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TITLE **OMEGA PHASE FORMATION IN TITANIUM AND
TITANIUM ALLOYS**

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OMEGA PHASE FORMATION IN TITANIUM AND TITANIUM ALLOYS*

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Abstract

Although the response of titanium alloys to dynamic loading is receiving increased attention in the literature (particularly in the area of shear-band formation), a more limited experimental database exists concerning the detailed structure/property relationships of titanium alloys subjected to shock loading. In this study, preliminary results concerning the influence of alloy chemistry on the propensity of omega-phase formation and its structure in three titanium alloys are presented. The influence of shock-wave deformation on the phase stability and substructure evolution of high-purity (low-interstitial) titanium, A-70 (3700 ppm oxygen) titanium, and Ti-6Al-4V were probed utilizing real-time velocity interferometry (VISAR) and "soft" shock-recovery techniques. VISAR wave profiles of shock-loaded high-purity titanium revealed the omega-phase pressure-induced transition to occur at approximately 10.4 GPa. Wave profile measurements on A-70 Ti shocked to pressures up to 35 GPa and Ti-6Al-4V shocked to pressures up to 25 GPa exhibited no evidence of a three-wave structure indicative of a pressure-induced phase transition. Neutron and X-ray diffractometry and TEM analysis confirmed the presence of retained ω -phase in the electrolytic-Ti and the absence of ω -phase in the shock-recovered A-70 Ti and Ti-6Al-4V. Suppression of the α - ω in A-70Ti, containing a high interstitial oxygen content, is seen to simultaneously correlate with suppression of deformation twinning. It is postulated that the suppression of the α - ω phase transition in titanium is related to increased lattice shear resistance due to interstitial oxygen. This is considered to be a significant new observation which is believed to be basic to the mechanism of the α - ω pressure-induced transition. Neutron diffraction was used to measure the in-situ bulk lattice constants and volume fraction of the α and ω phases in the recovered high-purity titanium samples that were shock loaded. A volume fraction of ~28% ω -phase with lattice parameters $a=0.4614$ nm and $c=0.2832$ nm was measured in the 11 GPa shock-recovered sample. The influence of alloy content on the kinetics of formation / retention of ω -phase and substructure evolution is discussed and contrasted in light of previous literature studies.

Phase transition
spec

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Introduction

Although the response of titanium alloys to dynamic loading and instabilities continues to be examined, no self-consistent set of experimental data exists concerning the influence of chemistry on the detailed structure/property relationships of titanium alloys subjected to shock loading. These studies, in particular those focused on the polymorphic omega-phase transition at high pressure, are complicated by the known sensitivity of the deformation response of titanium to alloying additions, especially interstitials[1]. The ω -phase transformation in α -Ti under shock or hydrostatic soaking conditions exhibits a large hysteresis that is responsible for retention of the high-pressure ω -phase to atmospheric pressure[2,3]. The ω -phase induced in pure Ti is morphologically similar to ω -phase formed in as-quenched β -phase alloys based on Zr, Ti, and Hf[4]. Crystallographically, the phase transformation is believed to be a diffusionless displacive transition[3,4]. It has been proposed that the transition results from an ordered atomic displacement (shift) of close-packed $\langle 1210 \rangle$ alpha planes lying in the (0001) plane resulting from the propagation of a lattice-displacement wave through the crystal involving atomic shuffles[4]. The movement of these linear defects shift the close-packed hexagonal rows of the structure into the ω -phase in response to softening of select phonon modes. Under high pressure the stability of the alpha lattice to $\langle 1210 \rangle$ slip decreases leading to the α - ω transition[4]. This phonon softening is a precursor to a first-order martensitic-type transition[2,4].

The object of the present study was to investigate the α - ω shock-induced transition in titanium using "real-time" VISAR and shock recovery techniques. Experiments were conducted on three titanium alloys to probe the influence of alloy chemistry on the high-pressure phase stability of titanium under shock conditions. Characterization of the structure of retained ω -phase in shock-loaded titanium was done utilizing neutron diffraction and transmission electron microscopy.

Experimental Procedures

This investigation was performed on electrolytic alpha titanium(hereafter referred to as high-purity Ti), supplied by the Alta Group as 38mm x 100mm x 254-mm-bar stock, Ti-6Al-4V(hereafter Ti-6-4) in the form of 120-mm-dia-bar stock, and on A-70 AMS 4921 Titanium(hereafter A-70 Ti) in the form of 50-mm-dia-bar stock. The analyzed chemical compositions (in wt. %) of the titanium materials studied are listed in Table I. The high-purity Ti was cross-rolled in multiple passes at room temperature from 38 mm to 12.5 mm with intermediate anneals, (600°C for 30 minutes followed by a water quench) at 75 and 50 % rolling reductions. The high-purity Ti was recrystallized at 600°C for 4 hours, followed by a water quench, yielding an equiaxed grain structure with a 20 μm grain size. The Ti-6-4 was studied in the as-received condition; the starting microstructure possessing a duplex microstructure, sometimes called a bimodal microstructure, comprised of lamellar areas of α and β and equiaxed α grains of nominally 3 μm . The A-70 Ti was also studied in the as-received condition; the starting microstructure possessing an equiaxed microstructure with an α grain size of nominally 30 μm . Ultrasonic shear and longitudinal wave measurements of the titanium studied revealed the average sound speed to be 4.92, 4.95, and 4.87 km/sec for the high-purity Ti, Ti-6-4, and the A-70 Ti, respectively.

To investigate the influence of alloy content on ω -phase formation, wave profile (VISAR) and shock recovery experiments were conducted on the three titanium alloys as a function of shock pressure. Shock recovery experiments were performed on an 80-mm single-stage launcher utilizing a shock assembly consisting solely of titanium[5,6]. The wave profiles were measured with a VISAR built at Los Alamos using the design developed by Willard Heming[7]. Precision of the wave velocity measurements is believed to be approximately 1% in particle velocity. Specially designed photomultiplier circuits were utilized that had 1 ns risetimes. Symmetric impacts were performed in all VISAR shots. The VISAR wave profile experiments had tilts at impact of the order of 1 mrad for gas shots and 3 mrad for powder

shots. Samples for optical metallography and transmission electron microscopy (TEM) were sectioned from the heat-treated and deformed samples. TEM foils were jet-polished with a solution of 84 % methanol, 10% butanol, and 6% perchloric acid at -40°C and 10 volts using a Struer's Electropolisher. Observation of the foils was made using a JEOL 2000EX at 200kV, equipped with a double-tilt stage. Neutron diffraction experiments were done on the High Intensity Powder Diffraction (HIPD) at the Los Alamos Neutron Scattering Center (LANSCE). Bulk specimens were examined without special preparation because the neutron penetration is large enough to sample the entire specimen.

TABLE I - Titanium Alloys Chemical Compositions (wt.%)

ELEMENT	High-Purity Ti	Ti-6Al-4V	A-70 Ti
O	360 ppm	0.18 wt%	0.37 wt%
C	60 ppm	200 ppm	170 ppm
N	10 ppm	110 ppm	240 ppm
H	14 ppm	7 ppm	8 ppm
Al	4 ppm	6.33 wt%	-
V	3 ppm	4.23 wt%	-
Fe	5 ppm	0.2 wt%	0.18 wt%
Ti	BAL	BAL	BAL

Results and Discussion

VISAR wave profile measurements were made on high-purity Ti from 6 to 22 GPa. Below 10 GPa a classic elastic-plastic two-wave structure was observed. At 15 GPa impact stress a three-wave structure (elastic plus two bulk waves) was observed (Figure 1), characteristic of a high pressure first-order phase transition. The best estimate of the transition pressure is 10.4 GPa. Several shots were fired at 15 GPa. All shots reproduced this three-wave structure although there was some variation in the transition pressure and shape of the transition wave. Shots were fired on 4.5-mm and 9-mm-thick samples to see if the transition pressures were comparable in samples with this range of thickness. Both samples exhibited approximately the same transition pressure. At 22 GPa only a two-wave elastic-plastic structure was observed, indicating the bulk transition wave had already overtaken the initial bulk shock in the α -phase. In other words, the high-purity Ti wave profiles are consistent with a material undergoing a first-order phase transition at 10.4 GPa with a small volume change.

VISAR wave profiles were made on A-70 Ti to study the α - ω transformation and the "apparent" phase transformation at 17.5 GPa reported by McQueen et al.[8]. The observed wave profiles up to 35 GPa consisted of a large elastic wave (1.8 GPa) followed by a bulk wave with a few nanosecond risetime. This elastic wave is over a factor of two larger than the elastic wave observed in the high-purity Ti. No evidence of a phase transition was observed over this pressure range. The VISAR U_s - U_p points smoothly extrapolated into the higher pressure flash-gap data above the kink in the U_s - U_p curve. The titanium used by McQueen et al. [8] in their Hugoniot measurements was very similar in chemical composition to the A-70 Ti used in this study. The VISAR data conclusively showed the kink in the U_s - U_p curve was an artifact of the flash-gap instrumentation. A possible reason for the kink in the flash-gap Hugoniot data was the large elastic wave prematurely closed the flash gaps. This interpretation is consistent with the shape of the low-pressure portion of the U_s - U_p curve. One would expect the greatest departure from linear extrapolation at low pressure and gradually diminishing to zero at a velocity slightly greater than the measured longitudinal velocity at zero pressure. Two-stage light gas gun data was used to extend the pressure range from 110 GPa to 260 GPa. The high pressure gun data fell on a linear extrapolation to high pressure of the VISAR / flash-gap Hugoniot data.

VISAR wave profile measurements were also made up to 25 GPa on Ti-6-4 to investigate the possible α - ω transition in this material [9]. Similar to A-70 Ti a classic elastic-plastic two-wave structure was observed over the entire pressure range investigated. The elastic precursor had an amplitude of 2.8 GPa, which was significantly larger than either the high-purity Ti or the A-70 Ti elastic waves. The VISAR data was positioned on a linear extrapolation to lower pressure of the flash-gap Hugoniot data. No evidence of a three-wave structure characteristic of a first-order phase transition was present. A qualification is in order about using loading-wave profiles to observe shock-induced phase transformations, because some types of transitions cannot be detected using this technique. The transitions that cannot be observed are transitions with small volume changes or have sluggish transformation kinetics. In these cases monitoring the sound velocities in the shock state[10] would be the appropriate tool to study these transitions.

Samples of the three titanium materials were also shock loaded to 11 GPa at room temperature and soft recovered to assess the post-shock substructure and phases present. Neutron and X-ray diffractometry and TEM selected area diffraction (SAD) analysis confirmed the presence of retained ω -phase in the electrolytic-Ti and absence of ω -phase in the shock-recovered A-70 Ti and Ti-6-4. Bulk x-ray identification of the ω -phase was found to be very sensitive to the sample surface preparation with careful polishing required to avoid mechanical reversion of the ω at the surface. The substructure of the shock-loaded titanium was observed to consist of a high density of deformation twins, interspersed with areas containing retained ω -phase.(Figure 2a) The deformation twins in the high-purity titanium were found to be (1121) type twins that are similar to those observed previously in shock-loaded Ti-6-4[5]. SADP analysis confirmed the orientation relationship $(0001)_{\alpha} // (1210)_{\omega}$ and $\langle 1120 \rangle_{\alpha} // \langle 0001 \rangle_{\omega}$ between the α and ω -phase previously determined for titanium[2] and for Zr by Rabinkin et al.[4]. The SADP (Figure 2b) additionally shows streaking of the ω -phase pattern parallel to $(1120)_{\omega}$ planes. These streaks are thought to be related to either: 1) the morphology of the ω -phase, 2) internal stacking faults within the ω formed during the transition, or 3) elastic distortion of the ω producing diffuse scattering in the direction of the distortion.

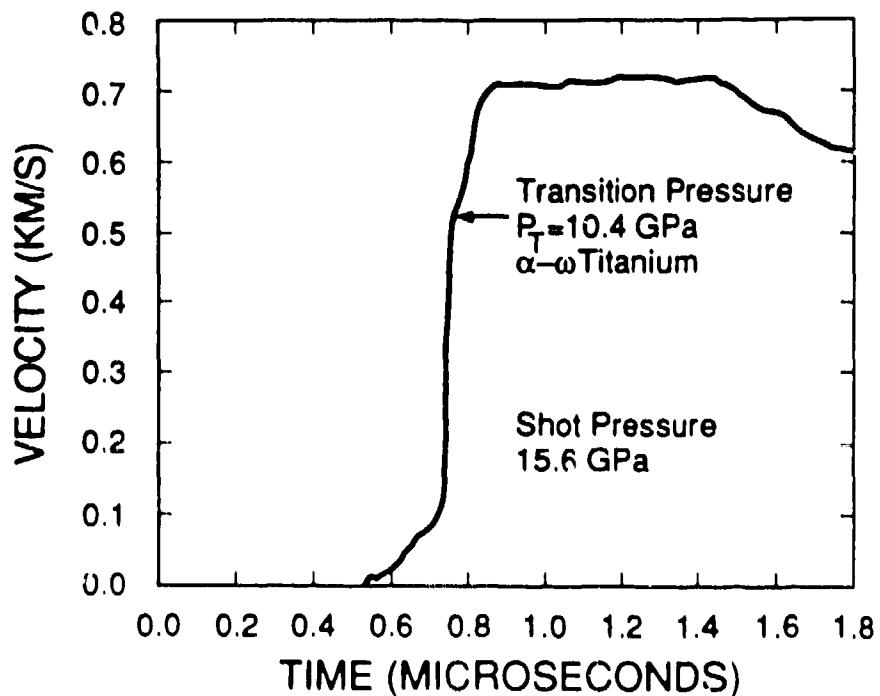


Figure 1 - VISAR wave profile showing the α - ω pressure-induced phase transition in high-purity titanium

The substructures of Ti-6-4 and A-70 Ti were both found to consist of primarily planar dislocation debris and in addition some isolated deformation twins in the case of Ti-6-4[5].

The planar nature of the substructure in these alloys is consistent with the aluminum and oxygen contents in the Ti-6-4 and A-70 Ti, respectively[1,11]. The coincident suppression of deformation twinning and ω -phase in the A-70 Ti is believed to be consistent with the influence of interstitial oxygen on the elastic constants, "c" lattice parameter, and dislocation mobility[1,11-13]. While it has been postulated that oxygen may suppress the α - ω transition in titanium due to phase equilibrium considerations[2,14], the results of the current study suggest that geometric constraints and increased lattice strength, rather than chemical effects may be controlling the transformation kinetics. Solid solution strengthening of the α -phase by oxygen has shown that the concentration dependence of the yield stress varies approximately by $c^{1/2}$ [1]. While increasing oxygen and impurity content suppresses the α - ω transition in titanium[14], increasing oxygen content is also known to suppress deformation twinning in titanium under quasi-static loading[11,13]. In polycrystalline titanium samples oriented so as to be stressed along their c-axes tended to deform largely by twinning in low oxygen (<0.15 wt.%) alloys and to deform by c+a slip in high oxygen (> 0.20 wt.%) alloys[13].

The concurrent absence of both twins and ω -phase in the A-70 Ti alloy therefore suggest a potential link between both shear processes. In iron alloys it has been shown that deformation twins are suppressed with increasing interstitial carbon content[15]. This observation was shown to be consistent with the fact that the lattice shear accompanying twinning in iron carries two-thirds of the interstitial carbon atoms to improper octahedral interstitial lattice sites requiring a large number of atomic shuffles[15]. This lack of lattice registry leads to a crystallographic restraint causing a change in the twinning stress of iron with carbon content, and eventually a total suppression of twinning. It is postulated that the relatively large scatter in the α - ω transition pressure[2] due to interstitial or impurity content is caused by increased lattice shear resistance, similar to the previously mentioned case of deformation twinning, leading to different α - ω transition pressures. While the interaction between the dislocations and interstitial oxygen is no doubt largely elastic in nature, potential interstitial ordering at high oxygen contents may also be responsible for suppression of twinning and the α - ω transition[13].

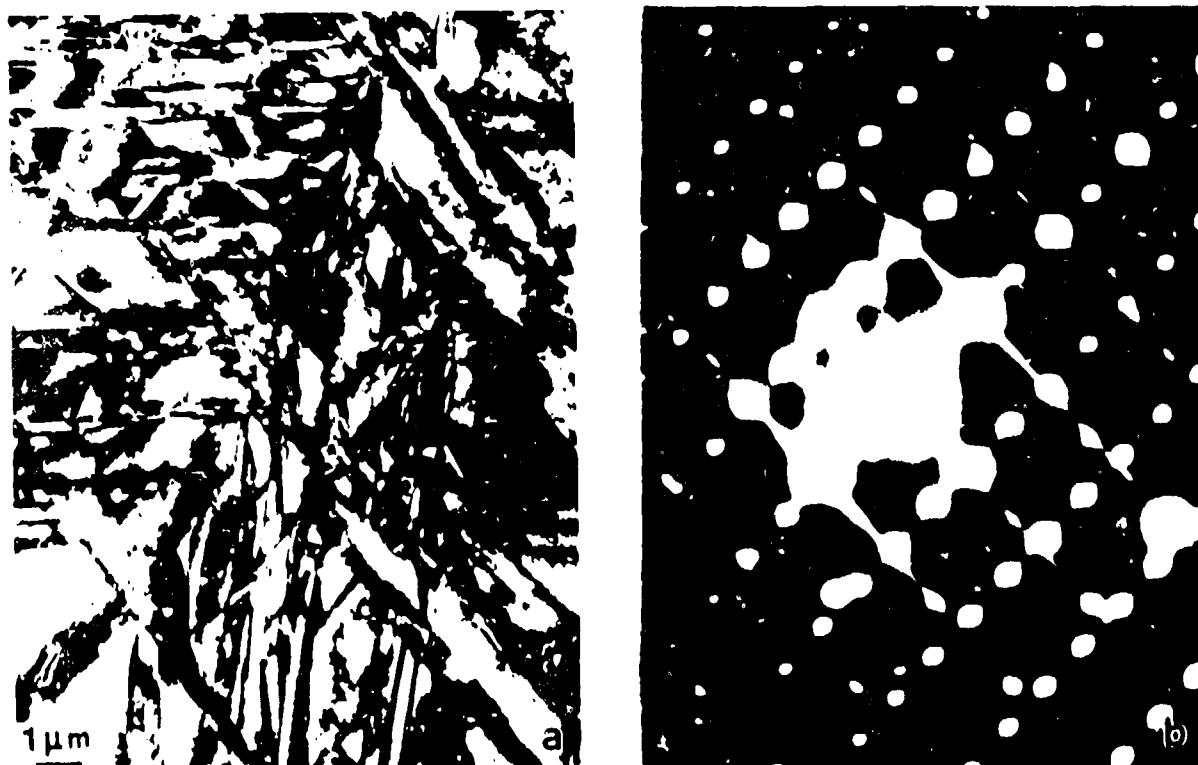


Figure 2 - a) Brightfield micrograph, and b) SADP showing retained omega phase and $\{1121\}$ deformation twins in high-purity titanium shock loaded to 11 GPa.

Neutron diffraction was used to measure the in-situ bulk lattice constants and volume fractions of the α and ω phases in the recovered samples that were shock loaded. These results are given in Table II. A volume fraction of ~28% ω -phase with lattice parameters $a=0.4614$ nm and $c=0.2832$ nm was measured in the 11 GPa shock-recovered high-purity titanium sample. The lattice parameters measured in the current study differ from those quoted in the literature[2]. Unit cell constants for ω -phase have previously given values for "c" ranging from 0.2813 to 0.2819 nm and "a" ranging from 0.4625 to 0.4643 nm[2]. Significant macrostrains are caused by the lattice mismatch between the α and ω -phases; the "a" of the α -phase tries to match the "c" of the ω -phase, and visa versa. These lattice values represent the global average lattice produced stains referenced to the lattice constants of pure titanium. This lattice mismatch is also seen to result in strains within both lattices which shows up as microstrain. The microstrains represent the distribution of peak broadening in the neutron diffraction pattern of the lattice spacings.

TABLE II - Strains and Lattice Constants for α - and ω -Titanium

	<u>High-Purity Ti</u>	<u>A-70 Ti</u>
volume fraction ω (%)	28	0
lattice constants (nm):		
$a(\alpha)$	0.29506(2)	0.29520(1)
$c(\alpha)$	0.46795(4)	0.46884(4)
$a(\omega)$	0.4614(1)	-
$c(\omega)$	0.2832(1)	-
micro-strain from peak widths (%):		
strain (α)	0.68	0.52
strain (ω)	0.74	-
macro-strain from lattice constants (%):		
$(a(\alpha) - a_0) / a(\alpha)$	0.05	-
$(c(\alpha) - c_0) / c(\alpha)$	0.19	-
$(a(\alpha) - c(\omega)) / a(\alpha)$	-4.02	-
$(c(\alpha) - a(\omega)) / c(\alpha)$	-1.40	-

The current shock-recovery findings are contrary to a previous study where shock loading pure Ti at room temperature to pressures of 12 to 50 GPa yielded no retained ω while finding retained ω if shock loading was conducted at 120K[3]. Shock recovery experiments in this study were conducted by shock loading the Ti in steel containers which may have influenced the ϵ_{res} and thermal history in the recovered samples[3]. Even in the 120K Ti shots[3], measurement of the phase distribution through the recovered sample thickness revealed the absence of ω at the near impact and rear sample surfaces while showing a uniform amount of retained phase in the sample interior. Due to the mechanical and thermal metastability of the ω -phase, it is believed that the ϵ_{res} in the sample, surface contact stresses, and thermal history recovery effects caused in the steel container resulted in reversion of the ω during release and deceleration at room temperature in the previous study. The variations in the phase-retention results graphically illustrate the importance of utilizing soft recovery techniques to accurately assess shock-induced structure/property relationships and thereby provide post-mortem physical data for comparison with real-time wave profile data.

Summary and Conclusions

Based on a study of the influence of shock-loading and alloy chemistry on the α - ω pressure-induced phase transition in titanium, the following conclusions can be drawn:

1. VISAR wave profiles of shock-loaded high-purity titanium revealed the omega-phase

pressure induced transition to occur at approximately 10.4 GPa.

2. Wave profile measurements on A-70Ti and Ti-6Al-4V shocked up to pressures of respectively 35 and 25 GPa exhibited no evidence of a three-wave structure indicative of a phase transition.

3. Neutron diffraction characterization of high-purity titanium shock loaded to 11 GPa and "soft" recovered displayed a 28% volume fraction of retained ω -phase with in-situ bulk lattice parameters of $a=0.4614$ nm, $c=0.2832$ nm, and $c/a=0.6137$.

4. Suppression of the α - ω in A-70Ti, containing a high interstitial oxygen content, is seen to simultaneously correlate with suppression of deformation twinning. It is postulated that the suppression of the α - ω phase transition in titanium is related to increased lattice shear resistance due to interstitial oxygen. This is considered to be a significant new observation which is believed to be basic to the mechanism of the α - ω pressure-induced transition.

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References

1. H. Conrad, "Effect of Interstitial Solutes on the Strength and Ductility of Titanium," *Prog. Matls. Sci.*, 26 (1981), 123-403.
2. S.K. Sikka, Y.K. Vohra, and R. Chidambaram, "Omega Phase in Materials," *Prog. Matls. Sci.*, 27 (1982) 245.
3. A.R. Kutsar and V.N. German, *Titanium and Titanium Alloys*, ed. J.C. Williams and A.F. Belov (New York: Plenum Press, 1982), 1633.
4. A. Rabinkin, M. Taliankar, and O. Botstein, "Crystallography and a Model of the α - ω Phase Transformation in Zirconium," *Acta Metall.*, 29 (1981) 691-698.
5. G.T. Gray III and C.E. Morris, *Sixth World Conference on Titanium*, ed. P. Lacombe, R. Tricot, and G. Beranger, (Paris, France: Les Editions de Physique, 1989), 269-274.
6. G.T. Gray III, *High Pressure Compression of Solids*, ed. J.R. Asay and M. Shahinpoor, (New York: Springer-Verlag, 1992), in press.
7. W.F. Heming, "Velocity Sensing Interferometer (VISAR) Modification," *Rev. Sci. Instrum.*, 50, #1 (1979) 73.
8. R.G. McQueen, S.P. Marsh, J.W. Taylor, J.N. Fritz, and W.J. Carter, "The Equation of State of Solids from Shock Wave Studies," in *High Pressure Impact Phenomena*, ed. R. Kinslow, (Academic Press, New York, 1970), 293.
9. C.E. Morris, M.A. Winkler, and A.C. Mitchell, "Ti-6Al-4V Alloy Wave Profile Measurements in the Shadow Region," in *Shock Waves in Condensed Matter-1987*, ed. S.C. Schmidt and N.C. Holmes, (Elsevier Science Publications, B.V., 1988) 265-268.

10. J.M. Brown and R.G. McQueen, "Melting of Iron Under Core Conditions," Geophys. Res. Lett., 7 (1980) 533.
11. J.C. Williams, A.W. Sommer, and P.P. Tung, "The Influence of Oxygen Concentration on the Internal Stress and Dislocation Arrangements in α Titanium," Metall. Trans., 3 (1972) 2979-2984.
12. J.C. Williams, A.W. Thompson, C.G. Rhodes, and J.C. Chesnutt, "Strengthening and Fracture Mechanisms in Titanium Alloys Critical Review," in Titanium and Titanium Alloys, eds. J.C. Williams and A.F. Belov, (Plenum Press, New York, 1982) 467-496.
13. N.E. Paton, J.C. Williams, and G.P. Rauscher, " " in Titanium Science and Technology, eds. R.I. Jaffee and H.M. Burke, (Plenum Press, New York, 1973) 1049-1069.
14. Y.K. Vohra, S.K. Sikka, S.N. Vaidya, and R. Chidambaram, "Impurity Effects and Reaction Kinetics of the Pressure-Induced α - ω Transformation in Ti," J. Phys. Chem. Solids., 38 (1977) 1293-1296.
15. C.L. Magee, D.W. Hoffman, and R.G. Davies, "The Effect of Interstitial Solutes on the Twinning Stress of B.C.C. Metals," Philos. Mag., 23 (1971) 1531-1540.