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# Tensile and fracture toughness properties of unirradiated and neutron irradiated titanium alloys

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#### Abstract

In the unirradiated condition the Ti6Al4V  $(\alpha + \beta)$  alloy has slightly higher tensile strength and noticeably lower ductility compared to that of the Ti5Al2.5Sn ( $\alpha$ ) alloy both at 50 and 350 °C. The fracture toughness behaviour of both alloys is similar at ambient temperature. At 350 °C, on the other hand, the fracture toughness of the ( $\alpha$ ) alloy is lower compared to that of the ( $\alpha + \beta$ ) alloy. Neutron irradiation at 50 °C to a dose level of 0.3 dpa caused hardening, plastic instability and a substantial reduction in fracture toughness of both alloys. Irradiation at 350 °C resulted in a substantial hardening and a significant decrease in the fracture toughness in the ( $\alpha + \beta$ ) alloy due to irradiation induced precipitation whereas only minor changes in the tensile and fracture toughness behaviour were observed in the ( $\alpha$ ) alloy. The tensile and fracture toughness properties of the ( $\alpha + \beta$ ) alloy are more strongly affected by neutron irradiation compared to that of the ( $\alpha$ ) alloy.

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# 1. Introduction

Titanium alloys have an attractive combination of thermophysical, mechanical and radioactive decay properties and therefore have been identified as possible structural materials for different components in fusion reactors [1,2]. Two widely used and industrially available alloys, Ti5Al2.5Sn ( $\alpha$ ) and Ti6Al4V ( $\alpha + \beta$ ), are being considered as candidate materials for flexible mechanical connectors between the blanket modules and the vacuum vessel of the ITER. Since not much is known about the effect of neutron irradiation on phase stability, defect accumulation and mechanical properties of these alloys in ITER relevant conditions [2,3], screening investigations were initiated to determine the response of these alloys to fission neutron irradiation at 50 and 350 °C.

The effects of neutron irradiation on microstructure, tensile and fracture toughness properties are described and discussed in the present paper.

### 2. Experimental details

Two types of high strength titanium alloys i.e. Ti5Al2.5Sn (produced by Howmet Mill) and Ti6Al4V (produced by Timet) were studied. The Ti5Al2.5Sn alloy was heat treated at 815 °C for 1 h and the Ti6Al4V alloy was mill annealed at 730 °C for 1.5 h followed by air cooling in both cases [4].

Tensile and fracture toughness specimens were irradiated with fission neutrons in the DR-3 reactor at Risø National Laboratory at 50 and 350 °C to a neutron fluence of  $1.5 \times 10^{24}$  n/m<sup>2</sup> (E > 1 MeV) corresponding to a displacement dose of about 0.3 dpa (NRT). Irradiations were carried out with a neutron flux of  $2.5 \times 10^{17}$ n/m<sup>2</sup>s corresponding to a damage rate of  $5 \times 10^{-8}$  dpa (NRT)/s. Irradiations were performed in an atmosphere of helium or a mixture of helium and argon. Irradiation at 350 °C was carried out in a temperature

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controlled rig where the irradiation temperature was monitored, controlled and recorded continuously throughout the whole irradiation period.

Both unirradiated and irradiated tensile specimens of Ti5Al2.5Sn and Ti6Al4V alloys were tested at a strain rate of  $1.3 \times 10^{-3}$  s<sup>-1</sup>. The thickness and the gauge length of the tensile specimens were 0.3 and 20 mm, respectively. Tensile tests at 350 °C were carried out in a vacuum of  $10^{-5}$  torr. Single edge notched bend SEN(B) fracture toughness specimens of dimensions  $3 \times 4 \times 27$ and  $10 \times 10 \times 55$  mm were machined from the rods corresponding to L-C and R-C orientations in Ti5Al2.5Sn and Ti6Al4V alloys, respectively. The notch and the 20% side grooves were machined by applying electric wire discharge machining. The applied prefatigued crack length to specimen width ratio was about 0.5. Fracture resistance curves were determined using displacement controlled three point bend tests with a constant displacement rate of  $2.5 \times 10^{-4}$  mm s<sup>-1</sup>. Fracture resistance testing at 350 °C was carried out in a silicon oil bath. Load, displacement and crack length measured using the dc-potential drop method, were recorded during the testing, and the fracture resistance curves were determined following the ASTM standard test method E 1737-96.

#### 3. Results

#### 3.1. Microstructure

The microstructure of both alloys showed equiaxed grain structure with an average grain size of about 20  $\mu$ m. The Ti5Al2.5Sn ( $\alpha$ ) alloy exhibited elongated and deformed primary recrystallised  $\alpha$ -grains with some retained  $\beta$ -phase mostly along the  $\alpha$ -grain boundaries. Neutron irradiation at 50 °C produced a high density of uniformly distributed small defect clusters. Irradiation at 350 °C resulted in coarse dislocation loops and thin plate-like precipitates, Fig. 1 which exhibited increased iron content in EDX analysis. Additionally, the depleted of defect clusters were observed to have formed along the grain boundaries.

The Ti6Al4V ( $\alpha + \beta$ ) alloy had an uniform microstructure with an equiaxed  $\alpha$ -phase, a transformed acicular  $\alpha$ -phase and a  $\beta$ -phase. The primary  $\alpha$ -phase contained clear cell structure with low angle boundaries and dislocation walls whereas the beta-phase consisted of martensitic substructure. Neutron irradiation at 50 °C produced uniformly distributed defect clusters in the  $\alpha$ phase of the  $(\alpha + \beta)$  alloy as in the case of the  $(\alpha)$  alloy. The irradiation at 350 °C induced a significant amount of uniform precipitation in the  $\alpha$ -phase. Precipitates were rich in vanadium and formed as thin platelets ( $\sim 5$ nm thick and  $\sim 40$  nm long) on  $(10\overline{1}0)$  and  $(10\overline{2}0)$ planes as illustrated in Fig. 1.

Fig. 1. Microstructure of (a) Ti5Al2.5Sn ( $\alpha$ ) alloy irradiated at 50 °C showing uniformly distributed defect clusters with beam direction  $[\bar{1}2\bar{1}3]$ , (b) Ti5Al2.5Sn ( $\alpha$ ) alloy irradiated at 350 °C showing coarse dislocation loops and iron rich precipitates and (c) Ti6Al4V ( $\alpha + \beta$ ) alloy irradiated at 350 °C showing vanadium rich plate-like precipitates.

## 3.2. Tensile tests

The tensile curves of the Ti5Al2.5Sn ( $\alpha$ ) alloy shown in Fig. 2(a) reveal that relative to the strength at 50 °C, the unirradiated alloy loses a large fraction of its strength when tested at 350 °C. Work hardening remains minimal for both test temperatures with a uniform elongation of  $\sim 15\%$  at 50 °C and  $\sim 18\%$  at 350 °C. The failure mode is ductile at both test temperatures in the unirradiated condition. Irradiation at 50 °C produced an upper yield point approximately 130 MPa higher than the sharp yield point observed in the unirradiated specimens tested at 50 °C. The upper yield point is followed by a small but clear yield drop. Additionally, the ultimate tensile strength has increased, but it is near



(1120)

(1010)

0002



Fig. 2. Stress–strain curves for (a) Ti5Al2.5Sn ( $\alpha$ ) alloy and (b) Ti6Al4V ( $\alpha + \beta$ ) alloy tested at 50 and 350 °C in the unirradiated and irradiated conditions.

the same level as the upper yield stress, showing that the irradiated samples are unable to work harden to any significant extent. The samples irradiated and tested at 350 °C exhibit the same yield plateau as exhibited in the unirradiated samples, however, there is a modest increase in both the yield and ultimate strength accompanied by some reduction in elongation.

Fig. 2(b) shows stress–strain curves for the Ti6Al4V  $(\alpha + \beta)$  alloy tested at 50 and 350 °C both in the unirradiated and irradiated conditions. In the unirradiated condition, the  $(\alpha + \beta)$  alloy is slightly stronger, particularly at 350 °C, than the  $(\alpha)$  alloy. However, even in the unirradiated condition the  $(\alpha + \beta)$  alloy is less ductile than the  $(\alpha)$  alloy both at 50 and 350 °C.

The irradiation at 50 °C causes an increase in the yield stress as well as in the ultimate tensile strength of  $(\alpha + \beta)$  alloy which is almost 100 MPa higher than that observed in the  $(\alpha)$  alloy. Unlike the  $(\alpha)$  alloy, the

 $(\alpha + \beta)$  alloy irradiated at 50 °C does not exhibit an yield drop. However, it shows very low uniform elongation and seems to suffer from plastic instability. The irradiation at 350 °C on the other hand causes a very substantial amount of hardening in the  $(\alpha + \beta)$  alloy with only a modest decrease in the uniform elongation and practically no decrease in its ability to work harden. The hardening observed in the  $(\alpha + \beta)$  alloy at 350 °C appears to be related to the radiation-induced precipitation in this alloy.

### 3.3. Fracture resistance tests

The effect of temperature and neutron irradiation on the fracture resistance behaviour of Ti5Al2.5Sn ( $\alpha$ ) and Ti6Al4V ( $\alpha + \beta$ ) alloys are shown in Fig. 3. In unirradiated condition the initiation fracture toughness of both the ( $\alpha$ ) and ( $\alpha + \beta$ ) alloys were similar at ambient temperature whereas the fracture toughness of the ( $\alpha + \beta$ ) alloy was clearly higher compared to that of the



Fig. 3. Fracture resistance curves of (a) Ti5Al2.5Sn ( $\alpha$ ) alloy and (b) Ti6Al4V ( $\alpha + \beta$ ) alloy at different test temperatures in unirradiated and neutron irradiated (0.3 dpa) conditions.

( $\alpha$ ) alloy at 350 °C. A significant reduction in initiation fracture toughness and almost complete loss of tearing resistance were observed in both alloys after neutron irradiation at 50 °C when compared to those in the unirradiated condition. After neutron irradiation at ambient temperature the initiation fracture toughness  $(J_{\Omega})$  of the ( $\alpha$ ) and ( $\alpha + \beta$ ) allows decreased from about 66 to 21 kJ m<sup>-2</sup> and from about 61 to 25 kJ m<sup>-2</sup>, respectively. It is interesting to note that although tearing resistance at ambient temperature was very low, the fracture surfaces exhibited ductile dimple fracture appearance in both the  $(\alpha)$  and  $(\alpha + \beta)$  alloys. After neutron irradiation at 350 °C the fracture resistance curves were similar for the  $(\alpha)$  alloy and a clear reduction was observed in the  $(\alpha + \beta)$  alloy when compared to unirradiated condition. The initiation fracture toughness  $(J_{\Omega})$  of the  $(\alpha)$  and  $(\alpha + \beta)$  alloys decreased from 74 to 69 kJ m<sup>-2</sup> and from 119 to 53 kJ m<sup>-2</sup>, respectively.

## 4. Discussion

The microstructural response of the Ti5Al2.5Sn ( $\alpha$ ) and Ti6Al4V ( $\alpha + \beta$ ) alloys to neutron irradiation to a dose level of 0.3 dpa at 50 °C is reflected in the build up of a high density of uniformly distributed defect clusters in the  $\alpha$ -phase without any significant changes in the overall dislocation or phase structures. The microstructural response of the ( $\alpha$ ) and ( $\alpha + \beta$ ) alloys to irradiation were different at elevated temperatures. In the  $(\alpha)$  allow the irradiation at 350 °C resulted in coarse defect structure with dislocation loops and iron rich precipitates. Formation of dislocation loops and fine  $\beta$ -precipitates in  $\alpha$ -phase have been reported in the Ti6Al4V  $(\alpha + \beta)$  alloy after neutron irradiation to a dose level of 2.1 and 32 dpa at 450 °C [5,6]. Extensive void formation and coarse, blocky  $\beta$ -precipitates were observed in the same alloy after irradiation to a dose level of 32 dpa at 550 °C [6]. The irradiation induced β-precipitation was observed in the Ti6Al4V ( $\alpha + \beta$ ) alloy irrespective of heat treatment condition i.e. mill annealed, duplex annealed or  $\beta$ -annealed. The causes of the precipitation was determined to be radiation-enhanced diffusion and radiation-induced segregation of the undersized β-stabilising element, vanadium, to the defect clusters. These results are consistent with those reported in [7] and the present observation of dense precipitation of fine vanadium rich plate-like precipitates in mill annealed Ti6Al4V ( $\alpha + \beta$ ) alloy irradiated to a dose level of 0.3 dpa at 350 °C.

Tensile and fracture toughness properties of the  $(\alpha)$  and  $(\alpha + \beta)$  alloys are consistent with the microstructural observations. Neutron irradiation at 50 °C resulted in hardening, plastic instability, decrease in fracture toughness and tearing resistance in both alloys. At 350

°C, on the other hand, the tensile and fracture toughness response of the ( $\alpha$ ) and ( $\alpha + \beta$ ) alloys to irradiation is different. In the ( $\alpha$ ) alloy the tensile and fracture toughness properties were only modestly affected by neutron irradiation whereas the ( $\alpha + \beta$ ) alloy showed significant hardening and reduction in fracture toughness due to radiation induced precipitation.

## 5. Conclusions

The tensile and fracture toughness properties of the  $(\alpha + \beta)$  alloy are more strongly affected by neutron irradiation compared to those of the  $(\alpha)$  alloy.

In unirradiated condition the Ti6Al4V ( $\alpha + \beta$ ) alloy has a slightly higher strength and noticeably lower ductility compared to that of the Ti5Al2.5Sn ( $\alpha$ ) alloy both at 50 and 350 °C. The fracture toughness behaviour of both ( $\alpha$ ) and ( $\alpha + \beta$ ) alloys are similar at ambient temperature. At 350 °C the fracture toughness of the ( $\alpha$ ) alloy is lower compared to those the ( $\alpha + \beta$ ) alloy.

Neutron irradiation at 50 °C to a dose level of 0.3 dpa caused hardening, plastic instability and reduction in fracture toughness in both ( $\alpha$ ) and ( $\alpha + \beta$ ) alloys. Irradiation at 350 °C resulted in substantial hardening and significant decrease in fracture toughness in the ( $\alpha + \beta$ ) alloy whereas only minor changes in tensile and fracture toughness behaviour in the ( $\alpha$ ) alloy were observed.

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