

Available online at www.sciencedirect.com



journal of nuclear materials

Journal of Nuclear Materials 367-370 (2007) 627-632

www.elsevier.com/locate/jnucmat

# Effect of displacement dose and irradiation temperature on tensile and fracture toughness properties of titanium alloys

S. Tähtinen <sup>a,\*</sup>, P. Moilanen <sup>a</sup>, B.N. Singh <sup>b</sup>

<sup>a</sup> VTT Materials Performance, P.O. Box 1000, FI-02044 VTT, Espoo, Finland <sup>b</sup> Materials Research Department, Risø National Laboratory, DK-4000 Roskilde, Denmark

# Abstract

At a low dose level of 0.001 dpa the Ti6Al4V ( $\alpha + \beta$ ) alloy showed softening and at higher doses an increase in hardening and lack of work hardening after irradiation at 60 °C. The ( $\alpha + \beta$ ) alloy seemed to suffer from plastic instability when irradiated to a dose level of 0.3 dpa at 60 °C. At elevated temperatures a substantial amount of hardening was observed in the ( $\alpha + \beta$ ) alloy when tested in the irradiated condition. Earlier studies have shown that the fracture toughness behaviour of the irradiated Ti5Al2.5Sn ( $\alpha$ ) and ( $\alpha + \beta$ ) alloys were quite similar at ambient temperatures. At elevated temperatures, fracture toughness of the irradiated ( $\alpha + \beta$ ) alloy decreased more than that of the ( $\alpha$ ) alloy when compared to the results obtained in the unirradiated condition. The large irradiation hardening and loss of fracture toughness in the ( $\alpha + \beta$ ) alloy. © 2007 Elsevier B.V. All rights reserved.

# 1. Introduction

Based on an attractive combination of thermophysical, mechanical and radioactivity decay properties, titanium alloys have been identified as candidate structural materials for different components in fusion reactors. More specifically, two classical and industrially available alloys, Ti5A12.5Sn ( $\alpha$ ) and Ti6A14V ( $\alpha + \beta$ ), are being considered as candidate materials for flexible mechanical connectors between the blanket modules and the pressure vessel of ITER. The earlier results have clearly demonstrated that both tensile and fracture toughness properties of the ( $\alpha + \beta$ ) alloy were more strongly

E-mail address: seppo.tahtinen@vtt.fi (S. Tähtinen).

affected by neutron irradiation than that of the  $(\alpha)$ alloy particularly when irradiated and tested at 350 °C. Irradiations at 50 °C, on the other hand, to the same dose level of 0.3 dpa had rather similar effects on tensile as well as fracture toughness behaviour of both ( $\alpha$ ) and ( $\alpha + \beta$ ) alloys. Thus, the mechanical performance of the  $(\alpha + \beta)$  alloy appears to be more dependent on irradiation temperature than that of the  $(\alpha)$  alloy. On the basis of these results and the results of microstructural investigations it was concluded that the source of the high sensitivity of mechanical properties of the  $(\alpha + \beta)$  alloy to neutron irradiation was, most probably, due to irradiation enhanced diffusion and segregation of vanadium to the defect clusters and irradiation-induced precipitation of vanadium-rich precipitates [1,2]. In view of these results, it was deemed necessary to further investigate the effects

<sup>\*</sup> Corresponding author. Tel.: +358 20 722 1111; fax: +358 20 722 6990.

<sup>0022-3115/\$ -</sup> see front matter @ 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.jnucmat.2007.03.042

of irradiation temperature and displacement dose on mechanical properties of the  $(\alpha + \beta)$  alloy. We report the main results of these additional investigations.

# 2. Experimental details

Two types of high strength titanium alloys i.e.,  $(\alpha + \beta)$  and  $(\alpha)$  were studied in the as received condition. The  $(\alpha + \beta)$  alloy was mill annealed at 730 °C for 1.5 h followed by air cooling. The  $(\alpha)$  alloy was heat treated at 815 °C for one hour, and was also air cooled. Details of chemical composition and thermomechanical treatments have been described by Marmy et al. [3].

Tensile testing at elevated temperatures was carried out in a vacuum of  $10^{-5}$  Torr using a strain rate of  $1.3 \times 10^{-3}$  s<sup>-1</sup>. Single edge notched bend [SE(B)] fracture toughness specimens of dimensions  $3 \times 4 \times 27$  mm with notch and the 20% side grooves were used. The applied prefatigued crack length to specimen width ratio was about 0.5. Fracture resistance curves were determined following the ASTM Standard Test Method for J-Integral Characterisation of Fracture Toughness, E 1737-96 a constant displacement rate of  $2.5 \times 10^{-4}$  mm s<sup>-1</sup>.

Tensile specimens of the  $(\alpha + \beta)$  alloy were irradiated with fission neutrons in the BR-2 reactor at SCK CEN Mol at 60 °C to three neutron fluence levels of  $4.0 \times 10^{21}$ ,  $3.2 \times 10^{22}$  and  $3.5 \times 10^{23}$  n/m<sup>2</sup> (E > 1 MeV) corresponding to displacement doses of 0.001, 0.01 and 0.1 dpa (NRT). Tensile specimens were also irradiated at 350 °C to dose levels of 0.1, 0.2 and 0.3 dpa. Some tensile and fracture toughness specimens of  $(\alpha + \beta)$  alloy were also irradiated in the BR-2 reactor at 150 °C (the mean temperature varied between 139 and 159 °C) to a dose level of  $\sim$ 0.3 dpa. Irradiations were carried out with a neutron flux of  $2.1 \times 10^{17}$  n/m<sup>2</sup> s corresponding to a displacement rate of  $3.5 \times 10^{-8}$  dpa (NRT)/s. Details of irradiations at 60, 150 and 350 °C carried out in the BR-2 reactor at Mol are given in references [4,5]. Additional tensile specimens of Ti6Al4V alloy were irradiated at 150 °C to a neutron fluence level of  $3.1 \times 10^{24} \text{ n/m}^2$  (E > 1 MeV) corresponding to a displacement dose of 0.3 dpa (NRT). These irradiations were carried out with a neutron flux of  $3.5 \times 10^{17} \text{ n/m}^2 \text{ s}$  corresponding to a displacement rate of  $5 \times 10^{-8}$  dpa (NRT)/s. Irradiations were performed in an atmosphere of helium or a mixture of helium and argon in a fission reactor at KFKI/ **AEKI Budapest.** 

# 3. Results

1200

1000

# 3.1. Tensile behaviour

The dose dependence of the tensile properties of the Ti6Al4V alloy at the test temperature of 50 °C is presented in Fig. 1. Irradiation to a dose level of 0.001 dpa at 60 °C resulted in lower strength but similar work hardening behaviour compared to the unirradiated condition. At higher irradiation dose levels of 0.01 and 0.1 dpa at 60 °C tensile results show an increase in the yield strength, loss of work hardening capability and decrease in uniform and total elongations. The results suggest that the ( $\alpha + \beta$ ) alloy starts to suffer from plastic instability at a dose level of about 0.1 dpa at the irradiation temperature of 60 °C.

The dose dependence of the tensile properties of the  $(\alpha + \beta)$  alloy irradiated and tested at 350 °C is presented in Fig. 2. The irradiation at 350 °C to a dose level of 0.1 dpa causes a substantial amount of hardening in the  $(\alpha + \beta)$  alloy with negligible change in work hardening properties. The magnitude of hardening increases with increasing dose level with some decrease in the uniform elongation and practically no change in work hardening properties.

The increase in the yield strength of the  $(\alpha + \beta)$ alloy with dose tends towards a saturation level at a dose level of about 0.3 dpa when irradiated at 60 °C. At the irradiation temperature of 350 °C the increase in the yield strength with dose is much

unirradiated

0.001 dpa

т

0.1 dpa



Fig. 1. Dose dependence of tensile behaviour of Ti6Al4V alloy tested at 50  $^\circ$ C after neutron irradiation at 60  $^\circ$ C.



Fig. 2. Dose dependence of tensile behaviour of Ti6Al4V alloy tested at 350  $^{\circ}$ C after neutron irradiation at 350  $^{\circ}$ C.

steeper than at 60  $^{\circ}$ C and remains steep up to the dose level of 0.3 dpa (see Fig. 3).

The tensile curves of the unirradiated  $(\alpha + \beta)$ alloy shown in Fig. 4(a) reveal that the yield strength of the unirradiated alloy decreases from about 996 MPa to about 520 MPa when testing temperature increases from 50 to 350 °C. The amount of work hardening is relatively small and is almost constant in the test temperature range of 50–350 °C. The uniform elongation is about 10% in this test temperature range. The corresponding



Fig. 3. Dose dependence of yield strength of Ti6Al4V alloy at different temperatures. Open symbols correspond to unirradiated conditions.



Fig. 4. Tensile stress-strain curves of Ti6Al4V alloy (a) in unirradiated condition and (b) after neutron irradiation to a dose level of 0.3 dpa at different temperatures. Note that the test temperatures are marked on the appropriate stress-strain curves.

tensile curves of the  $(\alpha + \beta)$  alloy after neutron irradiation to a dose level of 0.3 dpa at different temperatures are shown in Fig. 4(b). The  $(\alpha + \beta)$  alloy shows very low uniform elongation at the test temperature of 60 °C and seems to suffer from plastic instability. At 350 °C, on the other hand, a relatively high uniform elongation and a positive work hardening are observed.

The dose and temperature dependencies of tensile deformation behaviour of the  $(\alpha + \beta)$  alloy shown in Figs. 1–4 illustrate the following interesting features of the effect of neutron irradiation on the hardening behaviour of this alloy:

- (i) At the irradiation temperature of 60 °C, both the yield stress and the flow stress are lower and the uniform elongation higher at the dose level of 0.001 dpa than that of the unirradiated alloy, i.e., this low dose irradiation causes softening rather than hardening and increases the ductility rather than causing a decrease (see Fig. 1)
- (ii) The dose dependence of radiation hardening is much stronger at 350 °C than at 60 °C (Figs. 2 and 3).
- (iii) The irradiation hardening at 350 °C is noticeably higher than at 150 °C and the work hardening remains positive at all doses up to 0.3 dpa (Figs. 2 and 4). At the irradiation temperature of 60 °C, on the other hand, the work hardening becomes negative and the ( $\alpha + \beta$ ) alloy suffers from plastic instability already at a dose level of 0.1 dpa.

At present there appears to be no clear and credible explanation for these observations. In order to obtain further insight into these issues, the details of the microstructure of these specimens irradiated under these conditions are being investigated and the results will be reported later.

#### 3.2. Fracture toughness properties

The effects of neutron irradiation to a dose level of 0.3 dpa at different temperatures on the fracture toughness behaviour of the  $(\alpha + \beta)$  and  $(\alpha)$  alloys are shown in Fig. 5. The initiation fracture toughness  $J_{\Omega}$  of unirradiated ( $\alpha + \beta$ ) alloy increases with increasing test temperature and has a maximum at about 250 °C [1,2]. After neutron irradiation to a dose level of 0.3 dpa the initiation fracture toughness decreases notably to a value of about  $25 \text{ kJ m}^{-2}$  at 20 °C and first seems to increase to about 83 kJ m<sup>-2</sup> with increasing temperature and then decreases to about 53 kJ m<sup>-2</sup> at 350 °C. On the other hand, the initiation fracture toughness of the unirradiated  $(\alpha)$ alloy is almost constant in the temperature range from 20 to 350 °C [1,2]. After neutron irradiation to a dose level of 0.3 dpa the initiation fracture toughness appears to decrease notably to value of about 20 kJ m<sup>-2</sup> at 20 °C and then increases to about 69 kJ m<sup>-2</sup> with increasing temperature up to 350 °C. It is noteworthy that crack growth is stable with a reasonable amount of tearing resistance in both the  $(\alpha + \beta)$  and  $(\alpha)$  alloys after neutron irradiation at 150 °C as can be seen in Fig. 5.



Fig. 5. Initiation fracture toughness values for Ti6Al4V and Ti5Al2.5Sn alloy in unirradiated conditions and neutron irradiated to a dose level of 0.3 dpa at different temperatures.

Neutron irradiation to a dose level of 0.3 dpa at 50 °C produced an essentially similar uniformly distributed defect cluster structure in the  $\alpha$ -phase both in the  $(\alpha + \beta)$  and  $(\alpha)$  alloys as shown in Fig. 6. In the  $(\alpha + \beta)$  allow neutron irradiation to a dose level of 0.3 dpa at 350 °C induced substantial uniform precipitation in the  $\alpha$ -phase and along cell boundaries with depleted zones around grain and cell boundaries. It is also noted that no precipitates were observed along grain boundaries. Precipitates were rich in vanadium and formed as thin platelets ( $\sim$ 5 nm thick and  $\sim$ 40 nm long) on (1010) and (1020) planes as illustrated in Fig. 6. In the ( $\alpha$ ) alloy mostly dislocation loops and only some Fe-rich precipitates were observed when irradiated to a dose level of 0.3 dpa at 350 °C.

#### 4. Summary and conclusions

In the unirradiated condition the tensile properties of the  $(\alpha + \beta)$  alloy showed a decrease in strength with increasing temperature although ductility and work hardening behaviour seemed to remain almost unaltered in the temperature range of 50–350 °C. The fracture toughness, on the other hand, showed a tendency to increase with increasing temperature reaching a maximum at about 200 °C in the temperature range of 20–350 °C. Almost similar tensile behaviour but at somewhat lower strength level and higher ductility has been reported earlier for the unirradiated ( $\alpha$ ) alloy. The fracture toughness of the unirradiated ( $\alpha$ ) alloy was almost constant in the temperature range of 50–350 °C [1].

Fig. 6. Neutron irradiation (0.3 dpa) of (a) Ti6Al4V alloy at 50 °C induced a high density of loops and 'black dots' and (b) at 350 °C a high density of thin plate-like precipitates (V-rich) in the  $\alpha$ -phase (c) Ti5Al2.5Sb alloy at 50 °C induced a high density of loops and 'black dots' and (d) at 350 °C a high density of rafts of loops and small precipitates (Fe-rich) in the  $\alpha$ -phase.

The increase in the yield strength of the  $(\alpha + \beta)$ alloy showed a tendency to saturate at irradiation doses higher than 0.3 dpa at 50 °C. On the other hand, a much steeper increase and no indication of saturation in yield strength at irradiation doses of up to 0.3 dpa were observed at 350 °C. After irradiation to a dose level of 0.3 dpa the  $(\alpha + \beta)$  alloy suffered from plastic instability at temperatures below about 150 °C. Fracture toughness, on the other hand, first decreased dramatically at the ambient temperature and then increased almost linearly with increasing temperature before decreasing again at 350 °C. Dose dependence of tensile properties to a dose level of 0.3 dpa and fracture toughness behaviour at a constant dose level of 0.3 dpa indicate that different hardening mechanisms operate at ambient temperature than at 350 °C.

It has been shown that the microstructure of the  $(\alpha + \beta)$  alloy after neutron irradiation to a dose level of 0.3 dpa at 60 °C consists of a high density of homogeneously distributed defect clusters in the  $\alpha$ -phase without any change in overall dislocation or phase structures. Irradiation at 350 °C to a dose

level of 0.3 dpa leads to a dense population of fine vanadium-rich plate-like precipitates in the  $\alpha$ -phase. The microstructural response of the ( $\alpha + \beta$ ) and ( $\alpha$ ) alloys to irradiation was different at elevated temperatures. In the ( $\alpha$ ) alloy the irradiation at 350 °C seemed to coarsen the defect structure resulting in dislocation loops and small iron-rich precipitates [2].

# Acknowledgements

The work reported here was partly funded by the European Fusion Technology Programme and was carried out by Associations Euratom – Tekes, Finland and Euratom – Risø, Denmark.

### References

- S. Tähtinen, P. Moilanen, B.N. Singh, Mechanical and fracture properties of neuron irradiated titanium alloys. Research Report BVAL62-001077, May 2001, VTT Manufacturing Technology.
- [2] S. Tähtinen, P. Moilanen, B.N. Singh, D.J. Edwards, J. Nucl. Mater. 307–311 (2002) 416.

- [3] P. Marmy, T. Leguey, I. Belianov, M. Victoria, J. Nucl. Mater. 283–287 (2000) 602.
- [4] Patrice Jacquet, Neutron irradiation of titanium alloy specimens. Restricted contract report SCK CEN-R- 3927, June 2004, SCK CEN Mol, Belgium.
- [5] Patrice Jacquet, Low dose neutron irradiation of titanium alloy samples. Restricted contract report SCK·CEN-R-3935, August 2004, SCK·CEN Mol, Belgium.