Heavy Ion Irradiation of a Ti-6Al-4V Alloy

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FUSION TECHNOLOGY INSTITUTE

UNIVERSITY OF WISCONSIN

MADISON WISCONSIN

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P. Wilkes and G.L. Kulcinski

Fusion Technology Institute
University of Wisconsin
1500 Engineering Drive
Madison, WI 53706

http://fti.neep.wisc.edu

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Abstract

A titanium-6% Al-4% V alloy has been investigated after irradiation with 17.5 MeV Cu ions up to 1.5 dpa in the temperature range 250°C - 450°C. No voids were seen but copious precipitation was observed. The diffraction pattern from the precipitates at 450°C was consistent with β titanium in the Burgers orientation with the α matrix. The implications of this radiation-induced, non-equilibrium precipitation are discussed in terms of current phase stability theory.

1. Introduction

Titanium alloys have been recently proposed as possible structure materials for fusion reactors because of their low induced radioactivity, high strength to weight ratio and good fabricability at reasonable cost.[1] While there is a great deal known about the physical, mechanical, and thermal properties of a wide variety of Ti alloys, very little is known about the effects of neutron irradiation on these alloys. Furthermore, what data does exist[2] is usually from relatively low temperature tests (<200°C) and it is highly unlikely that fusion reactors could operate economically in that range.

The objective of the present work is to investigate the microstructure of a promising Ti alloy, Ti-6Al-4V, after high temperature (250°-450°C) bombardment with high energy ions. These preliminary investigations were primarily aimed at finding out whether this Ti alloy was susceptible to the formation of voids like most other structural metals in a high temperature, neutron environment. A secondary, but almost equally important goal was to define the experimental conditions under which simulation of high damage rates could be performed in Ti alloys. In the process of this work, we discovered a rather unexpected change in the precipitate microstructure which certainly should be pursued in the future.

2. Experimental Procedure

The Ti-6Al-4V alloy used for this work was provided by J. Davis of McDonnell Douglas Corporation. The alloy was in the fully annealed condition and originated from TIMET Heat N-0358. Three millimeter discs were cut from the 0.03 cm sheet and electropolished to provide a clean flat surface. The specimens were irradiated in the UHV irradiation chamber on the University of Wisconsin Van Der Graaf tandem accelerator described previously.[3]

The ions used were 17.5 MeV Cu^{4+} and during the irradiation the vacuum was maintained at $<5 \times 10^{-9}$ torr. The specimens were irradiated to 1.5 dpa (1 μm depth) at 250°, 350°, and 450°C. The damage curve as calculated from the Manning-Mueller code[4] is shown in figure 1. The average damage rate at 1 μm depth was $\sim 3 \times 10^{14}$ dpa/sec and all specimens were prepared for electron microscopy by polishing down to that depth before electro-thinning for TEM. Examination of the microstructure at 1 μm depth gives a reduced dose as compared with the peak in figure 1 but eliminates the possibility of contamination from implanted copper ions.

3. Results

The as-received alloy was typical of annealed α/β titanium alloys (fig. 2). During polishing the small amounts of β polished away and only the major α phase remained for examination. Control samples alongside the irradiated samples in the hot zone of the irradiation chamber retained this structure without change.

Irradiation at all temperatures studied introduced a profound change in microstructure. Fig. 3 shows the result at 250°C. It consists of a dense tangle of precipitates and dislocation loops. The structure is so dense that no detail could be resolved. Such damage structures are typical of irradiation below the void-forming temperature range where damage is not annealing out rapidly during irradiation. As a result loop damage is accumulated.

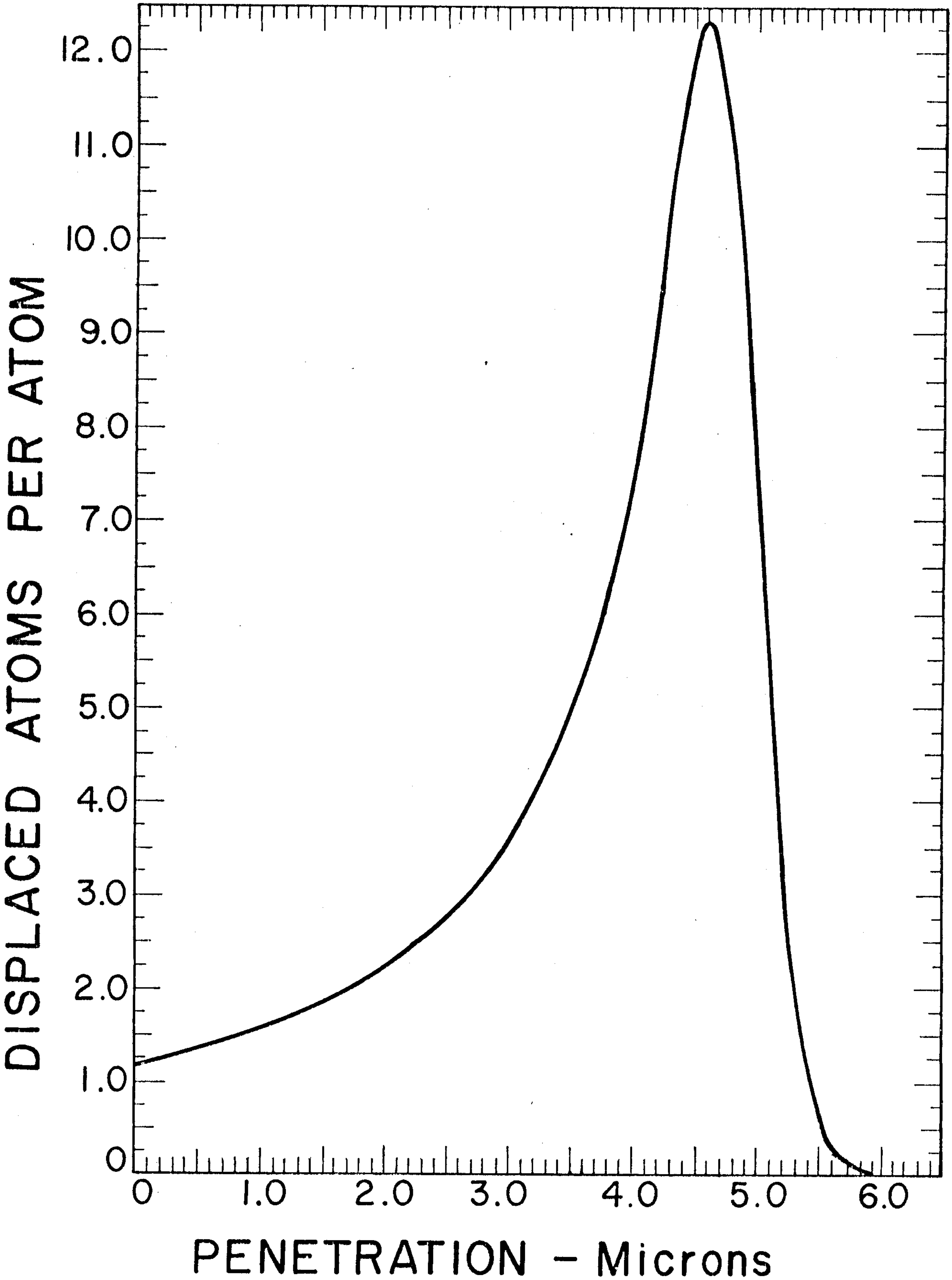


Fig. 1. Damage curve calculated after Manning & Mueller[4] for 17.5 MeV Cu ions. A displacement energy of 19.0 eV was used and the total fluence was 1×10^{16} ions/cm^2.

.25μ

Fig. 2. The as annealed microstructure of the Ti-6Al-4V alloy. This remained unchanged for unirradiated samples in the radiation chamber.

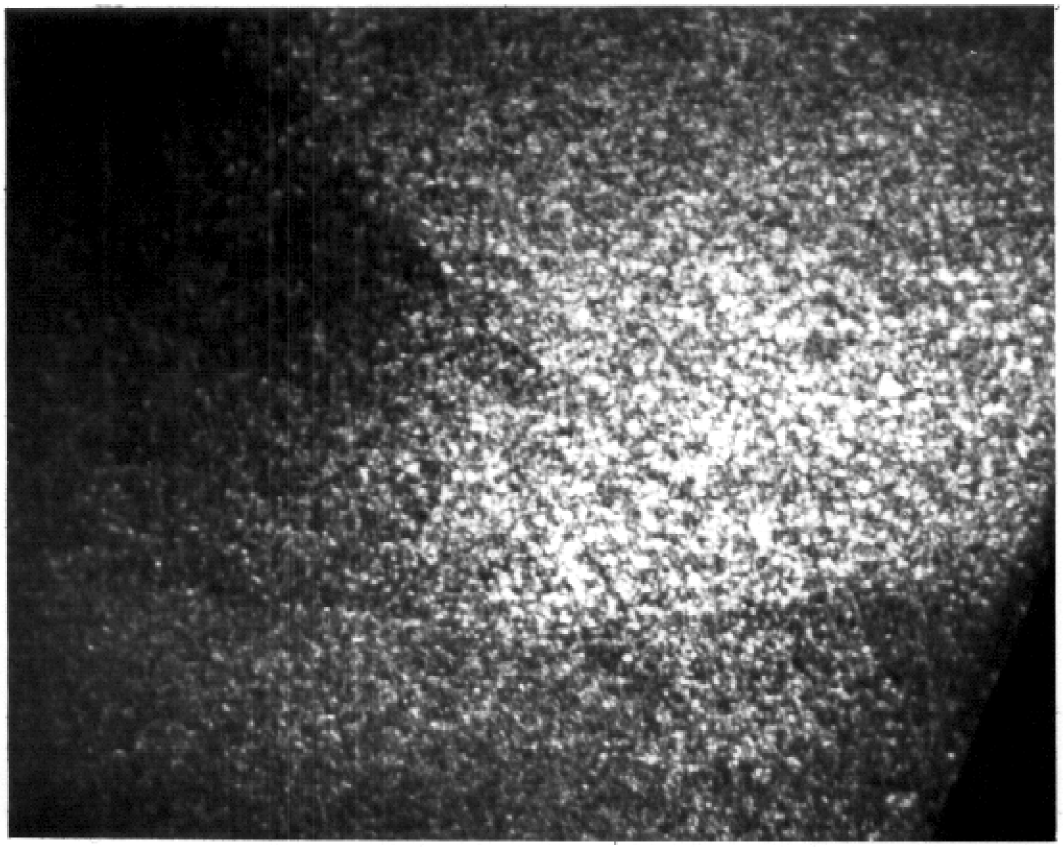


Fig. 3. Microstructure after irradiation at 250°C to 1.5 dpa at 1 μm depth, showing dense black spot damage.

At 350°C, fig. 4, the structure is similar but coarser, individual particles and/or loops can now be resolved in strain field contrast. In all specimens irradiated at 250°C and 350°C, no changes occurred in the diffraction patterns, in particular no additional reflections were observed from precipitates. The rate of annealing during irradiation is increased at 350°C and it is this which gives the somewhat coarser microstructure. However, the damage remains too dense for detailed loop or precipitate analysis.

At 450°C the process of loop annealing occurs much more rapidly and extensive loop damage is no longer visible (fig. 5). Precipitates are resolvable and now dominate the structure. Individual particles are clearly visible even though a very high precipitate density is present. Occasional extra reflections are visible on the diffraction pattern at this stage and were used to clarify the microstructure by dark field microscopy as in Fig. 5b. These extra reflections were consistent with the bcc β phase (fig. 6) oriented in the Burgers relation with the cph matrix

$$(00.1)_{\alpha} \parallel (110)_{\beta} .$$

The precipitates line up along forest dislocations as fig. 5 shows. In addition a strong tendency to form partially ordered precipitate arrays at grain boundaries is evident. The size of precipitates at grain boundaries was generally smaller than those in the matrix as fig. 5b illustrates.

4. Discussion

The formation of loops and dislocation tangles is expected for alloys irradiated in the 0.1 - 0.3 T_m temperature range and it is therefore not surprising to find such microstructures in these alloys. The absence of voids is encouraging and no swelling problem should be experienced for this

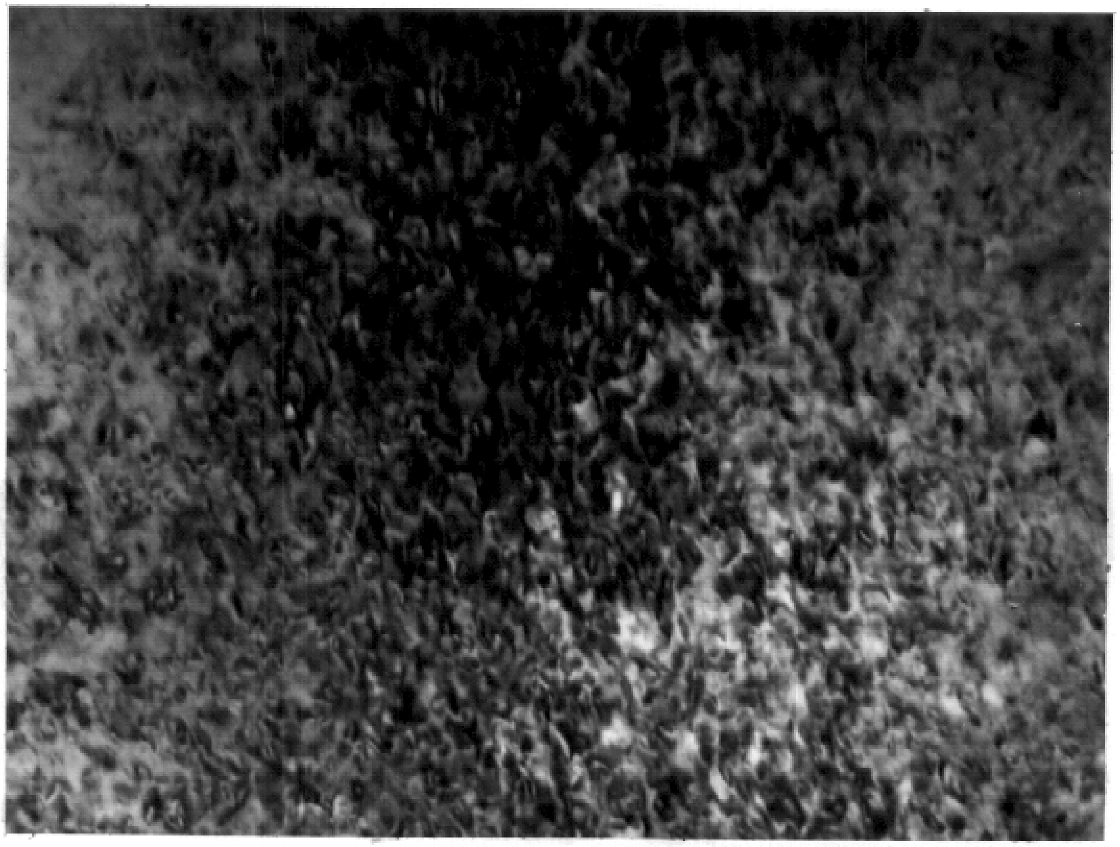
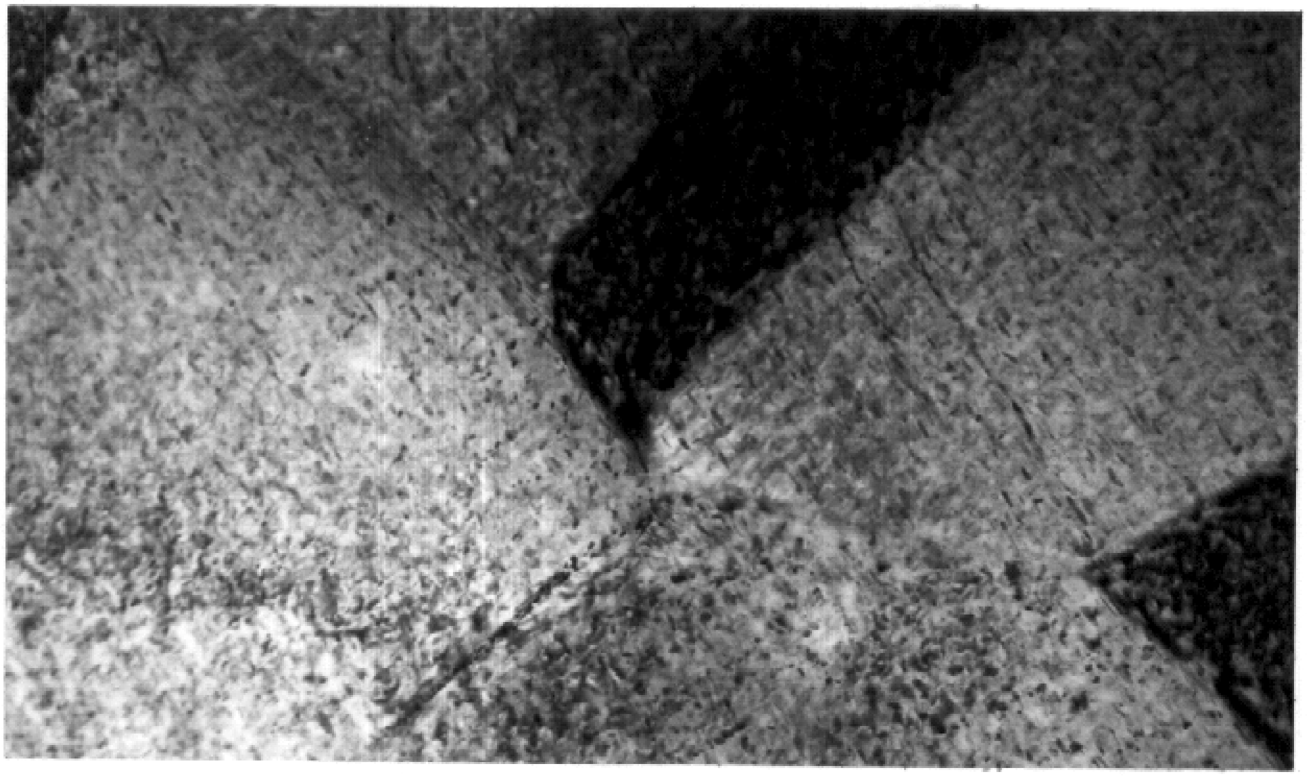
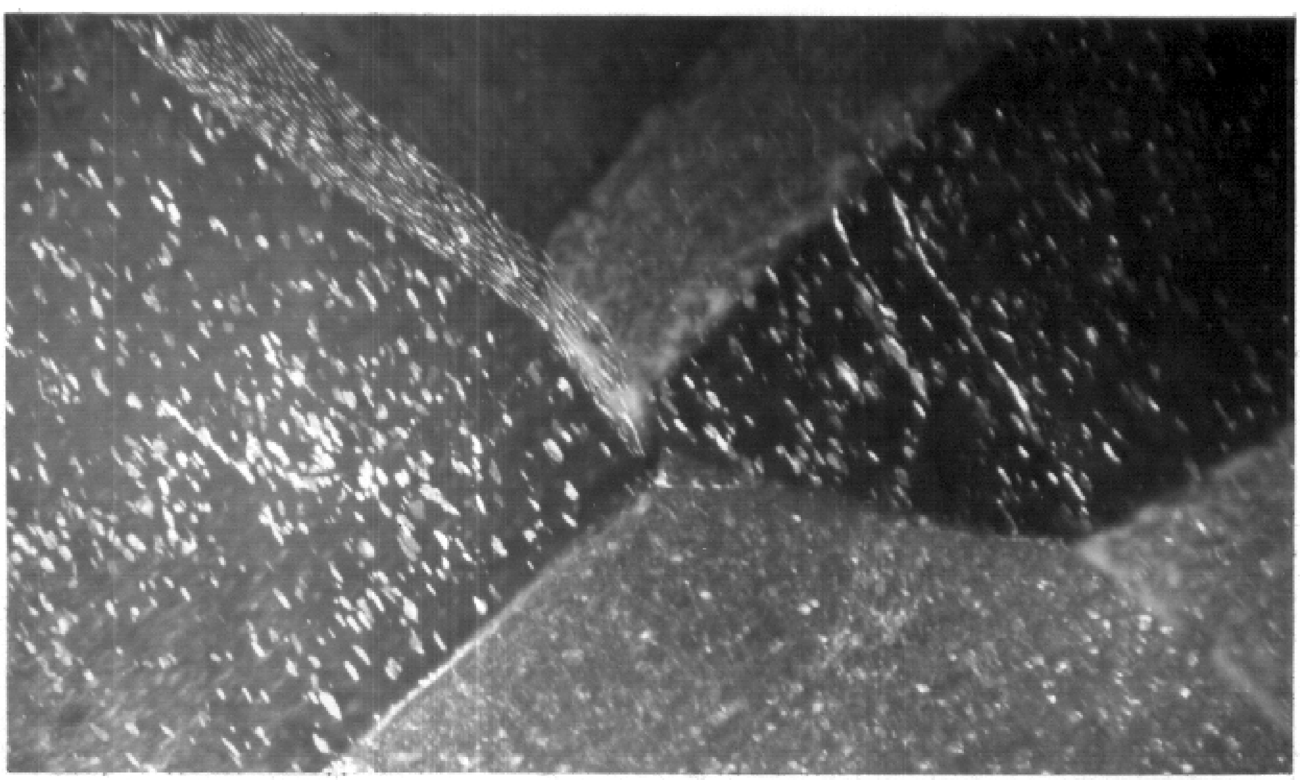


Fig. 4 As fig. 3 but at 350°C showing dense small loop damage.



a.



b.

Fig. 5a. Microstructure after irradiation at 450° showing precipitation.

b. Dark field using the precipitate reflection marked in fig. 6.

alloy in the temperature and dose range studied. To decide whether this resistance to swelling occurs at higher temperatures and larger doses requires further study.

The surprising and dominant feature of the damage microstructures is the dense precipitation. The fine size of the precipitates precluded conclusive diffraction identification. However, extra reflections observed were consistent with β formation and not with other obvious possibilities such as hydrides. If it is assumed that 250°C - 450°C lies within the expected void formation range in this alloy, then the absence of voids could be the result of selective absorption of vacancies at precipitate interfaces.

The main problem is to explain the existence of β precipitates within previously annealed and equilibrated α grains. Two possibilities occur:

i) The Cu ions used in the irradiation changed the alloy composition sufficiently to produce constitutional supersaturation in the α phase. However, the total amount of copper added in this way only amounts to a few parts per million in the depth range of 1 μm (even allowing for radiation enhanced diffusion). Previous calculations[5] on nickel irradiated at a similar displacement rate and comparable temperature range showed negligible enhanced diffusion of copper to the front surface in times less than 10^5 secs. In the titanium case where the distance from the end-of-range to the 1 μm depth is almost twice as great, we think the Cu contamination effect should be negligible for the few hours of irradiation.

ii) Radiation Induced Precipitation

Precipitation may occur during irradiation for both kinetic and thermodynamic reasons. If the material is in a non-equilibrium state (supersaturated) then radiation may promote nucleation of incoherent precipitates.[6] Also defect-solute binding can cause local segregation toward (or away from) defect sinks.[7] If such segregation exceeds the solubility limit then precipitation will occur and the phase precipitated need not be the equilibrium phase.

It has also been proposed that thermodynamic stability may be affected by irradiation.[8,9] There is experimental evidence that high defect concentrations and fluxes in diffusion couples are also associated with non-equilibrium precipitation.[10] However, the internal energy associated with point defects is small compared with the free energy differences usually involved in phase changes. This remains true even for defect concentrations $\sim 10^{-4}$ which are regarded as high.

The precipitation observed in this study cannot be explained by the promotion of incoherent nuclei since the precipitates are coherent. However since the α in this two phase alloy was necessarily saturated with respect to β, supersaturation could easily be attained by either a small amount of solute segregation to dislocation loops or by a radiation induced shift in the free energy-composition curves. In the present work it is not possible to distinguish between these alternatives.

The implications of these observations for alloys under irradiation are clearly important. Obviously two phase alloys are more likely to become supersaturated than alloys well inside a single phase field irrespective of the instability mechanism. From an alloy design point of view solid solution strengthening is preferred over precipitation for the elimination of phase instabilities. For titanium alloys therefore fully α or fully β structures are more resistant to precipitation.

On the other hand, precipitates may be favorable. They could strengthen the material or act as void inhibitors and this possibility is an attractive one from an alloy design viewpoint.

5. Conclusions

a) Ti-6Al-4V alloy in the annealed state has been irradiated with 17.5 MeV Cu^{4+} ions to 1.5 dpa in the temperature range 250°C - 450°C.

b) No voids were observed up to this dose.

c) Copious precipitation occurred which was coarse enough at 450°C to give diffraction reflections consistent with β phase.

d) From current theories in the phase stability area two phase alloys are more likely than single phase alloys to become unstable and form precipitates under irradiation.

6. Acknowledgements

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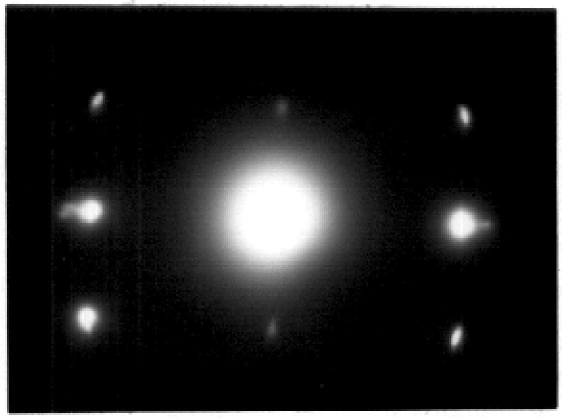
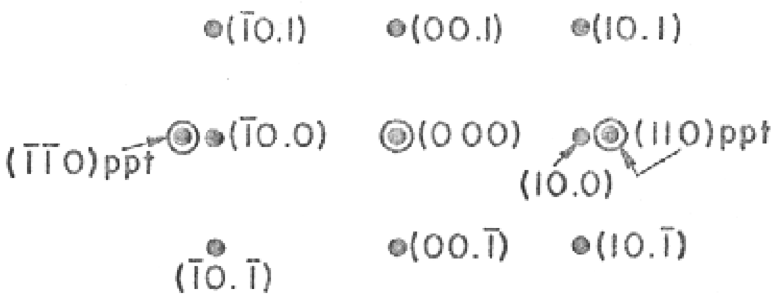


(a)



(b)

Fig. 6a. Diffraction pattern from the area shown in fig. 5. The dark field 5b) was taken with the precipitate reflection on the right.

b. Indexing, the cph α matrix (10.0) plane lies in the foil plane. Note the forbidden (00.1) and $(00.\bar{1})$ reflections occurring by double diffraction. The (110) reflections from the bcc β particles are shown. Misfit is minimized for the two structures if the $[1\bar{1}0]_\beta$ lies parallel with the $[00.1]_\alpha$. In that case the precipitate (002) lies in the foil plane.