Swift heavy ion irradiation damage in Ti-6Al-4V and Ti-6Al-4V-1B: Study of the microstructure and mechanical properties

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ABSTRACT

The $\alpha+\beta$ alloy Ti-6Al-4V(wt.%) and the boron-modified Ti-6Al-4V-1B(wt.%), due to their low activation, corrosion resistance, good mechanical properties, and their commercial availability, were chosen as candidate materials for the beam dump for the Facility for Rare Isotope Beams (FRIB) at Michigan State University: a new generation accelerator with high power heavy ion beams. Through this study our goal is to establish the first irradiation data of the recently developed Ti-6Al-4V-1B (wt.%) alloy and investigate the changes in microstructure and mechanical properties of Ti-6Al-4V due to Swift Heavy Ion (SHI) irradiation damage. The results of hardness measurements showed that the studied Ti-6Al-4V (wt.%) and Ti-6Al-4V-1B (wt.%) alloy, under the specified irradiation conditions, exhibited a high irradiation resistance. In fact, only a slight hardening was observed (~13%) in the tested samples and no changes in the microstructure were observed. Temperature, dose and electronic excitation effects were also discussed.

1. Introduction:

The Facility for Rare Isotope Beams (FRIB) [1] at Michigan State University is a new generation accelerator with high power heavy ion beams. A rotating water-cooled thin-shell metal drum was chosen as the basic concept for the beam dump. The FRIB will provide primary beams from O to U with an energy of 200 MeV/u for heavy ion beams, and higher energies for lighter beams. The target beam dump lifetime is one year of operations (5500 hours). For high energy ion beams, Electronic Energy Loss (S_e) was identified as the main damage process [2]. Even in metals such as pure titanium, high S_e could induce defect [3] and track [4] formations. In different materials, combined effects of electronic energy loss and nuclear energy loss processes are reported: in some cases high electronic energy loss induces an annealing of the defects created by elastic collisions and could lead to recrystallization (SiC [5], Ni [6], Fe for $S_e < 40 \text{ keV.nm}^{-1}$ [7]) or an enhancement of defect creation (Fe for $S_e > 40 \text{ keV.nm}^{-1}$ [7], Ti [3] and in a significant amount (three orders of magnitude) in AlN [8]).

The widely used Ti-6Al-4V was proposed as a candidate material for the FRIB beam dump due to its attractive mechanical properties and corrosion resistance [9]. In addition, 1wt.% boron-modified Ti-6Al-4V, Ti-6Al-4V-1B, demonstrated enhanced mechanical, fatigue, and creep resistance, [9-11] making it also a candidate material for the beam dump.

The dpa dose in a Ti-alloy shell beam dump was estimated between 0.5 dpa for the lightest beam and 6.3 dpa for uranium beam after one year of FRIB operations. The S_e in Ti-6Al-4V shell corresponds to a value between 0.1 keV.nm^{-1} to 13 keV.nm^{-1} . These high electronic excitations can induce track formations and structural modifications in metallic systems with combined effects of S_e and S_n [2]. We proposed to irradiate this compound with swift heavy ions, having an S_e representative of the FRIB conditions. In order to investigate the

combined effects of S_e and S_n , we used lower energy, leading to some ballistic effects. Lower energy ions also allow us to have similar electronic stopping powers but without the activation of the samples, which is very convenient for further analysis.

In previous studies, the irradiation damage in Ti-6Al-4V was mostly investigated using neutrons and protons for applications in the nuclear industry. Hardening, vanadium precipitate formation and α -to- β phase transformation were reported [13-17]. Tähtinen *et al.* suggested that different hardening mechanisms are operating for neutron-irradiated Ti-6Al-4V, irradiated with neutrons at 50°C and 350°C [17].

The boron-modified Ti-6Al-4V-1B has yet to be investigated under neutron or heavy ion irradiation. Concerns about a possible detrimental effect of boron addition in this Ti-alloy stem from an observed increase of defect concentrations in metals with the increase of impurities, like with ferritic alloys, due to precipitate segregation at the grain boundaries [18-19] and specifically in boron-doped graphite [20-21].

A thorough knowledge of the properties of both these Ti-alloys after irradiation is crucial to optimize the design and performance of the beam dump. In addition, the investigation of the parameters influencing the Swift Heavy Ion (SHI) damage in these alloys (temperature and dose) and the effect of boron addition on the irradiation resistance of Ti-6Al-4V would be of interest for future applications.

2. Experimental methods

2.1. Materials preparation

Before irradiation, Ti-6Al-4V and Ti-6Al-4V-1B samples, of a thickness of 0.5 mm, were electrodischarge machined (EDM) into dogbone-shaped tensile samples. For each material, all samples were made from the same metal sheet. The gage section of the tensile

samples was ~3 mm wide and the gage length was equal to 10 mm. They were mechanically polished up to a 4000 grit with SiC paper. Then, they were polished for 4 hours with a chemical etching solution of: 5 parts colloidal silica (0.04 μm) with 1 part H_2O_2 .

2.2. Microstructure characterization

A Mira 3 TESCAN (Czech Republic) Scanning Electron Microscope (SEM) was used to image the alloys' microstructure in Secondary Electron (SE) and Backscatter Electron (BSE) modes. The Electron Backscatter Diffraction (EBSD) detector from EDAX Company in combination with the TexSEM Labs Orientation Image MicroscopyTM software, were used to collect and analyze the EBSD Kikuchi pattern data. The microstructure of the samples was characterized before and after irradiation using SEM and EBSD.

2.3. Irradiation conditions

Two irradiation experiments with both Ti-6Al-4V and Ti-6Al-4V-1B samples were performed at the GANIL-IRRSUD facility, Caen France. The two beams used were ^{36}Ar at an energy of 36 MeV and ^{131}Xe at 92 MeV. The IRRSUD beam line was chosen due to comparable S_e values to the FRIB conditions without the activation of the sample. The electronic energy loss S_e and the nuclear stopping power S_n were calculated using SRIM-2013 (The stopping and range of ions in matter) [22]. The dose level (in dpa) was also calculated using SRIM-2013 [22] assuming a displacement energy E_d of 30, 25 and 40 eV respectively for Ti, Al and V [23]. The estimated values of the ranges and S_e at the surface were respectively, 6.6 μm and 7.4 $\text{keV}\cdot\text{nm}^{-1}$ for the Ar beam and 8.5 μm and 19.4 $\text{keV}\cdot\text{nm}^{-1}$ for the Xe beam. For the highest fluences, we reached a dose of 1.2 dpa just below the implantation depth for the Ar beam and a dose of 2.8 dpa for the Xe beam. As a result of the samples' high thickness, which was ~500 μm , the damage was not homogeneous and ion implantation occurred. All experiments were conducted both at

25°C and 350°C to investigate the dose and temperature dependence [17]. With the Ar beam, three samples were irradiated with a 6 µm commercially pure (CP) Ti foil on the surface in order to have higher damage closer to the surface as shown in Figure 1. Based on the foil thickness (6 µm) and the energy loss calculated from SRIM 2013 [22], the beam energy was estimated to be 0.78 MeV at the Ti sample surface (energy after a beam trajectory of 6µm in Ti). The concentrations of implanted Ar (fluence of 7.10^{15} ions.cm⁻²) and Xe (at maximum fluence of 7.10^{14} ions.cm⁻²) ions were estimated using SRIM-2013 [22] to be 0.4 at.% and 0.13 at.%, respectively. These values are small compared to typical concentrations reached where the effect of implantation was studied. For example, the maximum implanted N concentration was ~20 at.% in one study and this affected the abrasion properties [24]. In another study the maximum implanted N concentration was ~10 at.% and this affected the corrosion resistance [25]. We assume that the effect of the implanted Ar and Xe gas is negligible (no bubble formation). The irradiation conditions with the calculated displacement per atom, S_e , S_n and the performed characterizations on each sample are listed in Table 1.

2.4. Nano-indentation and Vickers hardness

Nano-indentation was performed on the surface of the samples containing a CP-Ti foil covering them as shown in Figure 1. These samples were labelled as I-T1-x, I-T2-x, I-TB-x and T5. A MTS Nano Indenter in continuous stiffness mode (CSM) with a Berkovich tip was used with a strain rate of 0.05 s⁻¹. The distance between the indents was 50 µm and the Poisson ratio was 0.33. We compared the results obtained in the irradiated areas to those obtained in an unirradiated sample. Nano-indentation was performed on the I-T2 sample, which did not have a CP-Ti foil over it. The results were compared with the I-T2-x sample, irradiated at the same conditions but with a 6 µm CP-Ti foil covering it to investigate the effect of the dose on the

irradiation damage in Ti-6Al-4V. Vickers hardness was performed on the gage section of the following Ti-6Al-4V samples: I-T1, I-T2, I-T3 and I-T4 and the results were compared with the unirradiated Ti-6Al-4V sample T5.

3. Results and discussion

3.1. Microstructure characterization

The Ti-6Al-4V alloy microstructure contained a matrix of equiaxed α -phase with a β -phase that mostly decorated the grain boundaries with some intragranular β -phase. The grain size of the α -phase ranged between 5 and 20 μm . The volume fraction of the β -phase was 0.066 calculated from measurements using ImageJ software on several BSE images. The volume fraction of the α phase was approximately 0.93. The microstructure of the as-received Ti-6Al-4V-1B alloy exhibited both an equiaxed ($d=7.4 \mu\text{m}$) and lenticular α -phase the total volume fraction of the α -phase was approximately 0.79. The β phase and TiB phase volume fractions were 0.15 and 0.059, respectively [10].

The microstructure of all samples was characterized by taking SE and BSE images before and after irradiation. The results are included in Figure 2 (a,c) for I-T3 and Figure 3 (a,c) for I-TB. In addition, we compared the EBSD Inverse Pole Figure (IPF) orientation maps, where a color is assigned for each crystallographic orientation as shown in Figure 2 (b,d), for I-T3, and Figure 3 (b,d) for I-TB. For samples irradiated with 92 MeV ^{131}Xe at a fluence of $7.10^{14} \text{ ions.cm}^{-2}$ and at room temperature, the surface of the irradiated areas on the sample was covered with a shiny golden layer. A similar oxidization effect was reported by Zuo *et al.* in Ti-6Al-4V irradiated with nitrogen ions at room temperature [26]. No detectable change in the microstructure or the crystallographic orientation after irradiation was observed in the irradiated Ti-6Al-4V and

similarly in the boron modified Ti-6Al-4V-1B as shown in Figures 2 and 3 for two samples I-T3 and I-TB.

3.2. Hardness measurements

The results of Vickers hardness for Ti-6Al-4V samples irradiated with 92 MeV ^{131}Xe at room temperature (I-T3 and I-T4) are shown in Figure 4. For loads from 10 g (indent depth $\sim 1\text{ }\mu\text{m}$) to 300 g (indent depth $\sim 5.7\text{ }\mu\text{m}$), the irradiated samples showed slightly higher hardness compared to the unirradiated samples, and the sample irradiated at a higher fluence ($7.10^{14}\text{ ions.cm}^{-2}$ IT4) exhibited a higher hardness increase. This typical increase in hardness after irradiation has been observed previously in Ti-6Al-4V irradiated with swift heavy ions [28], protons [14] and neutrons [17]. For higher loads, up to 1kg (11 μm), no difference between the irradiated samples and the non-irradiated sample was observed.

Vickers hardness measurements were not performed on samples irradiated with CP-Ti foils covering them due to the small penetration depth (only 0.6 μm). Instead, nano-indentation was performed on such samples, including Ti-6Al-4V (I-T1-x and I-T2-x) and Ti-6Al-4V-1B (I-TB-x) samples. The results are shown in the Figures 5-7. We observed an increase in hardness for irradiated Ti-6Al-4V of $\sim 13\%$ (average hardening at a depth of 200-300 nm) (see Figure 5) and $\sim 11\%$ for Ti-6Al-4V-1B samples (average hardening at a depth of 200-300 nm) (see Figure 6) compared to the unirradiated samples.

The depth of the affected area is estimated to be three times the indenter depth [29] (i.e., for an indent depth of 3.5 μm , the depth of the affected area was $\sim 10\text{ }\mu\text{m}$). The hardness value at the near surface ($<100\text{ nm}$) could be very high due to the oxidized layer and should not be taken into account. The relative hardness as a function of the dpa value was estimated as following: We measured the hardness at a certain depth (or the mean hardness around a certain depth) and

we calculated the average dpa value for a thickness of three times the average indent depth. For example, in Figure 5 for the sample irradiated at 350 °C, the average relative hardness between 200 nm and 400 nm is ~ 1.4 . This corresponds to an average dpa of 0.95 in the probed area (first 900 nm).

3.3. Discussion

a. The effect of Boron addition

In Figure 6, for the irradiated Ti-6Al-4V-1B sample, I-TB-x, we observed a similar hardening ($\sim 11\%$ average hardening at a depth of 200-300 nm) than what was observed for the Ti-6Al-4V I-T2-x ($\sim 13\%$ average hardening at a depth of 200-300 nm) sample. Thus, it appears that Boron addition did not have a detrimental effect on the irradiation resistance of Ti-6Al-4V for the investigated dose (~ 1.2 dpa).

b. Effect of S_e and S_n

Figure 7 shows a comparison between the hardness of the Ti-6Al-4V sample irradiated with ^{36}Ar at 350°C with and without the Ti-foil on the surface. We observed higher hardening for the sample with the Ti-foil on the surface characterized by a higher S_n on the surface ($\sim 0.25 \text{ keV.nm}^{-1}$) and lower S_e ($\sim 1.4 \text{ keV.nm}^{-1}$) compared to the sample without the Ti-foil ($S_n \sim 0.015 \text{ keV.nm}^{-1}$ and $S_e \sim 7.4 \text{ keV.nm}^{-1}$). This suggests that the hardening in Ti-6Al-4V is mainly dependent on the ballistic effect and that this alloy is resistant to the damage caused by high electronic excitations. Further investigations of the irradiation-induced hardening at higher electronic excitation energies would be required to confirm this result.

c. The effect of the temperature

Figure 8 presents a compilation of the data obtained from the hardness measurements on the studied Ti-6Al-4V samples irradiated with Ar at 36 MeV. We used only the relative

hardness measured close to the surface of the samples. For the Vickers hardness measurements, the hardness at a load of 10 g and an indent depth of $\sim 1 \mu\text{m}$ was used. For the nano-indentation measurements, the hardness at 200 nm was used. The dose was approximated at the estimated depth of the affected area, i.e. 3 times the indent depth [29]. This simplified method is used as an indicator of the temperature dependence with respect to an estimated dose.

From Figure 8, we can see that the increase in the hardness due to irradiation at low temperature was less dramatic than the increase in hardness due to irradiation at higher temperatures. It seems that, at low temperature, the relative hardness for the samples irradiated with Ar at 36 MeV is less dependent upon the dpa dose level. We have included a comparison of our results to calculated relative hardness measurements from a study performed by Dayal *et al.* [27] on Ti-6Al-4V samples irradiated at 25°C with successive Ar and He implantations at energies between 100 keV to 400 keV. The value of the hardness for room temperature Ar double implantation [27] agrees with the trend observed at low temperatures for our samples. At a higher dose of ~ 11.7 dpa, the hardening at low temperature was less than what was observed at high temperature for a much lower dose. For He double implantation, the hardening is higher at lower doses than what was observed in our samples due to the combined effect of bubble formation as a result of ion implantation and displacement damage [27]. This confirms that the low concentration of implanted ions in our samples did not affect the relative hardening of the Ti-6Al-4V. These results suggest that, for irradiation at higher temperatures, Ti-6Al-4V is more sensitive to the dpa dose level than at lower temperatures. Marmy *et al.* [14] reported a similar dual dose-temperature dependence in Ti-6Al-4V irradiated with protons at a dose of 0.3 dpa : room temperature tensile tests on samples irradiated with the high

temperature of 350°C showed a higher yield strength than for samples irradiated at low temperature (40°C).

The microstructure of irradiated Ti-6Al-4V was investigated by Tähtinen *et al.* [17] (Neutron irradiation at 350°C and at 0.3 dpa) and Wilkes *et al.* [30] (irradiation with 17.5 MeV Cu^{4+} at 250°C, 350°C and 450°C at 1.5 dpa). At high temperature, the microstructure of irradiated Ti-6Al-4V is characterized by V-rich precipitates [17] or β phase precipitation [30] at doses between 0.3 and 1.5 dpa. However, at low temperature, TEM investigations showed mainly a high density of dislocation loops [17]. The microstructure evolution induced different obstacles to the dislocation motion and could explain the higher hardening at high temperature and doses. Further TEM investigations are planned to verify whether we observe similar microstructural changes in our samples.

3.4. Summary and conclusions

The SHI irradiation damage was studied in Ti-6Al-4V, Ti-6Al-4V-1B through SEM/EBSD, Vickers hardness and nano-indentation. We used different irradiating particles (^{36}Ar , ^{131}Xe) at different energies (36 MeV, 92 MeV) and at low doses (1.2 and 2.8 dpa respectively). The irradiation temperatures were 25°C and 350°C.

SEM and EBSD characterization of the Ti-alloys before and after irradiation showed that no microstructural changes were observed. The analyzed hardness and nano-indentation results for Ti-6Al-4V suggest both a low sensitivity to high electronic excitation and that the irradiation damage is affected mainly by a dual dose and temperature dependence. In addition, we observed that boron addition did not affect the irradiation resistance of Ti-6Al-4V.

These results provide valuable information on the irradiation resistance of the candidate materials for FRIB beam dump. Further investigations are needed to study the combined

effects of high irradiation dose, corrosion and fatigue on the beam dump to better validate the material selection.

Acknowledgement:

This work was partially supported by the U.S. Department of Energy Office of Science under Cooperative Agreement DE-SC0000661, the State of Michigan and Michigan State University. Michigan State University designs and establishes FRIB as a DOE Office of Science National User Facility in support of the mission of the Office of Nuclear Physics. This work was also supported by Michigan State University under the Strategic Partnership Grant “FRIB - Materials in Extreme Environments”.

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Table of Figures:

Figure 1. (a) The SRIM-2013 calculation of the dose and S_e in a Ti-6Al-4V sample with a 36 MeV ^{36}Ar beam with a fluence of 10^{15} ions.cm $^{-2}$ – (b) experimental set up for the plate

Figure 2. BSE images and IPF maps before (a), (b) and after irradiation at the same area (c), (d) in the Ti-6Al-4V sample I-T3 irradiated with the 92 MeV ^{131}Xe beam at $T=25^\circ\text{C}$ and a fluence of 2.10^{14} ions.cm $^{-2}$

Figure 3. BSE images and IPF maps before (a), (b) and after irradiation at the same area (c, d) in the Ti-6Al-4V-1B sample I-TB irradiated with a 36 MeV ^{36}Ar beam at $T=350^\circ\text{C}$ and a fluence of 1.10^{15} ions.cm $^{-2}$

Figure 4. Vickers Hardness versus indentation depth for Ti-6Al-4V irradiated with the 92 MeV ^{131}Xe beam at $T=25^\circ\text{C}$ and two different fluences (2.10^{14} ions.cm $^{-2}$ (I-T3) and 7.10^{14} ions.cm $^{-2}$ (I-T4))

Figure 5. Hardness versus indentation depth for Ti-6Al-4V unirradiated (T5) and irradiated with a 0.78 MeV ^{36}Ar beam at a fluence of 1.10^{15} ions.cm $^{-2}$ and at two different temperatures (25°C (I-T-1-x) and $T=350^\circ\text{C}$ (I-T2-x)) with the CP –Ti foil on the surface like shown on Figure 1. The dark dashed line represents the dose profile vs. depth

Figure 6. Hardness versus indentation depth for Ti-6Al-4V-1B unirradiated and irradiated with a 0.78 MeV ^{36}Ar beam at $T=350^\circ\text{C}$ and a fluence of 1.10^{15} ions.cm $^{-2}$, with the CP –Ti foil on the surface like shown on Figure 1. The dark dashed line represents the dose profile vs. depth

Figure 7. Hardness versus indentation depth for Ti-6Al-4V irradiated with a 0.78 MeV ^{36}Ar beam at a fluence of 1.10^{15} ions.cm $^{-2}$ and $T=350^\circ\text{C}$ with the CP –Ti foil (I-T2-x) and without Ti-foil (I-T2)

Figure 8. Relative hardness measurements versus dose for Ti-6Al-4V samples irradiated with Ar @ 36 MeV at $T=25^\circ\text{C}$ and $T=350^\circ\text{C}$ and for the different dose levels compared to relative hardness extracted from Dayal *et al.*[25] for Ti-6Al-4V irradiated at $T=25^\circ\text{C}$ with Ar ions @ 100-400 keV (indent depth ~ 70-200nm) and He ions @ 100-400 keV (indent depth ~ 70-200nm)

Table 1. Description of the studied samples and the performed characterization

Samples	Material	Irradiated with Ti-Foil	Temperature (°C)	Fluence (ions.cm ⁻²)	Ion beam	Energy (MeV)	Range (μm)	S _e at the surface (keV.nm ⁻¹)	S _n at the surface (keV.nm ⁻¹)	Dpa at the surface	Dpa at the Bragg peak	SEM/EBSD	Nano-indentation	Vickers Hardness
I-T1	Ti-6Al-4V		25	10 ¹⁵	³⁶ Ar	36	6.6	7.4	0.015	0.03	1.2	x		x
I-T2	Ti-6Al-4V		350	10 ¹⁵	³⁶ Ar	36	6.6	7.4	0.015	0.03	1.2	x	x	x
I-T1-x	Ti-6Al-4V	x	25	10 ¹⁵	³⁶ Ar	0.78	0.5	1.4	0.25	0.53	1.2	x	x	
I-T2-x	Ti-6Al-4V	x	350	10 ¹⁵	³⁶ Ar	0.78	0.5	1.4	0.25	0.53	1.2	x	x	
I-T3	Ti-6Al-4V		25	2.10 ¹⁴	¹³¹ Xe	92	8.5	19.4	0.15	0.051	0.8	x		x
I-T4	Ti-6Al-4V		25	7.10 ¹⁴	¹³¹ Xe	92	8.5	19.4	0.15	0.182	2.8	x		x
I-TB	Ti-6Al-4V-1B		350	10 ¹⁵	³⁶ Ar	36	6.6	7.4	0.015	0.03	1.2	x		
I-TB-x	Ti-6Al-4V-1B	x	350	10 ¹⁵	³⁶ Ar	0.78	0.5	1.4	0.25	0.53	1.2	x	x	
T5	Ti-6Al-4V											x	x	x

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8