УДК 621.039

FRACTURE TOUGHNESS AND TENSILE PROPERTIES OF TWO TITANIUM ALLOYS BEFORE AND AFTER PROTON AND NEUTRON IRRADIATIONS AT 150°C

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Two titanium alloys, the α+β Ti6Al4V and the α phase Ti5Al2.5Sn alloy, have been irradiated at 150°C with neutrons, in the reactor of the Atomic Energy Research Institute, in Budapest and with 590 MeV protons in the PIREX facility of the Paul Scherrer Institute, in Switzerland, to doses of the order of 0.15 dpa. The proton irradiation induced hydrogen as a product from spallation reactions. Some of the neutron irradiated specimens were previously loaded with 150 wppm hydrogen. The tensile and fracture toughness properties have been analyzed as a function of the measured hydrogen content. At low levels, hydrogen is not influencing significantly the tensile properties. The effect of irradiation on ductility and strength is more pronounced in the α+β Ti6Al4V, due to radiation induced phase instabilities. At a test temperature of 150°C, hydrogen levels up to 150 wppm have moderate effect on fracture toughness, in the unirradiated condition. For the irradiated specimens in both alloys, increasing the hydrogen content decreases the fracture toughness.The fracture toughness after irradiation is strongly reduced at room temperature, in both alloys.

1. INTRODUCTION

The ITER first wall modules are attached to the vacuum vessel by a set of four radial flexible cartridges. Due to their excellent elasticity and strength properties, titanium alloys have been proposed as materials for fabricating the supports. Among a set of internationally proposed alloys, two alloys widely used in the industry, the α+β Ti6Al4V and the α-phase Ti5Al2.5Sn have been chosen as main candidates for the ITER application. In previous works, the tensile, fatigue and fracture toughness properties have been studied before and after irradiation (see references [1-3]). The influence of hydrogen loading up to 400 wppm on the fracture toughness properties has also been reported. The uptake of hydrogen in large quantities induces structural changes in the alloys but does not degrade much the resistance to fracture at high temperature. At room temperature a clear degradation of the unirradiated frac ture resistance takes place. The fracture toughness properties are strongly affected in both alloys, after an irradiation with neutrons at 60 and 350°C, specially when testing at room temperature [2-3]. Since the mean operating temperature of the cartridges is around 150°C, a new set of experiments was necessary to check the effective degradation during ITER operation.

It was been decided to use two types of irradiating particles, protons and neutrons, and to center the attention onto the effects of hydrogen, since the previous work had shown that the embrittlement is mainly controlled by the hydrogen content. The irradiation with high energy protons is generating hydrogen through spallation reactions, with possible detrimental effects on the properties. Because the neutrons do not generate any hydrogen, some specimens have been previously loaded with 150 wppm hydrogen. The hydrogen content has been accurately measured. The effects of proton and neutron irradiation on the tensile and fracture toughness properties are described and discussed taking particular attention to the role of hydrogen.

2. EXPERIMENTAL DETAILS

Materials and as received microstructure: After hot forming to a diameter of 31.75 mm, the Ti5Al2.4Sn alloy has been annealed 1hr at 815°C and then air cooled. The structure consists of equiaxed grains of 20 μm. Some larger grains of about 40 μm or more also exist. Usually the larger grains contain smaller ones. A precipitation of an iron riched phased has been detected in the grains, both by optical and transmission microscopy. The TiFe precipitates have a size around 100 nm and are located at the grain boundaries and inside the grains. They are generated by the high iron concentration (0.36 wt%) in the alloy.

After hot forming to a stock diameter of 150mm in the α+β field, the Ti6Al4V alloy has been annealed for 1.5 hr at 730 °C and then air cooled. The structure consists of equiaxed α grains of about 20 μm, containing secondary α zones surrounded by β intergranular phase. The fraction volume of the β phase is around 13 %. Due to its different composition, the β phase is quite visible at the boundaries and appears as intergranular bands of 0.05 to 1 μm width. Some small quantities of residual martensite can be observed in the larger β grains

No hydrides are present in the *as received* microstructure of both alloys [3-5].The chemical specification is given in table 1.

Specimens: The fracture toughness specimen is a mini-charpy DIN 50115 KLST with a size of 3x4x27 mm. It is labeled **C**, for easy differentiation. The pre/crack of 1mm length has been grown at the notch before the irradiation (total crack depth 2 mm), at high frequency, in a fatigue pre-test. The specimens for the neutron irradiation have been cracked after the irradiation, before the test, at low frequency. The tensile specimen for the proton irradiation is a PIREX flat specimen with a gauge size of 4x0.34x5.5 mm [5]. It is labeled **T**. The tensile specimen for the neutron irradiation is a DIN 50125 cylindrical specimen with a geometrical gauge length of 18 mm and 3 mm diameter. It is labeled **D**.

Table 1

Chemical compositions: [wt %]

	Al	C	Fe	Sn
Ti5Al2.5Sn	5.0	0.17	0.36	2.4
Ti6Al4V	6.08	0.0056	0.1399	-
H_2	N_2	O_2	V	Others
0.0036	0.010	0.179	-	
<0.0060	0.0065	0.176	3.95	<0.4

Proton Irradiation : The tensile and charpy specimens have been irradiated in the PIREX facility [6] where the heat deposited by the proton beam is removed by pressurized helium. The tensile flat specimens have been irradiated with a beam of 3 mm width and 4 mm height. The wobbler amplitude was ±2.5 mm, in order to distribute the protons evenly. The mini Charpy specimen was equipped with a central thermocouple placed into an hole that ended very close, at 1.5 mm from the pre/crack. A beam with a width of 6mm and a height of 3mm has been centered onto the specimen. It was not wobbled because only the center of the specimen had to be irradiated. In the cooling flow of pressurized helium, at 115 Nm^3/hr ,30 bars and 40°C, a proton beam intensity of 7µa has been adjusted in order to get a specimen irradiation temperature of 150°C. A dosimetry evaluation based on Sc46, which production cross section σ(Ti(p,x)) is estimated to 27.2 mbarn [7], has given the results listed in table 2. The specimens irradiated with the high energy protons are all in the *as received* condition. The materials already contain some level of hydrogen, as indicated in table 1.

Table 2

Fluence and displacements per atom after the irradiations in PIREX

SPECIMEN	Fluence p/cm^2	Dose dpa
I25T12	5.09E+19	0.1342
I25T14	1.63E+19	0.0428
I25C55	6.18E+19	0.1628
I25C54	6.16E+19	0.1623
I14T27	2.32E+19	0.0611
I14T28	6.4E+19	0.1685
I14C53	4.96E+19	0.1308
I14C54	4.99E+19	0.1315

The high energy proton irradiation increases the hydrogen content by spallation reactions [8].Therefore the hydrogen concentration has been determined carefully using a method based on gas mass spectrometry [9]. The results from five specimens are indicated in table 3.

Table 3

Measured hydrogen in proton irradiated samples

Sample	Net 1H wppm
Unirradiated:	
N14C62	47.9
N25C77	20.4
Irradiated:	
I14C53	63.6
I14C54	60.7
I25C54	62.4
I25C55	53.4
I25T12	47.2

It is also interesting to note that the predicted hydrogen production in titanium is around 650 appm/dpa [8], which corresponds to about 14 wppm H/dpa. The irradiated values of table 3 are for about 0.2 dpa. Therefore the observed rates of hydrogen deposition in alloy 14 and 25 were around 70 wppm/dpa and 150 wppm/dpa.- much higher than the prediction. The reason for this significantly higher deposition is currently not known.

Neutron Irradiation: The neutron irradiation has been performed at the Atomic Energy Research Institute in Budapest, in a VVRSZM research reactor of 10MW power. The mean flux at nominal power is $3.5x10^{13}$ n/cm^2 s (E>1MeV). The neutron spectrum of the reactor is closed to a fission spectrum. The specimens have been irradiated using the rig BAGIRA, in which a flow of He/N2 gas mixture is used for temperature control. The temperature has been monitored with five thermocouples and was kept between 140 and 150 °C during all the experiment. A dosimetry analysis based on the radioisotope Sc46, indicates that the mean neutron fluence is $1.08x10^{20}$ n/cm^2. Assuming a dpa cross section in the AEKI reactor of 1420 barn (n E>1MeV), the dose is estimated to 0.154 dpa.

The specimens I14C46 and 69, I25C46 and 49, I14D40 and I25D45 are all in the *as received* condition. Two specimens loaded with 150 wppm hydrogen have also been irradiated (I14C38 and I25C39), together with two annealed specimens (I14C6 and I25C30). The annealed specimens have an hydrogen level close to zero.

3. RESULTS AND DISCUSSION

3.1. TENSILE TESTS

All tests have been conducted at the same strain rate, $2.5x10^{-4}$ s^{-1}. To take into account the effect of specimen size, unirradiated tests have been performed with both specimen geometries. The total and uniform elongation values are similar in both geometries, whereas the reduction of area is clearly higher in the DIN cylindrical specimens. The yield stress and the ultimate stress are slightly higher in the DIN specimens. The Streckgrenze effect (sharp yield point) was visible in both geometries and slightly sharper in the cylindrical geometry.

Figure 1 shows the effect of the proton irradiation. The α alloy is less affected by the irradiation as compared to the α+β alloy. The hardening $\Delta\sigma_{irr}$ at 150°C and 0.06 dpa is only 14 MPa for the α alloy as compared to 150 MPa for the α+β alloy.

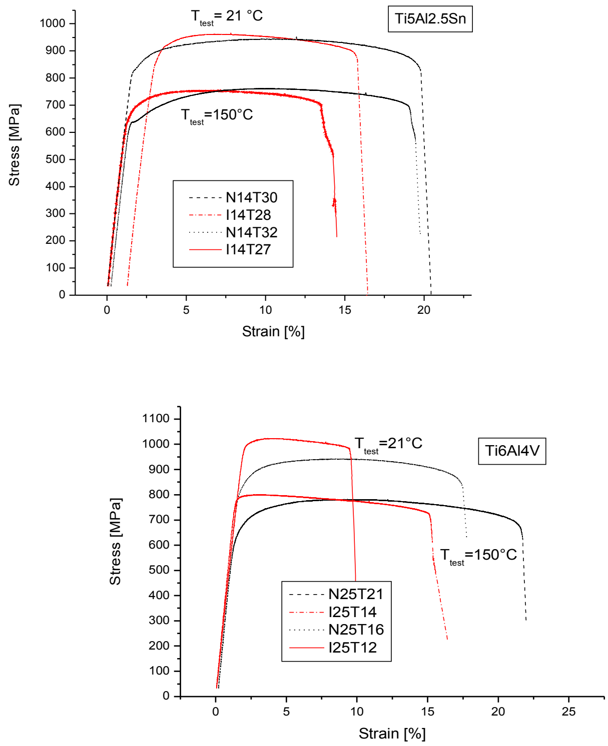

Fig. 1. Tensile curves of Ti6Al4V and Ti5Al2.5Sn before and after irradiation with protons at 150°C. I25T12, has a dose of 0.13 dpa, I25T14 has a dose of 0.04 dpa, I14T28 has a dose of 0.17 dpa and I14T27 has a dose of 0.06 dpa

The hardening $\Delta\sigma_{irr}$ at T_{test}=21°C and 0.13 dpa is clearly larger for the α+β alloy. Furthermore the Ti6Al4V tensile curves have an unstable shape with a low uniform elongation after irradiation. Although less strong in amplitude as compared to what was measured after an irradiation with neutrons at 350°C, the response observed is similar. It is probable that some phase instabilities already occur after the irradiation at 150°C, after a dose of only 0.04 dpa.

Figure 2 presents the effects after the irradiation with neutrons at the same temperature of 150°C. It is interesting to note that for the alpha alloy, the sharp yield point in the unirradiated case is reinforced after the irradiation. Nevertheless the material is still able to deform homogenously and reaches a uniform elongation of 7.5%. In the Ti6Al4V, the situation is worst as indicated by the unstable deformation curve of I25D45. The uniform elongation is very low after irradiation, considering that most of the deformation is elastic at the stability limit.

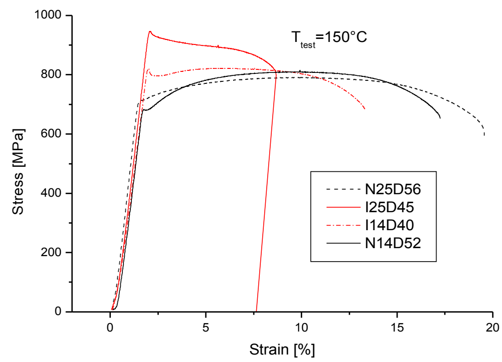

Fig. 2. Tensile curves of Ti6Al4V (N25D56, I25D45) and Ti5Al2.5Sn (N14D52,I14D40), irradiated with neutrons at 150°C to 0.154 dpa (T_{test} =150°C)

The irradiation dose of the proton irradiated specimens is significantly less than the dose reached in the neutron irradiated specimens. Comparing figures 1 and 2, the relative hardening after irradiation appears clearly stronger in the case of the Ti6Al4V alloy, after both the proton and the neutron irradiation. Compared on a dpa base, the neutrons seem more damage effective than the protons, in terms of ductility loss. The stronger irradiation hardening in the α+β alloy is probably due to the formation of irradiation induced vanadium precipitates, as shown in previous works in the same materials irradiated at 350°C [1, 2].The radiation induced precipitation is temperature assisted as it does not occur after a neutron irradiation at 50°C [2].

The hydrogen intake after proton irradiation is of the order of 20 to 40 wppm, as shown in table 3. Apparently this increase in the hydrogen level does not modify the tensile properties.

Dynamic strain ageing has not been observed during the experiments except in the case of N14D50 and N25D56, where it was qualified as very weak. Sharp yield points have been observed at 150°C only, in both alloys before irradiation and only in the α alloy after irradiation. Nevertheless it is possible that the flow instability appearing in the Ti6Al4V is hiding the sharp yield point.

3.2. FRACTURE TOUGHNESS TESTS

The fracture tests have been accomplished according to the ASTM norm E813, in a three point bend fixture. All specimens have been loaded with a deformation rate of 8.33x 10^{-3} mm/s. The measurement of crack extension was done using the compliance method which measures the apparent elasticity of the specimen. The effective crack length is then calculated using the compliance transfer relation of Jablonski [10].

For the unirradiated material, the values of J_Q are very similar in both alloys. The value at RT is around 60 kJ/m^2. This is in accordance with previous measurements [11]. The fracture toughness increases at 150°C , to reach a value around 100 kJ/m^2. The irradiated Ti5Al2.5Sn material is only slightly affected by the irradiation when tested at 150°C, whereas the Ti6Al4V alloy shows a clear reduction in the J_Q value. When tested at RT both irradiated alloys show a large reduction of J_Q. The data are easy to understand, if presented as a function of their hydrogen content, as shown in figures 3 and 4. As explained previously, hydrogen is already present in the material in the *as received* condition. It has also been loaded in a vacuum furnace or removed after annealing in vacuum. The proton irradiation introduces some hydrogen (see table 2) and the neutron irradiation is not changing the initial content.

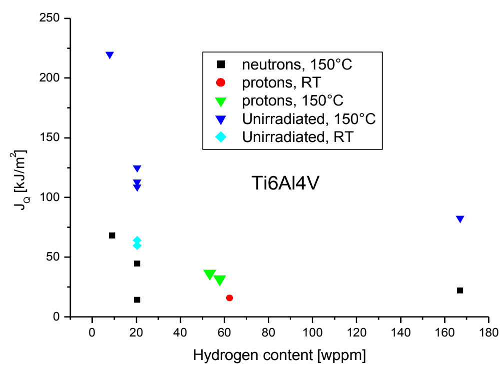

Fig. 3. Fracture toughness of Ti6Al4V alloy, unirradiated, irradiated at 150°C, with protons to 0.163 dpa and neutrons to 0.154 dpa. The data are plotted as a function of their hydrogen level

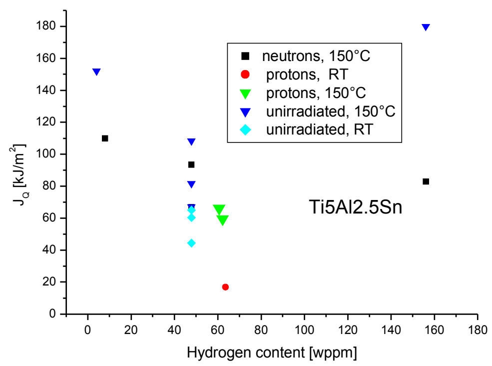

Fig. 4. Fracture toughness of Ti5Al2.5Sn alloy, unirradiated, irradiated at 150°C, with protons to 0.131 dpa and neutrons to 0.154 dpa. The data are plotted as a function of their hydrogen level

For the case of the α+β alloy shown in figure 3, we see that the fracture toughness of the unirradiated material is improved, when increasing the temperature from RT to 150°C. It is also improved if the hydrogen content is decreased. Nevertheless the dependence on hydrogen content at 150°C is not strong and for the highest level of the diagram (170 wppm H), J_Q remain of the order of 100 kJ/m^2. This is more than the value of the *as received* material at room temperature. At RT., the irradiated data show more dispersion than the unirradiated data, an indication for radiation embrittlement. The proton irradiated data fit quite well the trend of the neutron irradiated data. Since the fracture toughness of the 150 wppm H loaded material is clearly lower after than before irradiation, we can argue that the damage from the irradiation and from the hydrogen are additive.

The data for the alpha alloy are presented in figure 4. They show a similar behavior. At 150°C, as the hydrogen content increases, the fracture toughness decreases, whereas in the unirradiated material a reverse response is observed. This behavior is in fact not a consequence of the hydrogen impurity atoms but rather a consequence of the microstructural changes introduced by the hydrogen charging (see report [11]). Despite the toughness improvement observed at 150°C and 150 wppm H, after neutron irradiation a drastic decrease of toughness is observed. Nevertheless the value is approximately four times higher, as compared with the Ti6Al4V case. The proton irradiated data are slightly lower than the neutron irradiated data, when plotted against their hydrogen content (see figure 4).

The lowest fracture toughness value is measured at

ВОПРОСЫ АТОМНОЙ НАУКИ И ТЕХНИКИ. 2005. № 3.
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RT, after proton irradiation.

Comparing Fig.3 and 4, it seems that the Ti6Al4V data show more dependence on hydrogen level, as compared to the Ti5Al2.5Sn data. It is possible that more hydrides are present in the α+β alloy. At higher temperatures, the hydrides are expected to be dissolved [12].

In a previous work [3], the same alloys had been irradiated near room temperature and at 350°C. Using these data, figure 5 shows the behavior of J_Q as a function of the irradiation temperature, for a constant level of hydrogen. Figure 5,a shows the dependence when the alloys are hydrogen free, after a vacuum annealing of 5 hours at 750°C. Figure 5,b shows the behavior of the alloys when they are loaded with 150 wppm hydrogen. In the case of the hydrogen free alloys, increasing the irradiation temperature induces embrittlement, whereas for the hydrogen loaded alloys, increasing the irradiation temperature improves the fracture toughness. This may be the result of a structural stabilizing effect of hydrogen. This unexpected but positive effect of hydrogen is shown here in both alloys, for relatively low doses. It is unclear whether the effect will subsist at higher doses.

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The fractographs of the unirradiated and irradiated specimens have been analyzed under the scanning electron microscope. The general aspect of all fractographs is ductile. Relatively large effects are measured in the mechanical test but the difference does not show up in the appearance of the fractographs. This is shown in figure 6 which compares two extreme cases, I25C54 irradiated at 150°C and showing a low J_Q value and N25C95b, unirradiated and having a relatively high J_Q value.

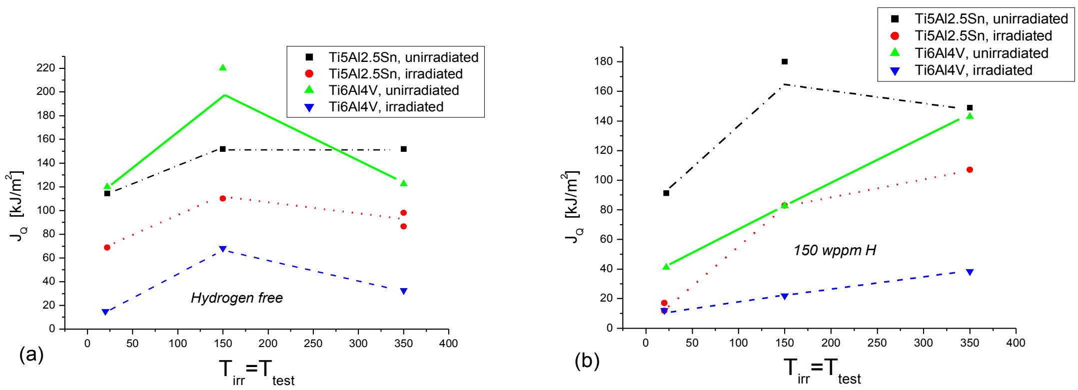

Fig. 5. Fracture toughness of Ti5Al2.5Sn and Ti6Al4V alloys as a function of the irradiation temperature. The crack initiation fracture toughness values are shown in (a) for the hydrogen free condition and in (b) for an hydrogen level of 150 wppm. Note the reduced fracture toughness values after irradiation at elevated temperatures, for the hydrogen free condition

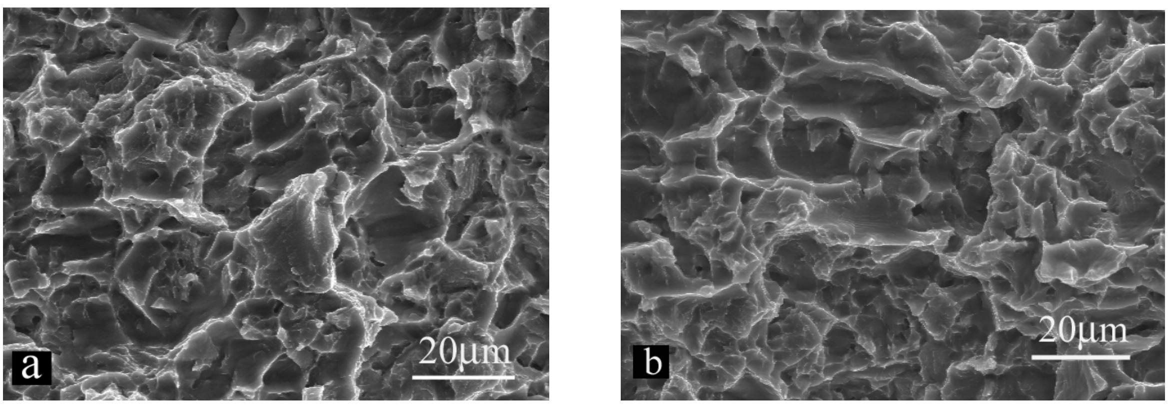

Fig. 6. Fractographs of the irradiated specimen I25C54 (a) with a J_Q value of 16 kJ/m^2 and of the unirradiated specimen N25C95b (b) with a J_Q value of 64 kJ/m^2, both tested at RT and showing a ductile appearance

Both fractographs show a fracture surface with dimples, indicating large local deformations. Careful observation, nevertheless, reveals a higher density of microcracks in the irradiated material. This difference is more apparent in the irradiated material tested at room temperature but also applies to the unirradiated material when comparing material tested at low and high temperature.

4. CONCLUSIONS

Tensile and fracture toughness specimens of Ti6Al4V and Ti5Al2.5Sn have been irradiated with high energy protons to a dose between 0.04 and 0.17 dpa, at 150°C. Tensile and fracture toughness specimens of Ti6Al4V and Ti5Al2.5Sn, some of them loaded with hydrogen, have been irradiated with neutrons at 150°C, to 0.15dpa.

The tensile tests seem to indicate that the Ti6Al4V is more affected by the 150°C irradiation in terms of hardening and reduction of ductility, as compared with the Ti5Al2.5Sn alloy. The irradiation at 150°C seems to induce vanadium rich precipitates in the α+β alloy, as it was shown earlier after an irradiation at 350°C.

The crack initiation fracture toughness values are strongly reduced after irradiation, especially when tested at room temperature. At 150°C, the reduction in toughness is more pronounced in the Ti6Al4V alloy. Nevertheless the fractographs indicate a ductile fracture for all conditions in both materials.

The fracture toughness data can be well understood, if represented as a function of their hydrogen content. Hydrogen levels up to 150 wppm have moderate effect on toughness, at a test temperature of 150°C. For the irradiated specimens in both alloys, increasing the hydrogen content decreases the fracture toughness.

The neutron and proton fracture toughness data show a consistent behaviour, when compared in relation to their associated hydrogen content.

Acknowledgements:

The Paul Scherrer Institute at Villigen, PSI, is sincerely thanked for its logistical and technical support throughout this project. The contributions of R. Brьtsch for the SEM microscopy, F. Hegedues for the dosimetry and B.Oliver for the gas spectrometry are greatly appreciated.

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ВЯЗКОСТЬ РАЗРУШЕНИЯ И ПРОЧНОСТЬ НА РАСТЯЖЕНИЕ ДВУХ ТИТАНОВЫХ СПЛАВОВ ДО И ПОСЛЕ ОБЛУЧЕНИЯ ПРОТОНАМИ И НЕЙТРОНАМИ ПРИ 150^0 С

П. Марми

Два титановых сплава α+β Ti6A14V и α-фаза Ti5A12.5Sn были облучены при 150˚С нейтронами в реакторе исследовательского Института ядерной энергии в Будапеште и 590 МэВ протонами на установке ПИРЕКС в Институте Поль-Шеррер, в Швейцарии до доз порядка 0.15 сна. Облучение протонами обусловливает образование водорода как продукта реакций расщепления. Несколько облученных нейтронами образцов были предварительно нагружены 150 wppm водорода. Прочность на растяжение и вязкость разрушения были проанализированы как функция содержания водорода. При низких уровнях водород не оказывает существенного влияния на прочность на растяжение. Влияние облучения на пластичность и прочность более выражено в α+β Ti6A14V вследствие радиационно-обусловленной фазовой нестабильности. При температуре испытания в 150˚С водород в количестве 150 wppm оказывает умеренное влияние на вязкость разрушения в необлученном состоянии. Для облученных образцов в двух сталях увеличение содержания водорода понижает вязкость разрушения. Вязкость разрушения после облучения сильно уменьшается при комнатной температуре в обоих сплавах.

В`ЯЗКІСТЬ РУЙНУВАННЯ ТА МІЦНІСТЬ НА РОЗТЯГ ДВОХ ТИТАНОВИХ СПЛАВІВ ДО ТА ПІСЛЯ ОПРОМІНЕННЯ ПРОТОНАМИ ТА НЕЙТРОНАМИ ПРИ 150ºC

П. Мармі

Два титанові сплави α+β Ti6A14V та α-фаза Ti5A12.5Sn були опромінені при 150˚С нейтронами в реакторі дослідницького Інституту ядерної енергії в Будапешті та 590 МеВ протонами на установці ПІРЕКС в Швейцарії до доз порядку 0.15 зна. Опромінення протонами зумовлює утворення водню як продукта розщеплення. Декілька опромінених нейтронами зразків були попередньо навантажені 150 wppm водню. Міцність на розтяг та в`язкість руйнування були проаналізовані як функція вмісту водню. При низьких рівнях водень не має суттєвого впливу на міцність на розтяг. Вплив опромінення на пластичність та на міцність більш виразний у сплаві α+β Ti6A14V внаслідок радіаційно-обумовленої фазової нестабільності. При температурі випробування в 150˚С водень у кількості 150wppm має помірний вплив на в`язкість руйнування в неопроміненному стані. Для опромінених зразків в двох сталях зростання вмісту водню знижує в`язкість руйнування. В`язкість руйнування сильно зменшується при кімнатній температурі в обох сплавах.

ВОПРОСЫ АТОМНОЙ НАУКИ И ТЕХНИКИ. 2005. № 3.
Серия: Физика радиационных повреждений и радиационное материаловедение (86), с. 18-23.