after EDM. Final sample sizes were in the order of 3 X 3 X 5 mm with the loading axis aligned with the long side of the samples.

A Philips XRG back-reflection Laue camera operating at 40 kV and 200 mA were used to confirm the final orientation of samples. In addition, the samples oriented for pyramidal and basal slip was oriented using the Back-Reflection Laue technique integrated with a software for indexing and simulating Laue patterns (OrientExpress). The use of the software minimized time and material needed to obtain the desired orientation. In particular, the Laue pattern of the rod material was indexed by OrientExpress to identify the starting orientation then simulations were used to predict the required rotations to enforce the highest resolved shear stress on a specific slip system. The predicted rotations were then applied on the real material using the goniometry supplied with the X-ray machine. The final measured Laue pattern is compare to the predicted one and then fine-tuning was done before cutting final samples with EDM (figure 4).

The compression samples were each oriented to maximize the resolved shear stress on one of the three unique $\langle a \rangle$ slip direction in the α phase on either basal slip plane or

prismatic slip plane. The samples were designated as slip direction followed by slip plane (e.g. a_1 -prism).

Samples oriented for prismatic slip had the basal plane parallel to the compression axis thus preventing slip on basal plane. A slip direction was aligned at 45° angle to the compression axis which resulted in the highest resolved shear stress on that slip direction.

Samples oriented for basal slip had a 45° angle between basal plane and the compression axis. One of the faces of the compression sample was parallel to the prism plane containing the a -type slip direction to be activated. This results in a zero resolved shear stress for the motion of the Burgers vector on its corresponding prism slip plane.

Cut samples were hand ground flat to a final grit size of 800 and then electropolished in a solution of 10 ml perchloric acid and 90 ml methanol at -30°C using a DC power supply with 28 volts for 2 minutes.

Constant strain rate simple compression tests were conducted at 815°C using an MTS machine at strain rate of 0.01/s to strains of 0.15. Samples were lubricated using glass slurry that reduced oxidation. Immediately after testing, samples were water quenched to inhibit recovery.

SEM observations of slip traces on the tested samples were investigated using Leica SEM operating at 20 kV and 8 nA. Thin foils were cut from the tested samples for TEM observation along $(0001)_\alpha$. A Philips CM200 LaB6 microscope operating at 200 kV was used for TEM investigations.

4. RESULTS AND DISCUSSION

The SEM micrographs of single colonies oriented for prism slip shows thin β lamellae running across the $(0001)_\alpha$ face of the sample (figure 3). In agreement with Suri et al. observations in Ti 6242 alloy [2], the β lamellae have irregular honeycomb morphology rather than a plate-like as (see the faces normal to the $(0001)_\alpha$). The observed near Burgers OR for α/β titanium alloys [2,3,4] has the effect of creating three unique hcp- $\langle a \rangle$ slip vectors in the colony structure (Figure 5). The Burgers OR is $(101)_\beta \square (0001)_\alpha$ and $[1-1-1]_\beta \square [2-1-10]_\alpha$. However, Suri et al. [2] reported a deviation from the Burgers OR in single α/β colonies of Ti6242 with a 0.7° misorientation between a_1 and b_1 , and a misorientation of 11.1° between the a_2 and b_2 direction. The third slip direction a_3 in the α -phase does not have a closely aligned $\langle 111 \rangle_\beta$ direction in the colony structure. The three $\langle a \rangle$ slip directions were always observed to be aligned at the same angle to the broad face of the β lamella. The angle between the a_1 slip vector and the broad face was observed to be 15° . Similarly, the a_2 and a_3 directions were shown to form 75° and 45° with the broad face. Consequently, the relative position of the β lamellae in the $(0001)_\alpha$ surface can be used to identify (hence, orient) the different slip directions $\langle a \rangle$ in the α phase.

The stress strain response for single colonies oriented for prismatic slip showed a significant anisotropy between a_1 -prism and a_2 -prism (Figure 6). The yield strength of a_2 -prism was 35% higher than the yield strength of a_1 -prism. In the rest of the report, the former will be designated as hard-prism and the later as soft prism. The hard-prism test showed higher flow stress associated with significant flow softening. In contrast, soft-prism had almost constant flow stress. The variation in yield strength and work hardening for single-colony crystals tested at room temperature had been reported for Ti-1-1-8-1 [1] and Ti-6246 [2]. They reported an increase in the yield strength for colony crystals where the slip direction is nearly normal to the α/β interface as is the case for colony a_2 -prism which is in agreement with the current observation. Two specific colonies 22 and 3 in the work by Chan et al. [1] and OA and OB in the work of Suri et al. [2] are the closest to a_1 -prism and a_2 -prism respectively. Although the current tests were conducted at 815°C, there is a significant agreement in the anisotropy of the yield strength with material tested at room temperature [1, 2]. A major difference is the flow softening observed for the current high temperature tests.

The anisotropy of deformation behavior between soft-prism and hard-prism samples could be explained by the Burgers OR between α and β phases in Ti alloys [2,3,4]. TEM [2] (figure 5) showed that a_1 slip direction in the α phase is almost parallel to b_1 slip direction in the β phase with small misalignment (0.56°) [2,4]. However, the a_2 slip direction in the α phase has a significant misalignment with b_2 direction in the β phase (11.5°). Hence, the α/β interface could provide different resistance to slip transmission between α and β phases. Therefore, slip along a_2 slip direction is expected to face higher resistance (hence higher yield strength) than slip along a_1 slip direction. High resistance for slip along a_2 slip direction is expected to be associated with dislocation pile-up at the α/β interface. As a result, dynamic recovery of piled dislocation is expected during hot compression of sample deformed along a_2 -slip direction. On the other hand, the α/β interface is expected to have minimum resistance to slip transmission during slip along a_1 which reduces dislocation pile-ups and consequently minimizing flow softening (figure 6).

Similar anisotropy was observed in the yield strength of samples oriented for basal slip (figure 7). However, the hard-basal had a yield strength that is only 4% higher than that for soft-basal. The flow stress of both colonies exhibited flow softening.

A comparison between the mechanical behavior of prism and basal slip shows that the flow stresses for basal slip were always higher than that for prism slip (figure 8). The rate of flow softening during deformation of the hard basal sample was much slower than that for hard prism. Moreover, soft-basal showed flow softening while the soft prism had minimum flow softening. The rates of flow softening of hard and soft basal were almost the same.

Back-Scattered SEM imaging did not reveal any shearing of the α/β interface in samples oriented for a_1 -prism slip (figure 9) in contrast to Suri et al [2] observations of interface shearing due to slip transmission at room temperature.

5. CONCLUSIONS

The float-zone technique succeeded in producing single colonies of α/β Ti-6Al-4V. The mechanical behavior of colonies oriented for different prismatic slip and basal slip showed significant anisotropy during hot compression tests at 815°C. Slip along a_2 slip direction resulted in higher yield strength than slip along a_1 slip direction for the same slip plane. For the same slip direction, slip on basal plane had higher yield strength than slip on the prismatic plane. Flow softening was observed for slip along a_2 slip direction with the highest softening rate during slip on prism plane. Deformation along a_1 -prism had almost constant flow stress.

6. ACKNOWLEDGEMENTS

This work was conducted as part of the in-house research activities of the Metals Processing Group of the Air Force Research Laboratory's Materials and Manufacturing Directorate. The support and encouragement of the Laboratory management and the Air Force Office of Scientific Research (Dr. J. Tiley, program manager) are gratefully acknowledged. The yeoman assistance of J.M. Scott and P.N. Fagin in conducting the experimental work are much appreciated. AAS was supported through Air Force Contract F33615-03-D-5801.

7. REFERENCES

1. Chan, K.S., Wojcik, C.C. and Koss, D.A., Metall. Trans. (1981) Vol. 12A, 1899
2. Suri, S., Viswasnathan, G.B., Neeraj, T., Hou, D.-H., and Mills, M.J., Acta mater., (1999) Vol 47, No. 3, 1019.
3. Furihara, T., Ogawa, T. and Maki, T., Phil. Mag. Lett., (1995), Vol. 72, 175.
4. Mills, M.J., Hou, D.H., Suri, S., and Viswanathan, G.B., Boundaries and interfaces in Materials, TMS, Warrendale, PA (1998), 295.

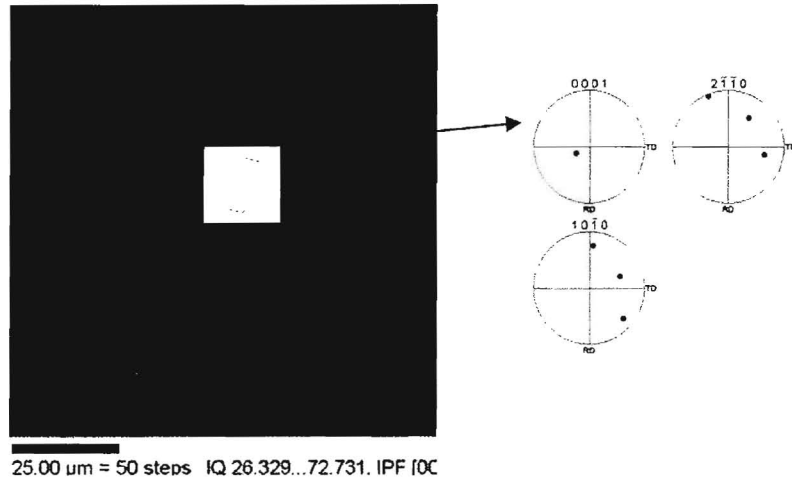


Figure 1. Using OIM technique to identify the exact orientation of α -phase. (a) IPF map for the compression axis (ND) and (b) the associated PF.

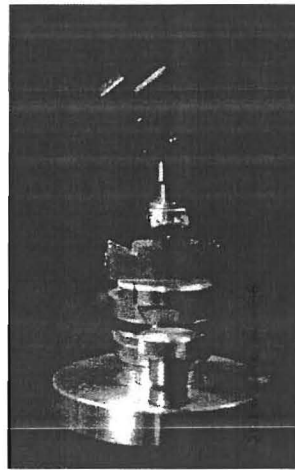


Figure 2. Fixture used to hold samples to be cut by EDM after determining the orientation using OIM technique.

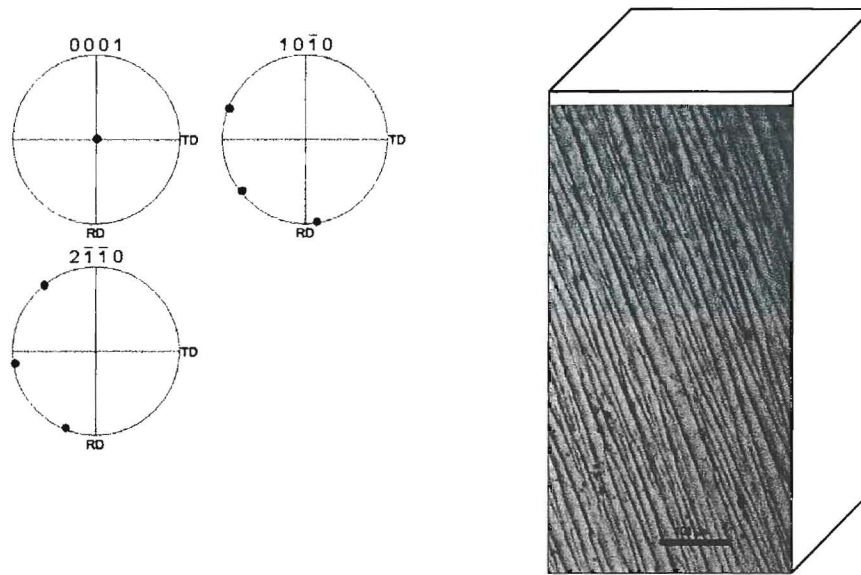


Figure 3. Sample oriented and cut for a2-prsim slip with the a_2 at 45° tilt from the compression axis (vertical direction in the micrograph).

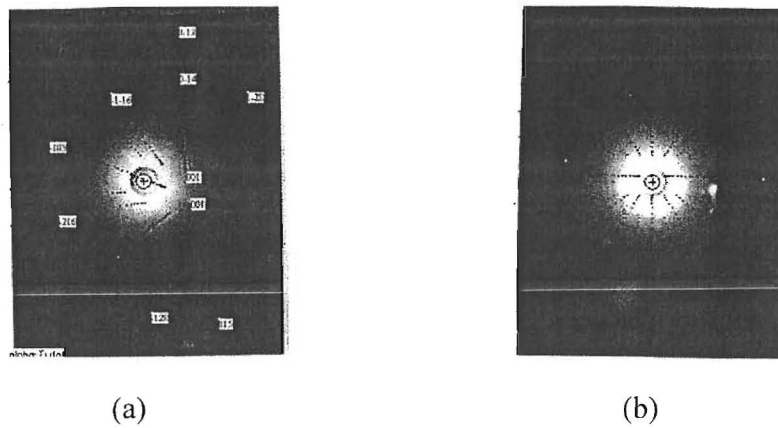


Figure 4. (a) Determining the orientation of single colonies using OrientExpress. (b) Simulated rotations to bring the c-axis in the direction of the X-ray beam.

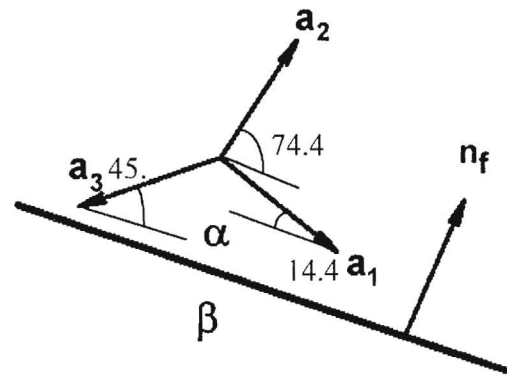
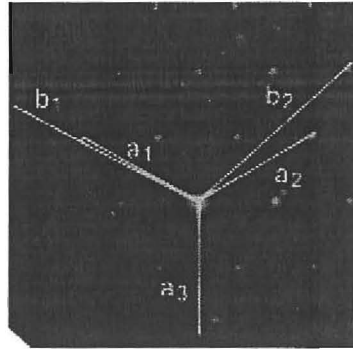


Figure 5. Diffraction pattern shown α and β slip directions with the associated misalignment for single colonies of Ti6242 [2].

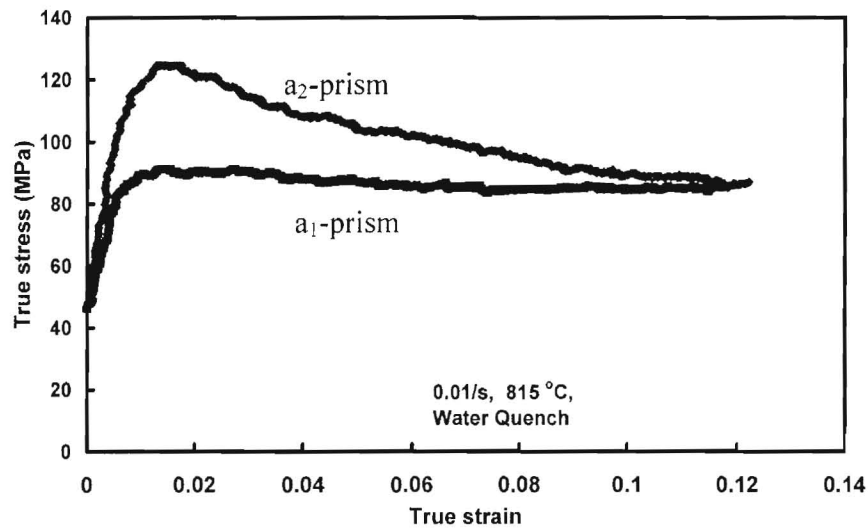


Figure 6. Constant strain rate stress-strain response for single colonies of Ti-6Al-4V oriented for prismatic slip in the α -phase under simple compression at 815°C.

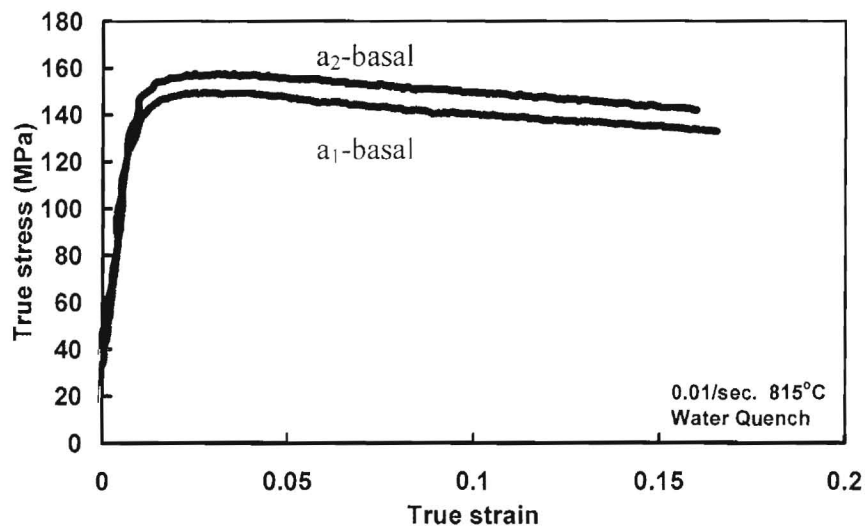


Figure 7. Constant strain rate stress-strain response for single colonies of Ti-6Al-4V oriented for prismatic slip in the α -phase under simple compression at 815°C.

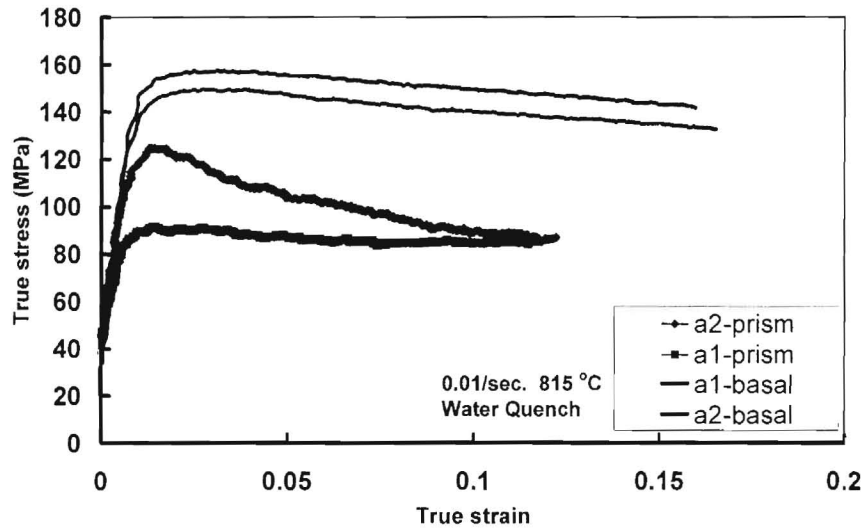


Figure 8. Constant strain rate stress-strain response for single colonies of Ti-6Al-4V oriented for prismatic slip and basal slip in the α -phase under simple compression at 815°C.



Figure 9. Back-scatter SEM image for soft-prism sample after hot compression at 815°C to true strain of 0.12 followed by water quenching. No interface sharing was observed anywhere in the sample.