Reports File
E-19-613
[illegible]

**GEORGIA INSTITUTE OF TECHNOLOGY**
OFFICE OF RESEARCH ADMINISTRATION

# RESEARCH PROJECT INITIATION

Date: September 26, 1972

Project Title: Deformation Mechanisms in Beta Titanium Alloys

Project No: E-19-613

Principal Investigator Dr. E. A. Starke, Jr.

Sponsor: Office of Naval Research

Agreement Period: From July 1, 1972 Until June 30, 1973

Type Agreement: Task Order No. N00014-67-A-0159-0015

Amount: $35,883.00

Reports Required: Annual Progress Report; Final Technical Report

Sponsor Contact Person (s):

Technical Matters
Director, Metallurgy Program
Material Sciences Division
Office of Naval Research
800 North Quincy Street
Arlington, Virginia 22217

Contractual Matters
(Thru ORA)
Mr. R. J. Whitcomb
ONR Resident Representative
Hinman Research Building
Campus

Assigned to: School of Chemical Engineering

COPIES TO:

| | |
|---|---|
| Principal Investigator | Library |
| School Director | Rich Electronic Computer Center |
| Dean of the College | Photographic Laboratory |
| Director, Research Administration | Project File |
| Director, Financial Affairs (2) | |
| Security-Reports-Property Office | |
| Patent Coordinator | Other |

RA-3 (6-71)

E-19-613


# GEORGIA INSTITUTE OF TECHNOLOGY

ATLANTA, GEORGIA 30332

SCHOOL OF
CHEMICAL ENGINEERING


January 9, 1973

SUBJECT: End-of-The-Year-Report
ONR Task-Order No.
N00014-67-A-0159-0015
NR 031-750

TO: Dr. W. G. Rauch, Director
Metallurgy Program
Material Sciences Division
Office of Naval Research
Department of the Navy
800 North Quincy Street
Arlington, Virginia 22217

REFERENCE: (A) 471:WGR:
NR 031-750
13 December 1972

ENCL: (a) Three (3) copies of Subject Report

1. In accordance with your letter, Reference (A), we hereby submit the enclosure (a) report.

Very truly yours,

Edgar A. Starke, Jr.
Principal Investigator

EAS:np
cc: Office of Naval Research Resident Representative
Room 276
Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

End of the Year Report

ONR Task Order No. N00014-67-A-0159-0015
NR 031-750

January 8, 1973

A. Deformation Mechanisms in Beta Titanium Alloys

1. This study is involved in determining the relationship between the microstructure of beta titanium alloys, as influenced by solute content, and the mechanical properties and deformation modes of the metastable alloys. The solute content influences the microstructure by its effect on: the martensitic transformation temperature (Ms and Md), the miscibility gap (or the relative shift in free energy that allows omega or beta phase separation to occur on quenching), and the twin fault probability.

2. The mechanical behavior of beta Ti-V alloys containing vanadium contents from 20 to 40 weight percent has been studied by hardness measurements, tensile tests, x-ray, optical and electron microscopy techniques. Single crystals with initial orientations of (1$\bar{1}$0) [001] and (001)[1$\bar{1}$0] and polycrystalline samples of approximately random texture were cold rolled to varying degrees. The deformed crystals, together with filings, were studied by x-ray line broadening techniques using Warren's Fourier analysis; the microstrain, particle size and twin fault probability developed during deformation being determined. The deformation textures of the deformed single and polycrystalline samples were also measured.

3. Alloys containing 20, 24, and 28 w/o V were found to contain small amounts of omega in the as-quenched condition; the amount decreasing with

increasing solute content. The twin-fault probability, B, was found to vary with vandium content, being large for both the 20 and 40% V alloys and having a minimum at 36%V. Normally the twin fault probability in bcc alloys increases with increasing solute content and this general behavior is observed in Ti-V alloys when the complicating influence of as-quenched omega is absent. However, this study shows that a small amount of omega can provide an easy means of twin formation. Optical microscopy on the gauge sections of tensile samples strained from 3 to 5 percent showed very coarse twins in the 20% V alloy. The coarseness decreased with increasing vanadium content to 28% V. Slip markings on the surface also became finer with increasing vandium content up to 32% V. Slip became coarser with larger solute contents.

These general observations were confirmed by transmission electron microscopy studies of foils prepared from the gauge sections of the tensile specimens. Extensive deformation twinning was observed in the 20% V alloy. Both primary and secondary twinning was found with primary twins varying in size from several microns to fractions of a micron. Trace analysis indicates that the twins are of the {112} <111> type; however these results do not eliminate the possibility of {332} twinning previously observed by Paton and Williams. The size and density of the deformation twins decreased with increasing vanadium content up to 32% V; the limit which has presently been studied by electron microscopy. These alloys contained well defined slip bands along with uniformly distributed dislocations. There was no evidence of any form of cell structure after the small amount of deformation given the tensile samples.

4. The deformation textures of both the single and polycrystalline

samples were found to vary considerably with vanadium content except for the (001) $[1\bar{1}0]$ oriented crystals. (001) $[1\bar{1}0]$ was found to be a stable end orientation, in agreement with previous work on bcc crystals. The single crystals which had a high twin-fault probability also showed twined-related maxima in their pole figures. For polycrystalline samples, the rolling texture of the 20% V alloy can be described by a major component (111) $[\bar{1}\bar{1}2]$ and a weak component (112) $[1\bar{1}0]$.The weak component increases in magnitude as the vanadium content increases and reaches a maximum with 28% V. Alloys containing 32% V to 40% V possessed the (001) [110] texture which is commonly found for bcc metals. This texture becomes sharper as the vanadium content increases to 40%. The (111) $[\bar{1}\bar{1}2]$ and (001) [110] textures were also found to be stable orientations for some of the single crystal specimens. After 90% reduction in thickness the polycrystalline samples were recrystallized at 900°C for one hour and water quenched. Regardless of the deformation texture, the recrystallized texture was the same for all alloys and can be described by (112) $[3\bar{1}\bar{1}]$. The recrystallized grain size did, however, vary with vanadium content; decreasing with increasing vanadium content.

5. The yield strengths, work hardening coefficients, and hardness values for as-quenched alloys, and hardness values for alloys rolled 50% and 90% have been measured. The variation of hardness with vanadium content is more or less proportional to that of the rms strain measured from filings, having its maximum value for 20 and 40% V and going through a minimum at approximately 36% V. There is a much higher increase in hardness with deformation for the 20% V alloy than for any of the others. This is due to a higher value of work hardening which can easily be correlated with the presence of a small amount of omega in the as-quenched sample of this alloy.

The yield strength was found to increase with increasing vanadium content and the work-hardening coefficient was found to decrease. The low-work-hardening of the 40% V alloy may be attributed to dynamic recovery by a twinning mechanism. The 20% V alloy also has a high twin fault probability, but for this alloy the presence of a second phase has a greater influence on work hardening.

B. Implication of ONR Contract Research in Terms of Applied Significance

The research conducted thus far on this contract has shown that the deformation mechanisms of beta titanium alloys can be greatly influenced by variations in solute content and/or the presence of small amounts of a second phase. Variations in deformation mechanisms result in variations in texture development during processing. The fundamental understanding obtained from correlations of structure, composition, deformation mechanisms and properties will aid in the design of processing parameters necessary for improved product control. In this regard, both workability during fabrication and property control of the end product are considered.

C. Publications and Reports

1. *Fu-Wen Ling and E. A Starke, Jr. "Thermal Etching of β Ti-V Alloys", Metallography 5 (1972) 399-407.

2. *Fu-Wen Ling, H. J. Rack, and E. A. Starke, Jr., "Deformation of Beta Ti-V Alloys", submitted to Metallurgical Transactions.

*Copies of these articles were submitted through Lockheed-Georgia Company to the Director, Metallurgy Programs, Material Sciences Division, Office of Naval Research on September 12, 1972 in lieu of a final report on Contract No. N00014-71-C-0412.

D. <u>Other Contracts</u>

1. "Effects of Interface Phenomena on the Mechanical Behavior of Metals", Air Force Office of Scientific Research, Co-Principal Investigator with B. R. Livesay.

2. "A Study of the Structure and Mechanical Properties of Ordered Alloys", Atomic Energy Commission, Co-principal Investigator with B. G. LeFevre.

Respectfully submitted,

Edgar A. Starke, Jr.
Principal Investigator

E-19-613

End of the Year Report

ONR Task Order No. N00014-67A-0159-0015
NR 031-750

December 17, 1973

A. Deformation Mechanisms in Beta Titanium Alloys

1. This study is involved in determining the relationship between the microstructure of beta titanium alloys, as influenced by solute content, and the mechanical properties and deformation modes of the metastable alloys. The nature and crystallographic aspects of slip, twinning, and texture development, and the correlation of these features with mechanical property measurements are being considered.

2. Single and polycrystalline beta titanium alloys, containing 20 to 40 pct V have been studied. In our last "End of the Year Report" dated January 8, 1973, we described the results obtained from the single crystal studies. The rolling textures of the crystals were shown to vary considerably with vanadium content. The results were related to certain aspects of the microstructure, i.e. the presence of small amounts of quenched-in and/or stress-induced omega, and the twin fault probability. During the past year these studies were extended to include as-quenched, and quenched and aged polycrystalline specimens of compositions similar to those used in the single crystal work.

3. Significant changes in the deformation characteristics were observed optically and by transmission electron microscopy for the as-quenched polycrystalline samples when the vanadium content was varied from 20 to 40 percent. These changes can be summarized as follows:

(a) Twinning is the predominate deformation mode for the 20% V alloy.

(b) There is a sharp drop in the ratio twinning to slip, followed by a slight rise as the vanadium content is increased.

(c) There is a smooth transition to coarser slip bands and finer twins as the vanadium content is increased.

4. Wide variations in properties as a function of alloy content for the as-quenched polycrystalline alloys, were also found when the alloys were deformed at room temperature. These property changes, which include yield strength ultimate strength, work hardening rate, deformation texture and recrystallization texture, may be related to variations in twinning probability and slip characteristics. Sheet textures in as-rolled and recrystallized materials display strongly anisotropic strength properties when measured parallel and 45° to the rolling direction. The extent of the anisotropy depends on the nature of the texture and can be qualitatively explained in terms of the Bishop-Hill model for texture strengthening. The quantitative aspects of this study are presented in detail in the publications listed at the end of this report.

5. Samples in the composition range 20 to 40 weight per cent vanadium were quenched and annealed for various intervals up to 10,000 minutes at temperatures of 300°C, 400°C and 450°C. A double-hardening effect was seen at the 400°C anneal and was most pronounced in the 20 and 25% alloys. These materials were examined by thin-foil transmission electron microscopy and selected area diffraction and also by x-ray diffraction to determine the type and distribution of phases associated with aging. The following microstructural effects were observed:

(a) A very dense distribution of cuboidal $\omega$ phase particles preceded the formation Widmänstatten $\alpha$ (Burger's $\alpha$ on $\{110\}_\beta$ planes).

(b) At the lower compositions (20 and 24% V) α formation began at prior ω interfaces and coarsened with time into platelets; whereas, for higher V compositions it formed preferentially at grain boundaries.

(c) The initial hardening is associated with the rapid formation of ω. As coherence is lost and α begins to form the hardness drops and then rises again with increasing volume fraction of α.

(d) The most pronounced hardening is associated with the initial formation of ω at the lower compositions. This is attributed to a large volume fraction of ω phase particles.

B. Implication of ONR Contract Research in Terms of Applied Significance

The research conducted thus far on this contract has shown that the deformation mechanisms of beta titanium alloys can be greatly influenced by variations in solute content and/or the presence of small amounts of a second phase. Variations in deformation mechanisms result in variations in texture development during processing. The fundamental understanding obtained from correlations of structure, composition, deformation mechanisms and properties will aid in the design of processing parameters necessary for improved product control. In this regard, both workability during fabrication and property control of the end product are considered.

C. Publications, Reports, and Talks

1. Fu-Wen Ling, J. J. Rack, and E. A. Starke, Jr. "Deformation of Metastable Beta Ti-V Alloys" Met. Trans. 4, 1671-1676 (1973).

2. Fu Wen Ling, E. A. Starke, Jr., and B. G. LeFevre, "Deformation Behavior and Texture Development in Beta Ti-V Alloys," Met. Trans. (in press).

3. B. R. Livesay and E. A. Starke, Jr., "Interactions of Dislocations with Interfaces," Acta Met. 21, 247-254 (1973).

4. E. A. Starke, Jr., G. Kralik and V. Gerold, "Plasticity of Al-Ge Single Crystals Containing Small Fractions of Precipitates," Mater. Sci and Eng. 11, 319-323 (1973).

5. K. C. Chen, Fu-Wen Ling and E. A. Starke, Jr., "Structure and Mechanical Properties of Stress-Ordered $Ni_4Mo$," Mater. Sci. and Engr. (in press).

6. "Short Range Order and the Development of Long Range Order in $Ni_4Mo$," talk presented at the International Symposium on Ordered Alloys, Tubingen, Germany, September, 1973.

7. "Plastic Deformation and Strengths of Materials," talk presented at the Alloy Selection and Failure Analysis Short Course, Atlanta, Georgia, September, 1973.

D. Degrees Granted

1. Keh-Chang Chen, Master of Science in Metallurgy, Thesis - "Structure and Mechanical Properties of Stress Ordered $Ni_4Mo$".

2. Bhaven Chakravarti, Doctor of Philosophy, Thesis - "Short Range Order and the Development of Long Range Order in Nickel 20 Atomic Percent Molybdenum Alloy".

E. Other Contracts

"The Effect of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation," Air Force of Scientific Research.

Respectfully submitted,

Edgar A. Starke, Jr.
Professor
Principal Investigator

OFFICE OF NAVAL RESEARCH

Contract N00014-67-A-0159-0015, NR 031-750

TECHNICAL REPORT 74-2

# AN X-RAY EXAMINATION OF DEFORMATION IN BETA Ti-V ALLOYS

by

Fu Wen Ling and E. A. Starke, Jr.
Metallurgy Program, School of Chemical Engineering
Georgia Institute of Technology
Atlanta, Georgia 30332

Reprinted from Metallurgical Transactions, 4 (1973)

January 8, 1974

Reproduction in whole or in part is permitted for any purpose of the United States Government.

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

## REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: 74-2

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle): AN X-RAY EXAMINATION OF DEFORMATION IN BETA Ti-V ALLOYS

5. TYPE OF REPORT & PERIOD COVERED: Technical Report

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s): Fu-Wen Ling and E. A. Starke, Jr.

8. CONTRACT OR GRANT NUMBER(s): N00014-67A-0159-0015 NR 031-750

9. PERFORMING ORGANIZATION NAME AND ADDRESS: Metallurgy Program, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, Georgia 30328

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS

11. CONTROLLING OFFICE NAME AND ADDRESS: Metallurgy Program, Office of Naval Research, 800 North Quincy Street, Arlington, Virginia, 22217

12. REPORT DATE: January 1974

13. NUMBER OF PAGES

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)

15. SECURITY CLASS. (of this report): Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report): Unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES: Reprint of article from Metallurgical Transactions, 4 (1973)

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

titanium alloys
microstructure
texture

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)


The mechanical behavior of beta Ti-V alloys has been studied by X-ray diffraction techniques. Single crystals of two orientations, containing 20 to 40 wt pct V were cold rolled varying degrees. Fourier analysis techniques were then used to determine the microstrain, particle size, and twin fault probability developed during deformation. The deformation textures of the deformed crystals were also examined and found to vary considerably with vanadium content. These results have been correlated with the observed variations in deformation modes produced by changes in solute content and microstructure.


DD FORM 1473, 1 JAN 73 — EDITION OF 1 NOV 65 IS OBSOLETE
S/N 0102-014-6601

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

# An X-ray Examination of Deformation in $\beta$ Ti-V Alloys

FU-WEN LING, H. J. RACK, AND E. A. STARKE, JR.

The mechanical behavior of beta Ti-V alloys has been studied by X-ray diffraction techniques. Single crystals of two orientations, containing 20 to 40 wt pct V were cold rolled varying degrees. Fourier analysis techniques were then used to determine the microstrain, particle size, and twin fault probability developed during deformation. The deformation textures of the deformed crystals were also examined and found to vary considerably with vanadium content. These results have been correlated with the observed variations in deformation modes produced by changes in solute content and microstructure.

RECENTLY there has been an increased interest in metastable $\beta$ titanium alloys for structural applications. These alloys offer higher strengths and better reliability (in terms of stress-corrosion resistance and fracture toughness) than do $\alpha$-$\beta$ titanium alloys. Moreover, the metastable $\beta$ alloys are more amenable to forming and shaping processes than are the $\alpha$-$\beta$ alloys. A previous study[1] of the deformation behavior of as-quenched $\beta$-III titanium (Ti-11.5 Mo-5.5 Zr-4.5 Sn) has suggested that the principal factors controlling the deformation nature in $\beta$ titanium alloys, as influenced by solute content, are: the martensitic transformation temperatures ($M_s$ and $M_d$), the occurrence of a miscibility gap (or the relative shift in free energy that allows $\omega$ or $\beta$ phase separation to occur on quenching) and the twin fault probability. The twin fault probability, in the present context, is analogous to the stacking fault energy, *i.e.*, it is a measure of the ability of a dislocation to cross-slip when it meets an obstacle. As the twin fault probability increases the ability of a dislocation to cross-slip decreases.

This paper describes the results of a study utilizing $\beta$ Ti-V alloys which was intended to more clearly delineate the relationship between solute chemistry, microstructure, and the deformation behavior of these alloys.

## EXPERIMENTAL METHODS

### a) Sample Preparation

Six Ti-V alloys, containing 20, 24, 28, 32, 36, and 40 wt pct V, were prepared by arc-melting high purity components under an argon atmosphere. The titanium was iodide crystal bar obtained from Foote Mineral Company with a total impurity content of ~1000 ppm. The vanadium had a total impurity level of ~100 ppm. The as-cast 50 g buttons were sealed in quartz under 0.5 atm partial pressure of ultrahigh purity argon, homogenized for 60 days at 1100°C, and quenched by breaking the quartz tubes under water. Large grains were then spark cut from each alloy and spark machined parallel to $\{100\}$ and $\{110\}$ planes, after which they were mechanically polished through 0.1 $\mu$ alumina followed by electropolishing using the procedure described by Rack *et al.*[1]

FU-WEN LING and E. A. STARKE, JR. are Research Scientist and Professor, respectively, Metallurgy Program, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, Ga. 30332. H. J. RACK is Member of Technical Staff, Sandia Laboratories, Albuquerque, N. M. 87115.

Manuscript submitted October 27, 1972.

### b) Mechanical Deformation

Deformation by rolling was carried out by placing the single crystals in close-fitting holes in a titanium plate of equal thickness, without top and bottom cover plates.

Rolling was carried out in increments of 10 pct reduction in thickness at room temperature, using 2.72 in. diam rolls. Samples for texture studies were initially $(1\bar{1}0)[001]$ and $(001)[1\bar{1}0]$ and the rolling direction was kept constant. Single crystals for line broadening studies were rolled in a random fashion in an effort to retain the (110) and (001) planes (which were to be used for the analysis) parallel to the surface. This attempt was unsuccessful for the (110) crystals and rotations of the [110] pole of 20 to 30 deg were observed. Measurements of the twin fault probability measurements were thus made using cold-worked powders prepared by filing at room temperature.

### c) Mechanical Tests

Diamond pyramid hardness measurements were made on electropolished, solution heat treated and quenched samples using a 1 kg load.

### d) X-ray Measurements

The $(110)_\beta$ single crystals from each alloy were examined by X-ray diffraction for the presence of the $\omega$-phase using a General Electric XRD-6 diffractometer equipped with a Warren doubly-bent LiF monochromator and automated step scanning. The monochromated $CuK_\alpha$ radiation, used in conjunction with pulse height discrimination against fluorescent radiation, and step scanning allowed the detection of less than 1 pct $\omega$. The $(110)_\beta$ crystals were oriented for diffraction from the $(222)_\beta$ planes and then examined by $2\theta$ scanning over the $(0001)_\omega$ and $(0002)_\omega$ reflections which are parallel to the $\{222\}_\beta$. The crystals were examined for $\omega$ in the as-quenched condition and for stress-induced $\omega$ after each 10 pct increment in rolling up to 50 pct reduction in thickness, and finally after 80 pct reduction in thickness. The volume fraction of $\omega$, when detectable, was estimated using the method of Hickman[2] and the particle size was calculated from the Scherrer equation, assuming no strain broadening of the $\omega$ reflections.[2*]

*Although extremely small amounts of $\omega$ are detectable using this technique, *i.e.*, less than one volume percent, the experimental accuracy (± 1 vol-pct) ultimately limits the resolution of this X-ray technique.

The rotation of the [110] pole away from the sample normal toward the rolling direction was measured for all alloys using a single crystal goniometer on the X-ray diffractometer. This method can resolve a rotation angle of 0.1 deg after 50 pct reduction in thickness. The (110) and (200) pole figures were determined on alloys containing 28, 32, 36, and 40 wt pct V, after 50 and 80 pct reduction by the Schulz technique utilizing a Siemens texture goniometer. At least three mils were removed from the surface of the as-rolled crystals by electropolishing prior to the texture measurements.

The domain size and microstrains developed during rolling of the single crystals were measured after 50 pct reduction in thickness by the X-ray line broadening method developed by Warren and Averbach and described by Warren.[3] The peak profiles of the (110) and (220) reflections were determined by automated step scanning in 0.02 deg ($2\theta$) increments using $CuK_\alpha$ radiation. Unfortunately, the long tails on the high angle side of the (400) reflection prevented its examination by $CuK_\alpha$ radiation. The intensity profiles were transformed into a Fourier series using computer techniques and corrected for the $\alpha_1\alpha_2$ doublet by the Rachinger[4] method. The diffraction profiles of the as-quenched samples were used for the instrumental correction employing the method of Stokes.[5] Finally, the corrected Fourier coefficients were used to calculate the domain size and rms strains in the [110] direction.

Normally, the faulting probability is determined from the asymmetry of the X-ray peak profiles. The asymmetry of the reflections from single crystals is very sensitive to slight changes in crystal orientation and the change in orientation with deformation of the (110) crystals prevented this analysis from being made. Therefore, powders were prepared for this purpose. Quantitative values of the twin fault probability, $\beta$, were calculated using the method of Cohen and Wagner.[6] They have shown that the value of $\beta$ can be calculated for bcc structures from

$$2\theta_{PM} - 2\theta_{PCG} = (14.6 X_{hkl} \tan \theta_{PM})\beta$$

where $2\theta_{PM}$ is the $2\theta$ value of the peak minimum, $2\theta_{PCG}$ is the center of gravity and $X_{hkl}$ is dependent on the particular reflection, having a value of 0.17 for (110) and −0.17 for the (220).[7] Since the (110) reflections distort toward higher angles, *i.e.*, $2\theta_{PM} > 2\theta_{PCG}$, and the (220) towards lower angles, utilization of both reflections minimize errors due to thermal scattering, instrumental effects, and so forth. It should be noted that for the present study, differences in $\beta$ values as a function of alloy composition are more important than their absolute values. For the calculation of the $\beta$ values from the powder samples, tungsten powder was used for the Stokes correction. The computational method also corrected for variations in the atomic scattering factors as a function of diffracting angle.

## RESULTS AND DISCUSSION

Of the alloys examined only the Ti-20 V alloy contained more than 1 pct $\omega$ with the amount decreasing with increasing vanadium. The as-quenched Ti-20 V alloy contained 20 vol. pct $\omega$ with a particle size of ~23Å. The presence of this large amount of $\omega$ prevented further analysis of the deformed samples of this alloy. No increase in $\omega$ was observed in any of the other alloys with deformation up to 50 pct reduction in thickness. However, after 80 pct reduction there was a substantial increase in the intensity of the $(0001)_\omega$ and $(0002)_\omega$ reflections for the 24 pct V alloy and to a much lesser degree for the 28 pct V alloy, thus indicating a stress induced transformation for these compositions.

The values of hardness, domain size ($D_{110}$), rms strain ($\langle \epsilon^2_{110} \rangle^{1/2}$), and twin fault probability, $\beta$, are listed in Table I. The differences in domain size, and

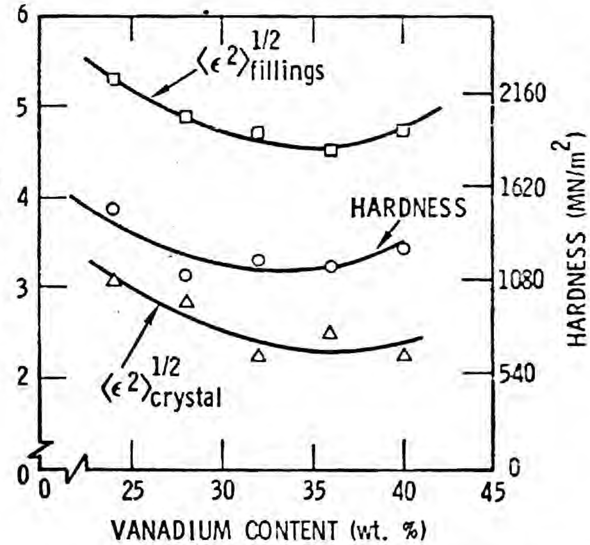



Fig. 1—RMS strain, $\langle \epsilon^2 \rangle^{1/2}$, and hardness as a function of vanadium content.

**Table I. Hardness, Domain Size, RMS Strain, and Twin Fault Probability**

| Pct Vanadium | As-Quenched Hardness, MN/m² | Single Crystals C.W. 50 pct Reduction $D_{110}$, Å | $\langle \epsilon^2_{110} \rangle^{1/2}$ | Fillings $D_{110}$, Å | $\langle \epsilon^2_{110} \rangle^{1/2}$ | $\beta_{110}$ | $\beta_{220}$ |
|---|---|---|---|---|---|---|---|
| 24 | 1536 | 180 | 0.00305 | 150 | 0.00539 | 0.00109* | 0.00019* |
| 28 | 1149 | 173 | 0.00281 | 155 | 0.00490 | 0.00026 | 0.00069 |
| 32 | 1197 | 160 | 0.00224 | 165 | 0.00474 | 0.00016 | 0.00046 |
| 36 | 1170 | 170 | 0.00252 | 167 | 0.00458 | 0.00014 | 0.00014 |
| 40 | 1271 | 185 | 0.00220 | 178 | 0.00474 | 0.00059 | 0.00109 |

*Values are invalid due to the presence of stress-induced $\omega$.

rms strain between the cold-rolled-single crystals and filings are due to the more extensive deformation of the filing process rather than to any basic difference in the deformation mechanisms involved. The influence of increasing vanadium content on the hardness and rms strain is shown in Fig. 1. A similar change in the twin fault probability, as a function of vanadium content, has also been observed, Fig. 2.

Figs. 3(*a*) and 3(*b*) are pole density stereograms of a Ti-32 V crystal (initial orientation was (001)[1$\bar{1}$0]), after 50 and 80 pct reduction in thickness, respectively. There is essentially no change from the original orientation, *i.e.*, (001)[1$\bar{1}$0] is a stable end orientation, in agreement with previous work on other bcc crystals.[8] These pole figures are typical of all of the Ti-V crystals examined whose initial orientation was (001)[1$\bar{1}$0]. Fig. 4 shows the preferred orientation developed in a Ti-28 V crystal whose initial orientation was (1$\bar{1}$0)[001]. Both $\chi$ and $\phi$ rotations occur simultaneously during the deformation.* As the [110] rotates toward the rolling direction, the [111] pole rotates towards the surface normal. The maximum rotation angles are 35 deg 15 min and 30 deg, after which the (111) plane is parallel to the surface. Three strong and three weak maxima are observed after 50 pct cold reduction which are twin related considering the [111] pole as the inversion center. The weak reflections are a result of deformation twinning. The rotation of $\phi$ results in the [1$\bar{2}$3] direction being parallel to the rolling direction instead of the [11$\bar{2}$] direction which is usually observed.[9] After 80 pct cold reduction, all six maxima have approximately equal intensity.

*$\chi$ is the rotation of the [110 pole from the surface normal toward the rolling direction and $\phi$, the rotation around the surface normal in the surface plane.

Fig. 5 shows the preferred orientation developed in the Ti-36 V alloy and should be compared with Fig. 4. The former contain very, very weak twin maxima and a very small rotation angle, $\phi$. In addition, an increase in deformation from 50 to 80 pct did not result in an increase in the twin maxima as observed for the 28 V alloy. The end orientation is (111)[11$\bar{2}$]. Finally, Fig. 6 shows the (110) pole figures for the Ti-40 V alloy after 50 and 80 pct deformation. No twin related maxima are observed after 50 pct reduction, but are seen in the 80 pct deformed crystal. For this alloy, large amounts of deformation increase the twin proba-

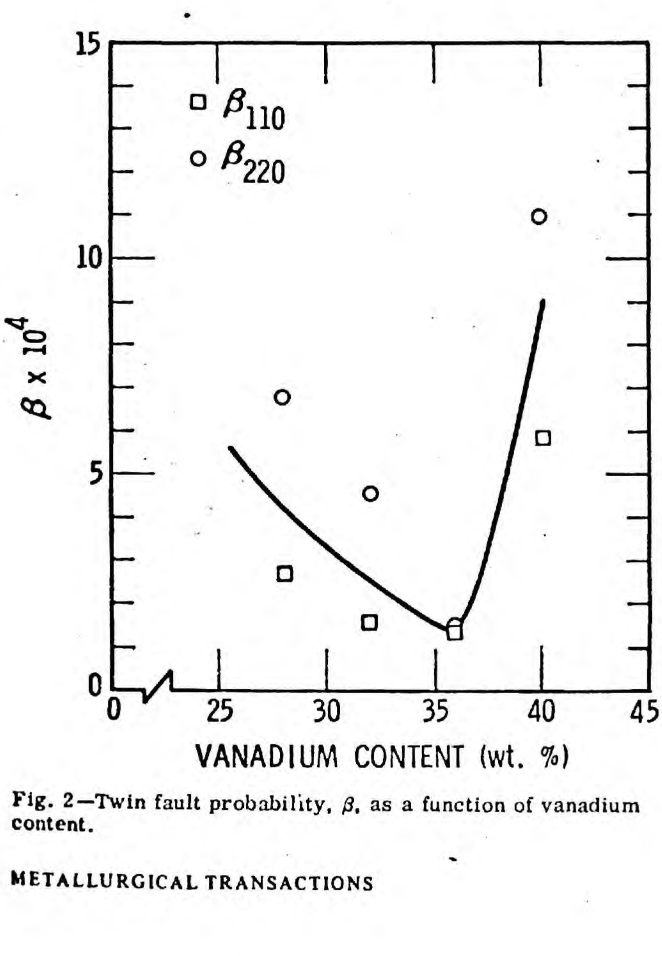



Fig. 2—Twin fault probability, $\beta$, as a function of vanadium content.

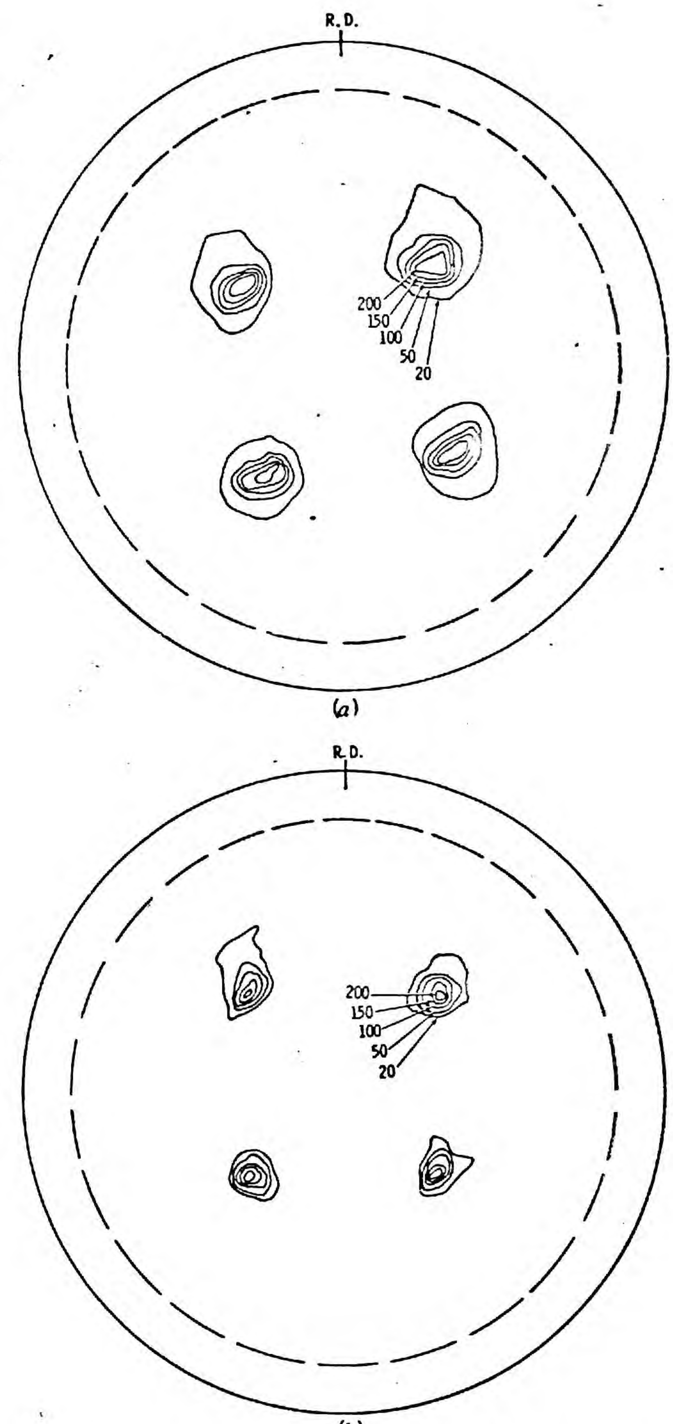



Fig. 3—Rolling texture of (001)[1$\bar{1}$0] Ti-32 V single crystal after (*a*) 50 pct reduction, (*b*) 80 pct reduction in thickness.

bility as illustrated by these texture measurements and confirmed by the line broadening studies of the filings. A summary of the rolling textures observed in this investigation is presented in Table II.

The present results indicate the importance of solute content, microstructure and twin fault probability in determining the nature of the deformation process in metastable $\beta$ titanium alloys. Koul and Breedis[10] have shown that the principal deformation mode in as-quenched Ti-22 V is twinning. The high hardness and rms strain in the Ti-24 V alloy used in this investigation should also be associated with this deformation mode. Thus the small amount of $\omega$ present in the as-quenched condition must provide an easy means of twin formation. However further coarsening of the $\omega$ phase during aging does eventually suppress twin deformation.[10] The exact nature of this interaction is not clear at this time and is the subject of continuing research at this laboratory.

Unfortunately, the presence of stress-induced $\omega$ did preclude the determination of the twin fault probability at least in the Ti-24 V alloy. However, there is a distinct minimum in the twin fault probability, Fig. 2, at higher solute contents. Indeed, this minimum appears to coincide with the disappearance of as-quenched $\omega$. Table I also shows that increased hardness and rms

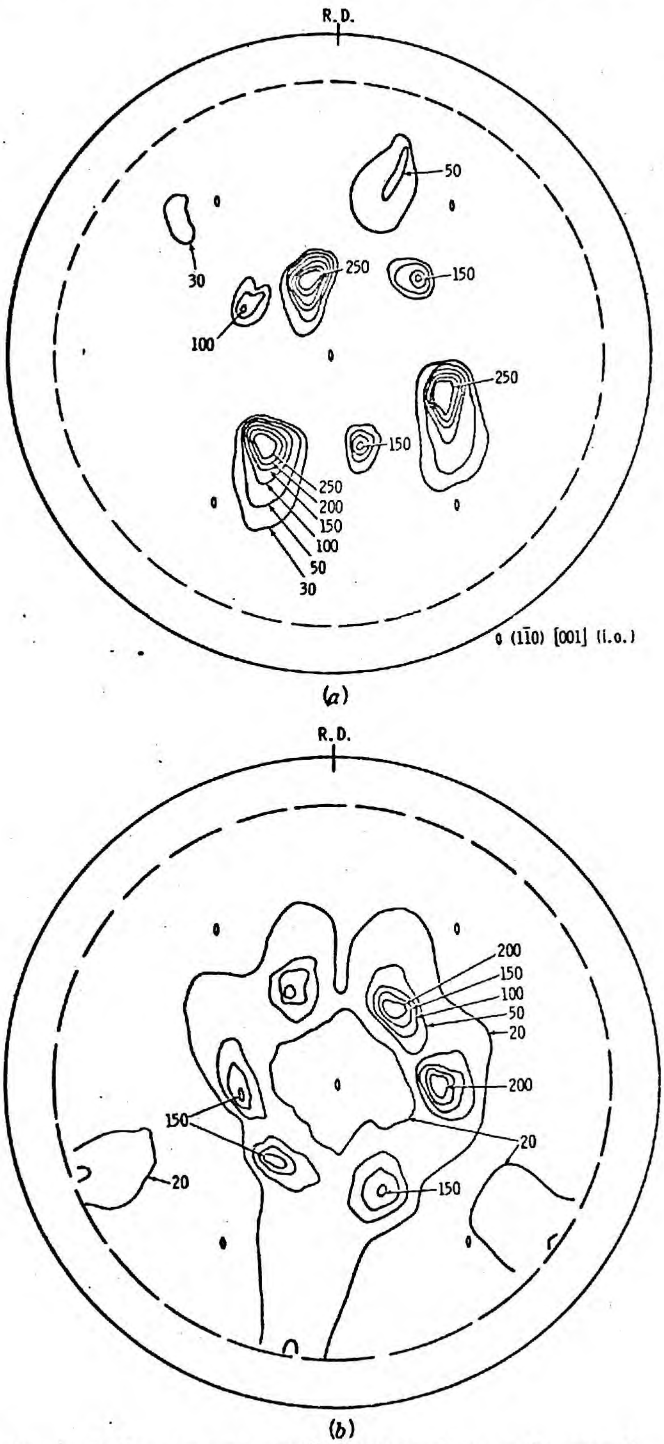



Fig. 4—Rolling texture of (1$\bar{1}$0)[001] Ti-28 V single crystal after (*a*) 50 pct reduction, (*b*) 80 pct reduction in thickness.

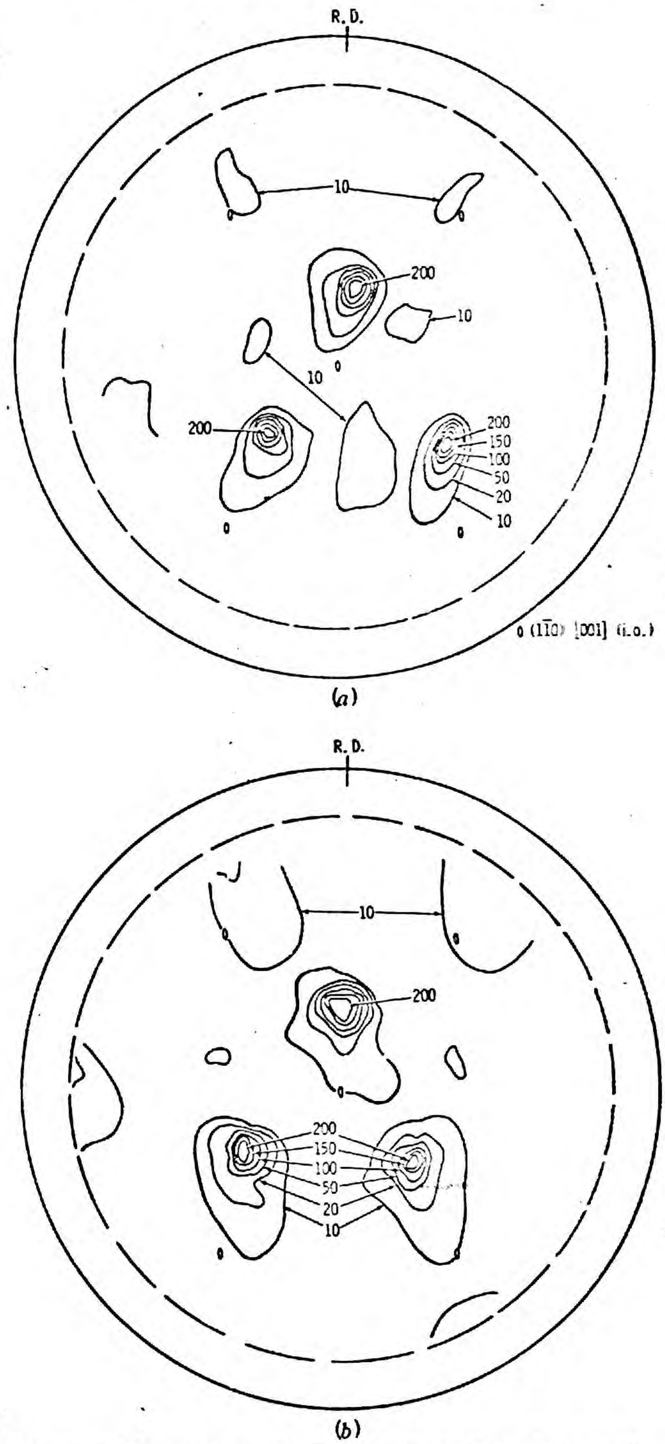



Fig. 5—Rolling texture of (1$\bar{1}$0)[001] Ti-36 V single crystal after (*a*) 50 pct reduction, (*b*) 80 pct reduction in thickness.

**Table II. Rolling Textures of Ti-V Single Crystals**

| Pct Vanadium | Original Orientation | 50 pct Rolling Texture | | | 80 pct Rolling Texture | | |
|---|---|---|---|---|---|---|---|
| | | New Orientation | χ deg | φ deg | New Orientation | χ deg | φ deg |
| 28 | (1$\bar{1}$0)[001] | (111)[$\bar{1}$23] + (111)[12$\bar{3}$] twins + v.w. (110)[1$\bar{1}$6] | 29.7 (24.3)* | 15 | (111)[10$\bar{1}$] + (111)[$\bar{1}$01] twins | 35 | 30 |
| 32 | (001)[1$\bar{1}$0] | (001)[1$\bar{1}$0] | 0 | 0 | (001)[1$\bar{1}$0] | 0 | 0 |
| | (1$\bar{1}$0)[001] | (111)[11$\bar{2}$] + v.w. (1$\bar{1}$0)[001] no twins | 29.9 (27.5) | 7 | not measured | | |
| 36 | (1$\bar{1}$0)[001] | (111)[11$\bar{2}$] + v.v.w. (111)[$\bar{1}\bar{1}$2] twins + + v.v.w. (110)[1$\bar{1}$7] | 31.2 (32.3) | 4 | (111)[11$\bar{2}$] + v.v.w. (111)[$\bar{1}\bar{1}$2] twins. note: (111)[$\bar{1}\bar{1}$2] twins are weaker than that of 50 pct roll. | 35 | 5 |
| 40 | (1$\bar{1}$0)[001] | (111)[21$\bar{3}$] no twins | 32.2 (31.7) | 10 | (111)[21$\bar{3}$] + v.w. (111)[2$\bar{1}$3] twins + v.w. (110)[1$\bar{1}$7] | 35 | 10 |

*Value measured before polishing the rolled surface.
v.w.—very weak.
v.v.w.—very, very weak.

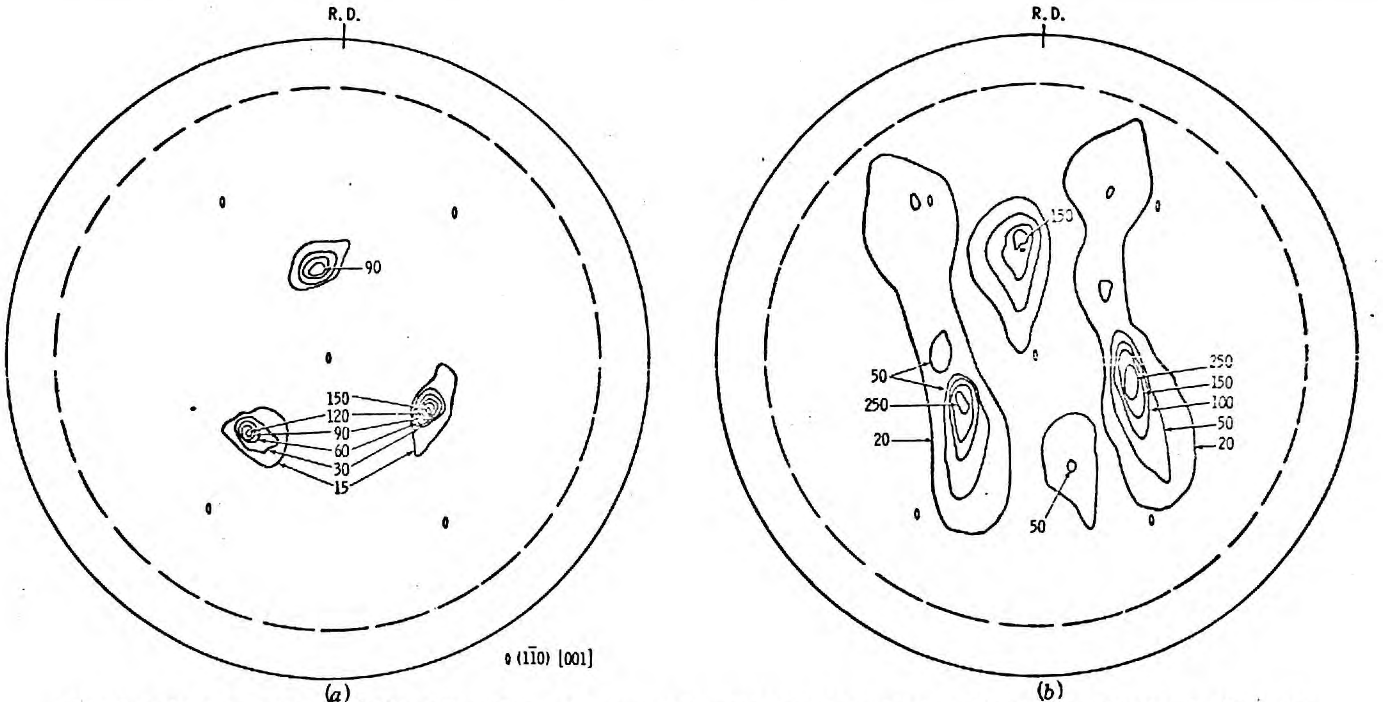



Fig. 6—Rolling texture of (1$\bar{1}$0)[001]Ti-40V single crystal after (*a*) 50 pct reduction, (*b*) 80 pct reduction in thickness.

strain cannot be explained by a difference in cell size since the latter does not vary, at least in the [110] direction, in the same manner. Finally, a twin textural component was observed in Ti-28 V but not in Ti-32 V.

Normally, the twin fault probability in bcc alloys increases with increasing solute content.[8] This general behavior is observed in Ti-V alloys when the complicating influence of the as-quenched ω phase is absent, compare Ti-36 V and Ti-40 V in Table I. The textural results also show that as the solute content increases from Ti-32 V to Ti-40 V twin component maxima are observed.

## CONCLUSIONS

The ω phase formed on quenching Ti-V alloys with vanadium contents up to but not including 36 wt pct V. Stress-induced ω was formed in alloys containing up to 28 wt pct V. The principal features controlling the nature of deformation and the preferred orientation produced in β Ti-V alloys are: the amount of ω formed during quenching, the solute content, and the twin fault probability.

## ACKNOWLEDGMENTS

We would like to acknowledge financial support by the United States Atomic Energy Commission and the Office of Naval Research under Contract N00014-71-C-0412 and helpful discussions with Dr. D. Kalish of the Bell Telephone Laboratories.

## REFERENCES


1. H. J. Rack, D. Kalish, and K. D. Fike: *Mater. Sci. Eng.*, 1970, vol. 6, p. 181.
2. B. S. Hickman: *J. Inst. Metals*, 1968, vol. 96, p. 330.
3. B. E. Warren: *X-ray Diffraction*, Addison-Wesley, 1969.
4. W. A. Rachinger: *J. Sci. Instr.*, 1948, vol. 25, p. 254.
5. A. R. Stokes: *Proc. Phys. Soc.*, 1948, vol. 61, p. 382.
6. J. B. Cohen and C. N. J. Wagner: *J. Appl. Phys.*, 1962, vol. 33, p. 2073.
7. C. N. J. Wagner: *Local Atomic Arrangements Studied by X-ray Diffraction*, J. B. Cohen and J. E. Hilliard, eds., Gordon and Breach, 1967.
8. H. Hu, R. S. Cline, and S. R. Goodman: *Recrystallization, Grain Growth and Textures*, chap. 8, ASM, Metals Park, Ohio, 1965.
9. C. G. Dunn: *Acta Met.*, 1954, vol. 2, p. 173.
10. M. K. Koul and J. F. Breedis: AFML-TR-69-236, June 1969.

OFFICE OF NAVAL RESEARCH

Contract N00014-67-A-0159-0015, NR 031-750

TECHNICAL REPORT 74-1

# THERMAL ETCHING OF BETA Ti-V ALLOYS

by

Fu Wen Ling and E. A. Starke, Jr.
Metallurgy Program, School of Chemical Engineering
Georgia Institute of Technology
Atlanta, Georgia 30332

Reprinted from Metallography 5 (1972)

January 8, 1974

Reproduction in whole or in part is permitted for any purpose of the United States Government.

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

# REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

| | |
|---|---|
| 1. REPORT NUMBER<br>74-1 | 2. GOVT ACCESSION NO. |
| 3. RECIPIENT'S CATALOG NUMBER | |
| 4. TITLE (and Subtitle)<br>THERMAL ETCHING OF BETA Ti-V ALLOYS | 5. TYPE OF REPORT & PERIOD COVERED<br>Technical Report |
| | 6. PERFORMING ORG. REPORT NUMBER |
| 7. AUTHOR(s)<br>Fu-Wen Ling and E. A. Starke, Jr. | 8. CONTRACT OR GRANT NUMBER(s)<br>N00014-67A-0159-0015<br>NR 031-750 |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS<br>Metallurgy Program, School of Chemical Engineering<br>Georgia Institute of Technology<br>Atlanta, Georgia 30328 | 10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS |
| 11. CONTROLLING OFFICE NAME AND ADDRESS<br>Metallurgy Program<br>Office of Naval Research, 800 North Quincy Street<br>Arlington, Virginia 22217 | 12. REPORT DATE<br>January 1974 |
| | 13. NUMBER OF PAGES |
| 14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) | 15. SECURITY CLASS. (of this report)<br>Unclassified |
| | 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE |

16. DISTRIBUTION STATEMENT (of this Report)

Unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

Reprint of article from Metallography, Vol. 5 (1972)

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

titanium alloys
microstructure
thermal etching

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This paper describes the thermal etching of Beta Ti-V alloys by annealing in vacuum at 900°C. The thermal-etch pits and facets are correlated with dislocations. Using crystals of known orientation it has been determined that the facets consist of either {100}, {110}, or {112} planes and that the morphology of the pits depends on the surface orientation.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE
S/N 0102-014-6601

Unclassified
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

# Thermal Etching of β Ti-V Alloys

FU-WEN LING AND E. A. STARKE, JR.
*Metallurgy Program, School of Chemical Engineering,*
*Georgia Institute of Technology, Atlanta, Georgia* 30332

This paper describes the thermal etching of $\beta$-Ti-V alloys by annealing in vacuum at 900°C. The thermal-etch pits and facets are correlated with dislocations. Using crystals of known orientation it has been determined that the facets consist of either {100}, {110}, or {112} planes and that the morphology of the pits depends on the surface orientation.

## Introduction

The etch-pit technique has been widely used for studying dislocation arrangements and plasticity in both nonmetallic and metallic crystals since Gevers et al. [1] first proved that dislocations could be revealed by etching methods. Various etching techniques have been used which include chemical etching [2], solution etching [3], preferential oxidation [4], electrolyte etching [5], and thermal etching [6]. Thermal etching techniques have been developed for revealing dislocations in silver [7], however, the evidence for their association with dislocation sites in most metals is not as conclusive as it is with chemical- and electrolytic-etch pits.

The formation of etch pits by dissolution at dislocations can occur either because of a favorable chemical potential due to the presence of impurities along dislocations or because of the extra energy concentrated along the dislocation in the form of core and strain energy. In either case a successful etching technique to delineate dislocations must be able to differentiate between small potential and/or energy differences. The observations by Bennett [8] of thermal etching of pure titanium when annealed in vacuum above the $\alpha$-$\beta$ transus suggests that dislocations in titanium might be revealed by thermal etching if the $\alpha$-$\beta$ transformation could be suppressed on cooling. The martensite transformation which occurs in pure titanium rumples the surface and completely obliterates all but grain boundary etching [9].

This paper describes a study of thermal etching on beta Ti-V alloys. Beta titanium and vanadium form a complete series of solid solutions and a single-phase body-centered cubic structure may be obtained in alloys containing greater than 20 wt. % vanadium by quenching from above 700°C. Consequently,

Copyright © 1972 by American Elsevier Publishing Company, Inc.

a study of thermal etching can be made on these titanium alloys without the complications of the allotropic transformation present in pure titanium.

## Experimental Procedure

Two Ti-V alloys, containing 20 and 28 wt. % vanadium, were prepared by arc-melting the high purity components under an argon atmosphere. The as-cast 50-g buttons were sealed in quartz under 0.5-atm partial pressure of argon and homogenized for 60 days at 1100°C. This extended heat treatment produced very large grains from which crystals of special orientations could be prepared. Samples were abrasive- and spark-cut from the large grained buttons and mechanically polished through 0.1-$\mu$ alumina followed by electropolishing using the procedure described by Rack et al. [10]. The polished samples were encapsulated in vycor under a vacuum of $4 \times 10^{-6}$ Torr and annealed for two hours at 900°C. Some samples were quenched by breaking the capsules in ice water and some were allowed to cool inside the unbroken capsule immersed in ice water.

After removing the annealed samples from the furnace it was evident that extensive evaporation of the alloy had occurred during heat treatment since the inside of the vycor tube was coated with a metallic film. Electron microprobe studies confirmed that the film was primarily titanium with little or no vanadium present.

The orientations of the various grains, which were subsequently examined by optical microscopy, were determined from x-ray diffraction Laue patterns. A slight tarnishing occurred on samples which came in contact with the water during quenching. The color of the tarnished grains could be used to distinguish between {100}, {110}, and {112} planes and was later used in conjunction with stereographic analysis for the determination of the orientation of the crystallographic facets of the thermal etch pits. Figure 1 is a stereographic projection showing the directions of the normals to the crystal surfaces examined.

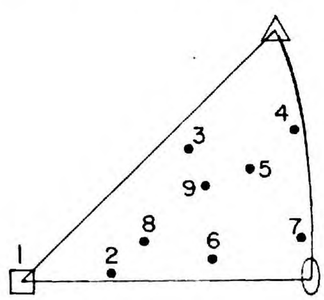



FIG. 1. Stereographic projection showing the directions of the normals to the unetched crystal surfaces.

## Results and Discussion

All the surfaces examined showed the effects of thermal etching. This did not result in general evaporation from the surface, but produced a number of local attacks which showed interesting features. Figure 2(*a*) is an optical micrograph of surface 4 and shows triangular pits which are bounded by {100} planes. The relationship of the pit topology with the surface normal is illustrated in Fig. 2(*b*). The orientation of the planes, which comprise the pits, was determined

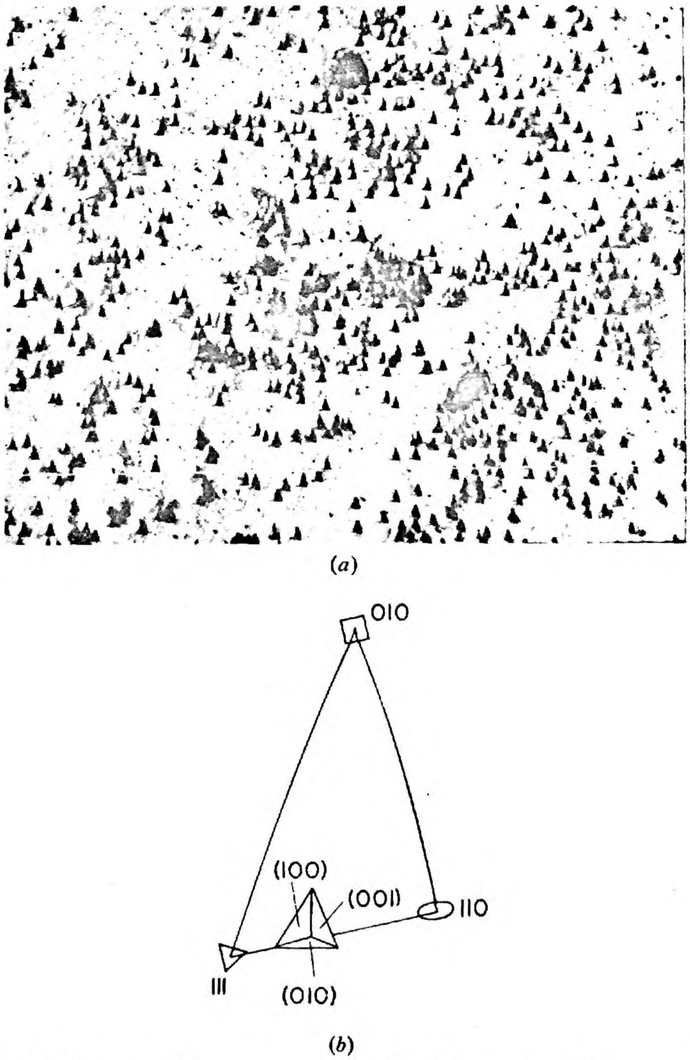



FIG. 2. (*a*) Thermal etch pits on surface number 4 after annealing in vacuum for two hours at 900°C. Mag. 600×. (*b*) Schematic showing the relationship of the pit topology and the orientation of the etched surface. The triple point of the small triangle corresponds to the surface normal.

by stereographic analysis from the angle of tilt from the surface (40°–60°) and confirmed by color metallography. Triangular pits on this surface could also be bounded by {110} planes; however, none of that orientation were observed. They were observed, however, on surface 3, as shown in Fig. 3(*a*). The relationship of pit topology with the surface normal is illustrated in Fig. 3(*b*). Figure 3(*a*) confirms that the pits are associated with dislocations since their density is high along scratches and greatly increases as the abrasivecut edge of this specimen is

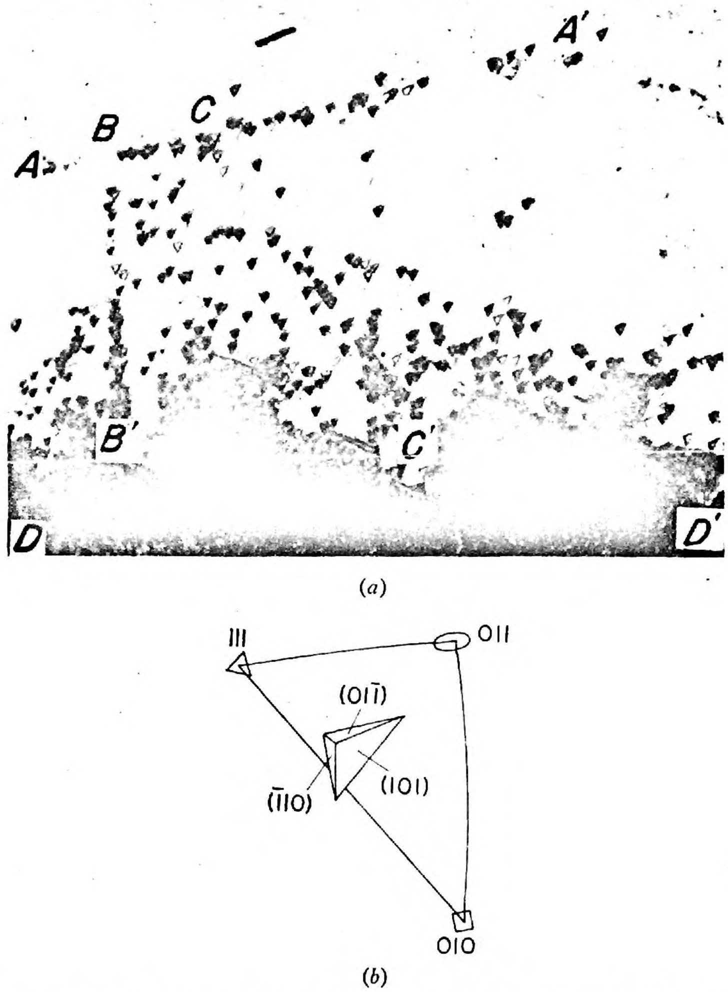
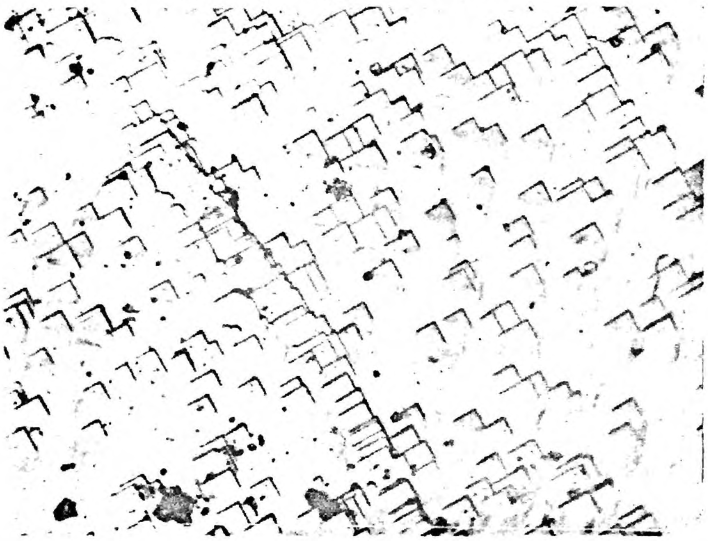


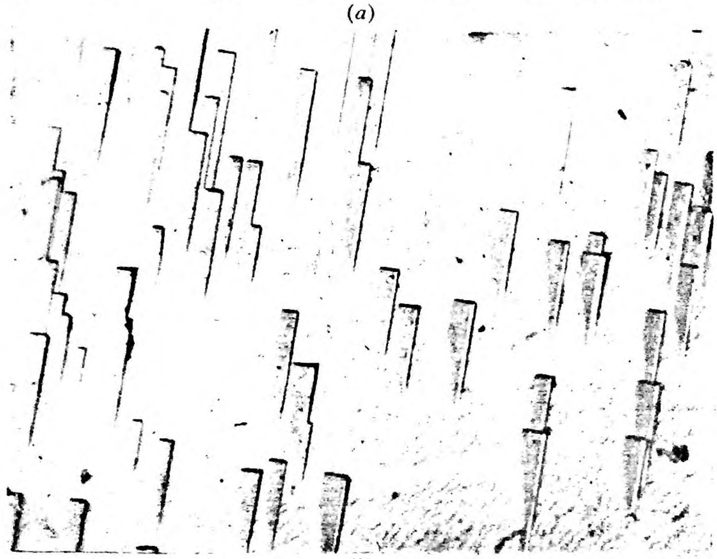

FIG. 3. (*a*) Thermal etch pits on surface number 3. AA′, BB′, and CC′ are scratches. DD′ is the abrasivecut edge. Mag. 600×. (*b*) Schematic showing the relationship of the pit topology, whose surfaces are bounded by {110} planes, and the surface normal, represented by the triple point of the small triangle.

approached. Although it is difficult to prove that all pits correspond to dislocations, the present results certainly indicate that the emergence points of dislocations can cause selective thermal etching. This is expected since the strain energy is reduced by evaporation of the deformed material in the immediate vicinity of the dislocation.

The pits (or facets) which developed on the surface oriented near {100} planes are shown in Figs. 4(*a*) and (*b*). The original surface normal was 2°–4°



(*a*)



(*b*)

FIG. 4. (*a*) Thermal facets on surface number 1 whose normal is 2°–4° from the ⟨100⟩. Mag. 600×. (*b*) Thermal facets on the same crystal but a different region as that shown in (*a*). Mag. 600×.

from the ⟨100⟩. The facets are bounded by three {100} planes. Since thermal faceting may be caused by a rearrangement of the surface atoms to produce a surface of minimum energy [6], it is not certain that these facets correspond to dislocation-surface interactions. However, Fig. 5 shows scratches across the boundary of surfaces 1 and 4. Triangular etch pits are associated with the scratches in grain 4 and facets similar to those of Fig. 4 are associated with the scratches in grain 1. In addition, a high density of the facets are present along the grain boundary. Consequently, their origin may be associated with the immerging point from the surface of a dislocation. However, further growth of the facets may also be associated with surface energy effects.

Figure 6 illustrates the relationship between grain orientation and etch pit topography. The darker grain, number 8, has triangular etch pits different from those of the lighter grain, number 9. Prior to the final anneal, the grain boundary was along $AA'$, which is extensively pitted. During annealing secondary recrystallization occurred and the boundary migrated to $BB'$. Careful examination shows that as one goes from $AA'$ to $BB'$ the topography of the pits gradually changes from that of grain 8 to that of grain 9. The curvature, between pits, of various segments of the boundary $BB'$ suggests that the dislocations are inhibiting grain boundary migration.

Surface 7 contained four different types of etch pits and/or facets. Figure 7(*a*) is an optical micrograph showing these features and Fig. 7(*b*) is a schematic illustration of the relationship between pit morphology and the crystallographic planes which bound their surfaces. The large angle (55°) pits might have developed from ones similar to the small needle-shaped pits by the replacing of one of the {112} facets by two {110} facets. It is not clear whether the different kinds of pits are associated with: (a) different types of dislocations; (b) dislocations lying on different glide planes; or (c) surface energy effects.

## Summary and Conclusions

The results of this study show that thermal etching can be used to study the dislocation structure of $\beta$ Ti-V alloys containing low dislocation densities. The etching mechanism appears to be involved with preferential evaporation of titanium atoms at the immergence points of dislocations from surfaces, although the surface energy may play an important role as the pits and facets are developed. In addition, the role of absorbed gases is unclear since the vacuum obtained, $4 \times 10^{-6}$ Torr, doesn't exclude the possibility of impurity adsorption.

*We would like to acknowledge financial support by the Office of Naval Research under Contract N00014-71-C-0412 and helpful discussions with Dr. Helen Grenga.*

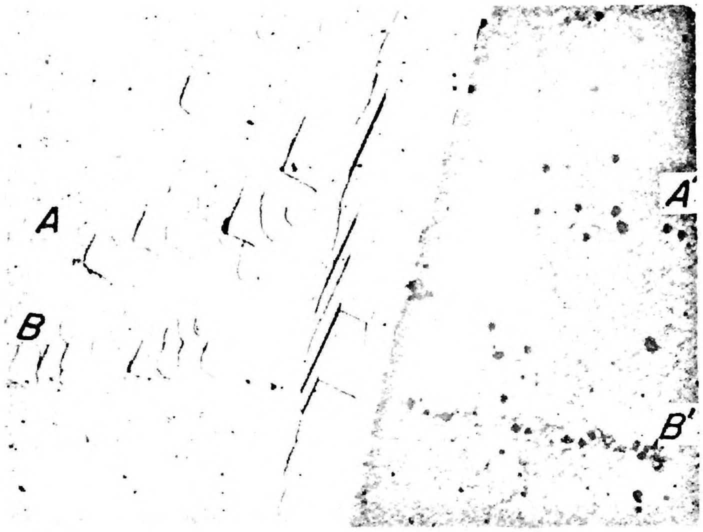



FIG. 5. Thermal pits and facets along scratches AA′ and BB′ across surfaces 1 (light area) and 4 (dark area). Magnification: 300×.

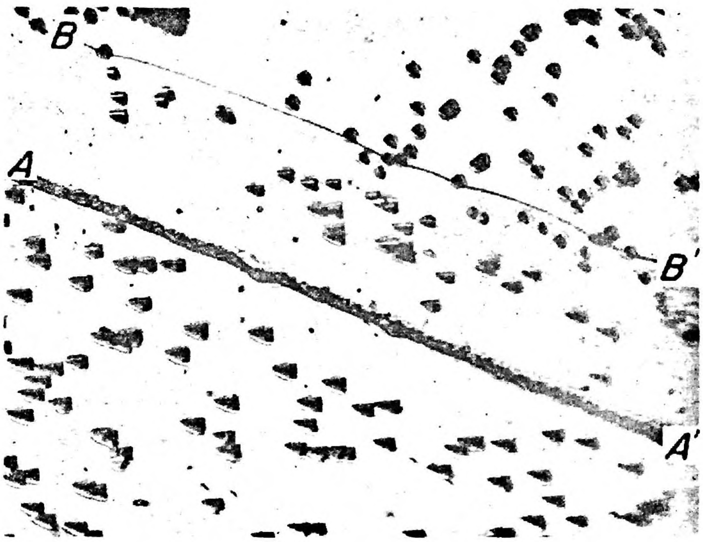
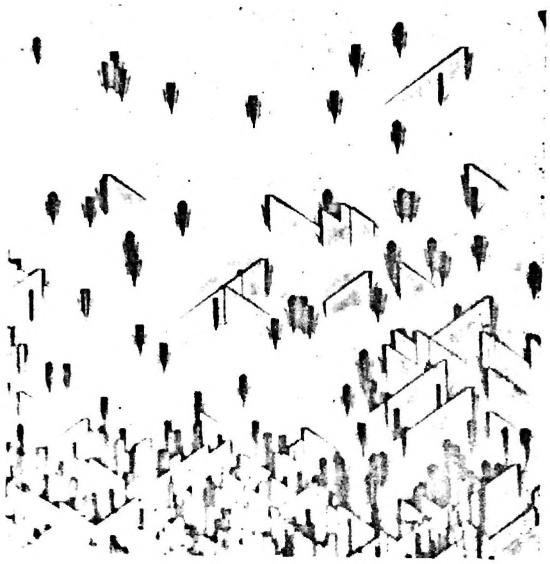
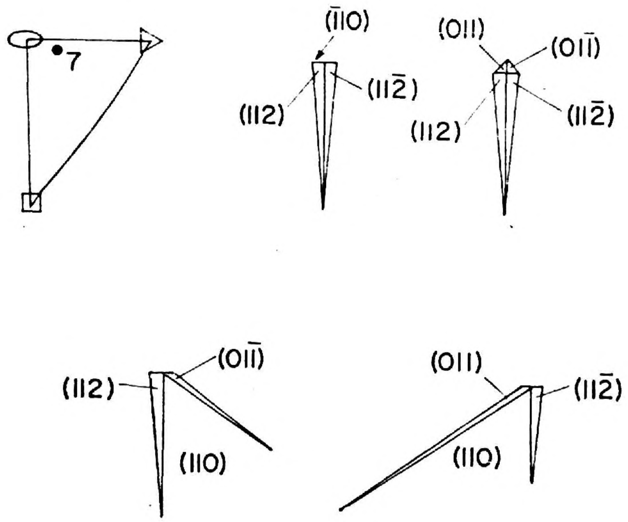



FIG. 6. Relationship between pit topology and grain orientation. AA′ represents the original grain boundary and BB′ the boundary after the secondary recrystallization. Mag. 600×.



(*a*)




(*b*)

FIG. 7. (*a*) Four types of thermal pits and/or facets on surface 7. Mag. 600 ×. (*b*) Schematic representing the crystallographic relationships with the pit morphology.

## References


1. R. Gevers, S. Amelincks, and W. Dekeyser, *Naturwissenschaften* **39,** 448 (1952).
2. W. G. Johnson, Dislocations etchpits in nonmetallic crystals, *Progress in Ceramic Science* (J. E. Burke, Ed.), Vol. 2, Pergamon Press, New York (1962).
3. S. Amelinckx, *Acta Met.* **2,** 848 (1954).
4. F. W. Young and A. T. Gwathmey, *J. Appl. Phys.* **31,** 225 (1960).
5. P. A. Jacquet, *Acta Met.* **2,** 725 (1954); 770 (1954).
6. A. J. W. Moore, Thermal faceting, in *Metal Surfaces* (Structure, Energetics and Kinetics Seminar of the ASM and the Metallurgical Society of AIME), American Society for Metals, Metals Park, Ohio (1963).
7. A. A. Hendrickson and S. E. Machlin, *Acta Met.* **3,** 64 (1955).
8. W. D. Bennett, *J. Met.* **7,** 322 (1955).
9. P. Evans, *Acta Met.* **5,** 342 (1957).
10. H. J. Rack, D. Kalish, and K. D. Fike, *Mater. Sci. Engr.* **6,** 181 (1970).

E-19-615

GEORGIA INSTITUTE OF TECHNOLOGY
ATLANTA, GEORGIA 30332

SCHOOL OF
CHEMICAL ENGINEERING

June 1[illegible] [illegible]973

Dr. Alan H. Rosenstein
Electronic and Solid State Sciences
Air Force Office of Scientific Research
1400 Wilson Boulevard
Arlington, Virginia 22209

Dear Dr. Rosenstein:

Transmitted herewith are sixteen (16) copies of our Int[illegible] [illegible]eport, "Effects of Interface Phenomena on the Mechanical Behavior o[illegible] [illegible]ls", for Grant No. AFOSR-71-2064, and sixteen (16) reprints of "I[illegible] [illegible]tion of Dislocations with Interfaces", by B. R. Livesay and E. A. [illegible]e, Jr. which appeared in Acta Metallurgica.

Very truly yours,

Edgar A. Starke, Jr.
Professor
Metallurgy

EAS:np
Encl.

# EFFECTS OF INTERFACE PHENOMENA ON THE MECHANICAL BEHAVIOR OF METALS

by

Edgar A. Starke, Jr.
and
Billy R. Livesay

June, 1973

This research was supported by the Air Force Office of Scientific Research (AFSC) under Grant No. AFOSR-71-2064.

Approved for public release; distribution unlimited.

Qualified requestors may obtain additional copies from the Defense Documentation Center; all others should apply to the Clearinghouse for Federal Scientific and Technical Information.

## SUMMARY OF RESEARCH

The mechanical behavior of crystalline materials is primarily explainable in terms of the dynamics of dislocations. A number of factors are known to influence dislocation dynamics in the neighborhood of an interface. The goals of this research program are to determine the relative importance of various interface parameters such as differential modulus, lattice misfit, crystallographic orientation, structure, etc. on the mechanical behavior of materials which contain interfaces, either on surfaces or internally.

Differential Modulus Effects

Our initial studies were concerned with the effect on mechanical behavior of a differential elastic modulus across an interface using single crystal composite specimens prepared in configurations which minimize the influence of other factors. Tensile specimens were prepared from thin copper single crystals and epitaxial metal films were grown on the surfaces by vapor deposition to provide desired interfaces. The principal material combinations studied were copper-nickel and copper-cobalt. In addition, copper films grown onto copper tensile specimens substrates were used to evaluate the influence of certain imperfections. The elastic modulus difference was found to provide the primary strengthening mechanism for Cu-Ni and Cu-Co interfaces. Surface and interfacial energy terms were also evaluated and found not significant compared to contributions from other interface factors. Crystal structure changes induced in cobalt coatings with deformation resulted in high work-hardening compared to that of the Cu-Ni systems. The major results of these studies have been published in Acta Metallurgica (1), and sixteen copies of this article are attached.

An interface model which more closely matches the modulus gradient expected for extended interfaces has been developed. The interface is partitioned into a sequence of discrete lamella to which specific elastic modulus values can be assigned. The number of lamella, their thickness and the individual modulus values are selected to approximate a desired interface configuration. The multiple image contributions to the force on a single screw dislocation parallel to the interface are then calculated by numerical summation procedures on the Georgia Tech Univac 1108 computer. A linearly decreasing, stepped variation of elastic modulus centered at the interface has been assumed. The systems for which these computations have been carried out correspond to Cu-Ni, Cu-Pt, Cu-Fe, Cu-Co and Cu-Re.

Composite specimens containing 30 to 50 layers have been prepared by the evaporation of alternate metal layers onto NaCl crystals. Individual layer thickness values were selected between 200 Å and 300 Å for all the specimens studied. Films of pure copper and of pure nickel were deposited so that each had a thickness corresponding to the accumulated Cu and Ni content of the composite structure. This allowed a direct comparison of the strength of films of the pure metals with that of the composite. The films were produced in the shape of our standard necked tensile specimens by depositing through photo-machined stainless steel masks onto the NaCl. The specimens were subsequently removed by floating them from the NaCl surface in distilled water. A strengthening effect of about 100% is found to occur for the composite specimens as compared to the combined strengths of the pure copper and pure nickel specimens.

The experiments mentioned above have been extended to include a new sample configuration and a different coating technique. The cylindrical sample design now being used permits sectioning along selected planes after tensile tests so that electron microscope examinations of the slip planes may be made.

This will allow direct observations of the dislocation-interface interactions. Interfaces produced by ion-plating onto single crystal substrates are being studied and compared with those produced previously by vapor deposition. Ion plated films are quite different from those produced by vapor deposition. The plating atoms strike the surface with significant energy to travel some distance into the material before coming to rest. This produces a film of unusually good adherence.

The penetration of the film atoms in vapor deposition is extremely small since they possess only thermal energy, thus the interface is quite sharp. In ion plating the penetration is significant with the result that there is a gradual interface which consists of a zone of substrate, sputtering and plating atoms. The physical status of this zone is unknown but may be any of a variety of forms from simply injected interstitial atoms to alloys of previously unobserved phases. The initial study in the ion plating effort is being directed to the characterization of this interface zone. The state and depth of the zone should be sensibly dependent on the sputtering and plating atoms, the accelerating voltage, and the temperature. Our first experiments will separate the parameters to determine their individual effects. We have done some preliminary sputtering experiments with argon gas and evaluated the strain using x-ray line broadening methods. The calculated strain was very small ($3 \times 10^{-4}$), which may be real, but is likely due to the very thin zone relative to the x-ray penetration.

Mechanical properties such as critical resolved shear stress, extent of easy glide, and work hardening coefficient are being measured on the metal-metal single crystal systems which contain interfaces of various materials produced by ion plating. In addition, low cycle fatigue behavior is being studied since fatigue life is very sensitive to the surface condition. The deformation modes in both the primary and secondary slip systems of the substrate are being characterized by transmission electron microscopy and the interaction of the dislocations with the various interfaces are being examined.

Twin Interfaces in Single Crystals of $Cu_3Au$

Four flat (110) $Cu_3Au$ single crystals were heat treated to various degrees of order; 0, 0.5, 0.8, and 1. These crystals were deformed at room temperature under plain-strain compression with the $[\bar{1}10]$ as the flow direction. A discontinuity, i.e., a sudden reduction, in work hardening rate was observed at certain points on the stress-strain curves. Concurrently, deformation bands were observed on the surface of the crystals. By comparison with optical micrographs of similar work on iron-cobalt alloys (2), it was concluded that these bands contained deformation twins. The stress levels associated with this deformation behavior increased rapidly with degree of order for high degrees of order; however, no such increase was noted when the degree of order was low. Assuming 1/6 $[11\bar{2}]$ (110) shear for mechanical twin formation for all degrees of order, changes in neighbor relationships during twinning are significant only for high degrees of order. The observed increase in twinning stress is attributed to this factor. The influence of stacking fault energy on the twinning stress was not as significant. However, a small decrease in twinning stress during the early stage of ordering is associated with a large decrease in stacking fault energy during this period.

The Effect of Interface Parameters on Friction and Wear Behavior

The surface of a bearing is subjected to complex combinations of mechanical stress modes. In the most simple cases, a volume of material near the surface alternately experiences stresses corresponding to tension, shear and compression. The usual situation is even more complicated with the introduction of other factors such as environmental effects and fatigue. The interface strengthening processes investigated in this program can be applied to the design of improved bearing materials using properly selected coating systems. A few simple experiments were carried out here to verify the validity of extending these concepts to friction and wear problems. A simple accelerated wear test was carried out on two copper crystals with regions coated with a 1500 Å Co-2.5% Fe film. A 25 micron diamond stylus was loaded normal to the flat specimen surface and the specimen was vibrated at 30 Hz with an amplitude of about 8mm. Wear tracks were made in both coated and uncoated regions of the crystals at loads ranging from 1-50 dyne and from 1800 to about 9000 cycles. SEM observations of the resulting wear tracks revealed that the cobalt alloy coating provided significantly greater wear resistance over that of the free surface.

Investigations of the Effect of Stable Crystal Structure of Coating on Mechanical Behavior

The Co-Fe alloys ranging from 0-6% Fe provide a useful system for investigations of the effect of the stable crystal structure of a coating on dislocation interactions at an interface since the stable structure of cobalt varies from hcp to double hcp to fcc in this alloy range. Preparation conditions identical to those employed for coatings of pure cobalt resulted in single crystal films for certain alloy compositions in this range. Mechanical tests conducted on copper crystals having 2000 Å coatings of alloys in this range have indicated the surprising result that a crystal specimen with a Co-2.5% Fe coating has a significantly lower yield stress than do crystals with either no coating or with the Co-6% Fe alloy coating. Analysis of the slip lines on low cycle fatigue specimens of copper crystals having these three surface configurations were consistent with the observed mechanical behavior.

Single slip systems were activated in crystals having either a free surface or coated with the Co-2.5% Fe alloy. The slip lines on the free surface were coarse whereas fine slip is found for a specimen coated with the Co-2.5% Fe alloy. A secondary slip system was activated with a Co-6% Fe coating indicating that the back stress of the 6% Fe alloy inhibited the operation of the primary slip system.

Effects of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation

Four alloys based on the composition developed for 7050 by Alcoa for the Air Force have been prepared. These alloys include the commercial 7050 alloy, an alloy of identical composition with the exclusion of Zr, an alloy of identical composition with the exclusion of Zr and half of the Cu and an alloy of identical composition with the exclusion of Zr and all of the copper. This sequence of alloys will allow the isolation of various interface parameters, which is necessary for a theoretical analysis of the interaction of dislocations with the interfaces in an age hardening alloy such as 7050.

Our initial studies have been concerned with the kinetics of the decomposition of the super-saturated solid solid solution and the characterization of the microstructure developed. In addition techniques for growing single crystal, electron microscopy, small-angle x-ray scattering, and mechanical testing (both monotonic and cyclic) have been established.

Improvements in Facilities

A significant improvement was made in the Micromechanics Laboratory by rebuilding one of our devices into a highly versatile mechanical testing system. It is currently being used for the low cycle fatigue studies of 1-2mm thick coated copper crystals. Specimen mounting fixtures provide for tests either in air or in corrosion test cells. A Schmidt trigger circuit and other electronic devices provide for completely automatic cycling and recording of data. Low-cycle fatigue facilities (a modified Instron Machine) have also been established for testing of bulk single and polycrystalline samples.

## SCIENTIFIC PAPERS AND REPORTS

E. A. Starke, Jr., G. Kralik and V. Gerald, "Plasticity of Al-Ge Single Crystals Containing Small Fractions of Precipitates", Mater. Sci. and Engr. 11, 319-323 (1973).

B. R. Livesay and E. A Starke, Jr., "Dislocation Interactions at Interfaces", Acta Met. 21, 247-254 (1973).

B. R. Livesay and S. Spooner, "Magnetic Anisotropy of Ordered Cobalt Films", AIP Conference Proceedings No. 10, 1583 (1973).

Fu-Wen Ling and E. A. Starke, Jr., "Thermal Etching of β Ti-V Alloys", Metallography 5, 399-407 (1972).

D. A. Mauney, E. A. Starke, Jr. and R. F. Hochman, "Hydrogen Embrittlement and Stress Corrosion Cracking of Ti-Al Binary Alloys", Corrosion (in press).

K. C. Chen, Fu-Wen Ling and E. A. Starke, Jr., "Structure and Mechanical Properties of Stress-Ordered $Ni_4Mo$", Mater. Sci and Engr. (in press).

Fu-Wen Ling, H. J. Rack, and E. A. Starke, Jr., "Deformation of Metastable Beta Ti-V Alloys", Met. Trans. (in press).

Fu-Wen Ling, E. A. Starke, Jr. and B. G LeFevre, "Deformation Behavior of Beta Ti-V Alloys", (submitted to Met. Trans.).

## PROFESSIONAL PERSONNEL

Dr. Edgar A. Starke, Jr., Professor of Metallurgy, School of Chemical Engineering;

Dr. Billy R. Livesay, Senior Research Scientist, Physical Sciences Division, Engineering Experiment Station.

## GRADUATE STUDENTS

Mr. S. Chakrabortty, Ph.D. Candidate, "Twin Interfaces in Single Crystals of $Cu_3Au$"; Thesis Advisor: Professor E. A. Starke, Jr.

Mr. E. Y. Chen, Ph.D. Candidate, "Ion Plated Interfaces on Copper Single Crystals", Thesis Advisor: Professor E. A. Starke, Jr.

Mr. T. H. B. Sanders, Ph.D. Candidate, "Effects of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation"; Thesis Advisor: Professor E. A. Starke, Jr.

Mr. A. Ansah, Ph.D. Candidate, "Effects of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation"; Thesis Advisor: Professor E. A. Starke, Jr.

## INTERACTIONS WITH OTHER GROUPS

Dr. E. A. Starke, Jr. attended the Spring Meeting of the Metallurgical Society in Boston in May, 1972. Attended Non-ferrous Metals Committee Meeting.

Dr. E. A. Starke, Jr. attended the Wire Association Meeting in Chicago in October, 1972 and received the Medal Award of the Nonferrous Division.

Dr. E. A. Starke,Jr. attended the Fall Meeting of the ASM-AIME in Cleveland in October, 1972 to meet with the Phys. Met. Committee to organize an Aluminum Symposium.

Dr. E. A. Starke, Jr. met with Drs. B. A. Wilcox of Battelle and H. L Gagel of AFML at AFML in December, 1972 to review abstracts and finalize program for Spring Meeting of AIME.

Dr. E. A. Starke, Jr. attended the symposium on Metallurgical Effects at High Strain Rates in Albuquerque in February, 1973.

Dr. E A. Starke, Jr. attended the Spring Meeting of AIME in Philadelphia in May, 1973 and was Chairman of a Session on the Advances in the Physical Metallurgy of Aluminum alloys.

Dr. E. A. Starke, Jr. was elected secretary of the Nonferrous Metals Committee of AIME and Chairman of a Committee to organize a symposium on modulated structures to be held in the Spring of 1974.

Dr. B. R. Livesay participated in a workshop for the preparation of a policy statement on deterioration of engineering materials for the National Commission on Materials Policy, June 26-28, 1972.

Dr. B. R. Livesay attended the 2nd National Conference on Crystal Growth, Princeton, July 30-Aug. 3, 1972.

Dr. B. R. Livesay attended 18th Annual Conference on Magnetism and Magnetic Materials, Denver, Colorado. He presented a paper there: "Magnetic Anisotropy of Oriented Cobalt Films".

Dr. B. R. Livesay visited Dr. L. R.Kramer at the Martin Marietta Corporation, Denver, Colorado and discussed common interests in surface mechanics.

Dr. B. R Livesay participated in an Air Force Materials Laboratory program in conjunction with the Ga. Tech School of Textile Engineering to determine the failure modes and wear resistance of certain fiber materials.

Dr. B. R. Livesay participated in an Army Missile Command program concerning the development and analysis of certain magnetic garnet materials for microwave applications.

## PATENTABLE INVENTIONS

No patentable inventions have resulted from the sponsored research.

## REFERENCES

1. B. R. Livesay and E. A. Starke, Jr., Acta Met. 21, 247 (1973).
2. G. Y. Chin, W. F. Hosford, and D. R. Mendorf, Proc. Ray. Soc. A. 309, 433(1969).

Respectfully submitted:

Dr. E. A. Starke, Jr.
Co-Principal Investigator

Dr. B. R. Livesay
Co-Principal Investigator

Security Classification

# DOCUMENT CONTROL DATA - R & D

*(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)*

| | |
|---|---|
| 1. ORIGINATING ACTIVITY (Corporate author)<br>Georgia Institute of Technology<br>School of Chemical Engineering & Physical Sciences Div<br>Atlanta, Georgia 30332 | 2a. REPORT SECURITY CLASSIFICATION<br>Unclassified<br>2b. GROUP |

3. REPORT TITLE

Effects of Interface Phenomena on the Mechanical Behavior of Metals

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Scientific Interim April 1, 1972 to March 31, 1973

5. AUTHOR(S) (First name, middle initial, last name)

Edgar A. Starke, Jr. and Billy R. Livesay

| 6. REPORT DATE | 7a. TOTAL NO. OF PAGES | 7b. NO. OF REFS |
|---|---|---|
| June, 1973 | | |

| | |
|---|---|
| 8a. CONTRACT OR GRANT NO.<br>AFOSR-71-2064<br>b. PROJECT NO.<br>c.<br>d. | 9a. ORIGINATOR'S REPORT NUMBER(S)<br>9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) |

10 DISTRIBUTION STATEMENT

Approved for public release; distribution unlimited

| 11 SUPPLEMENTARY NOTES | 12. SPONSORING MILITARY ACTIVITY |
|---|---|
| Tech and Journal<br>Acta Metallurgica V21 n3<br>pp 247-254 March, 1973 | Air Force Office of Scientific Research (NE)<br>1400 Wilson Boulevard<br>Arlington, Va. 22209 |

13. ABSTRACT

The effect of various interface factors on the mechanical behavior of metals has been investigated. These include expitaial metal films on single crystal substrates, twin interfaces, and interfaces produced by the decomposition of supersaturated solid solutions. For the composite samples, elastic nodulus differences were found to provide the primary strengthening effect. Multilayered specimens, utilizing this strengthening effect, show an increase in yield strength of 100% over that expected for the combined strengths of the pure materials.

DD FORM 1 NOV 65 1473

Security Classification

GEORGIA INSTITUTE OF TECHNOLOGY
ATLANTA, GEORGIA 30332

SCHOOL OF
CHEMICAL ENGINEERING

December 11, 1974

SUBJECT: End-of-The-Year-Report
ONR Task-Order No.
N00014-67-A-0159-0015
NR 031-750

TO: Dr. W. G. Rauch, Director
Metallurgy Program
Meterial Sciences Division
Office of Naval Research
Department of the Navy
800 North Quincy Street
Arlington, Virginia 22217

REFERENCE: (A) 471:BAM:las
NR 031-750
17 October 1974

ENCL: (a) Three (3) copies of Subject Report

1. In accordance with your letter, Reference (A), we hereby submit the enclosure (a) report.

Very truly yours,

Edgar A. Starke, Jr.
Principal Investigator

EAS:ejh
cc: Office of Naval Research Resident Representative
Room 276
Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

End of the Year Report

ONR Rask Order No. N00014-67A-0159-0015
NR 031-750

December 9, 1974

## A. Deformation Mechanisms in Beta Titanium Alloys

This research program deals with the microstructures and mechanical properties of beta titanium alloys as influenced by solute content and thermal processing. Emphasis has been on the relationship between strength deformation modes, texture development and ductility with studies being made on both single and polycrystalline specimans of binary Ti-V. During the past year attention has been focused on the influence of the metastable $\omega$ phase, (formed during quenching or aging) o~~r~~n deformation twinning and embrittlement.

There has been considerable attention given in the literature to the formation of the $\omega$ phase in Beta titanium alloys and the ensuing embrittlement that occurs in certain compositions and volume fractions. In the present study we have found that the presence of $\omega$ phase (athermal or isothermal) also induces {112}, a/6 [110] twinning as a deformation mode in Ti-V. We have further found that there is a distinct slip-to-twin transition at the point of $\omega$ embrittlement (if the volume fraction is high enough) or at the point during aging where the $\omega$ phase loses coherency and non-burgers $\alpha$ begins to form. It appears that the ability of the material to twin is a critical factor in retaining ductility while aging in the $\beta$-$\omega$ region. These observations have led to electron microscopy (TEM) studies aimed at understanding the twinning mechanism in two-phase $\omega$-$\beta$ systems as affected by size, shape, misfit and volume fraction of $\omega$. Mechanisms have been considered in a current paper (manuscript in preparation) recently presented at the Fall 1974 TMS meeting in Detroit. The key points are:

a) β matrix slip modes are not compatable with the hexagonal symmetry of the ω phase hence slip requires either reversion of ω or deformation confined to the matrix. At high volume fractions of isothermal ω the latter situation prevails with ensuing macro-brittleness and micro-ductility.

b) At moderate volume fractions of coherent ω, the reduced symmetry and internal strains produced during ω formation, i.e. contraction perpendicular to {111} planes, favor twinning. Since the dilations are reversible, (deFontaine, Paton and Williams) it is proposed that ω reverts in advance of the twin interface and reforms afterward. Consideration of atomic movements indicate that if this were not true, ω would not exist as it does within the twins.

c) Differences in twin mode with lattice misfit (e.g. {332} twins in βIII) are not explained at this point.

B. Implication of ONR Contract Research in Terms of Applied Significance

The research conducted thus far on this contract has shown that the deformation mechanisms of beta titanium alloys can be greatly influenced by variations in solute content and/or the presence of small amounts of a second phase. Variations in deformation mechanisms result in variations in texture development during processing. The fundamental understanding obtained from correlations of structure, composition, deformation mechanisms and properties will aid in the design of processing parameters necessary for improved product control. In this regard, both workability during fabrication and property control of the end product are considered.

C. Publications, Reports and Talks

"The Mechanical Behavior of Quenched and Aged Beta Isonorphous Titanium-Vanadium Alloys", H. G. Paris, B. G. LeFevre and E. A. Starke, Jr., TMS-AIME Fall Meeting, Detroit 1974, Abstract Session on Mechanical Behavior.

1. Fu-Wen Ling, E. A. Starke, Jr., and B. G. LeFevre, "Deformation Behavior and Texture Development in Beta Ti-V Alloys." Met. Trans. 5, Jan. 1974, 179.

2. H. G. Paris, B. G. LeFevre and E. A. Starke, Jr., "The Mechanical Behavior of Quenched and Aged Beta Isomorphous Titanium Alloys", Submitted to Met. Trans.

D. Other Contracts

"The Effect of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation," Air Force of Scientific Research.

Respectfully submitted,

Edgar A. Starke, Jr.
Professor
Principal Investigator

Bruce G. LeFevre
Associate Professor
Associate Investigator