Impact of heat treatment on tensile properties of 97W-2Ni-1Fe heavy alloy

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Abstract: To avoid failures caused by the inherent brittleness of pure tungsten below its ductile to brittle transition temperature, tungsten heavy alloy was proposed as an alternative solution of the plasma facing material. Plasma facing components manufactured of tungsten heavy alloy have been installed partly in the tokamak ASDEX Upgrade instead of pure tungsten targets. Opposite to the pure tungsten targets, no deep cracks have been identified in these targets after one experimental campaign. In order to understand and improve the material behaviour of tungsten heavy alloy as plasmam facing material in ASDEX Upgrade, the impact of heat treatment on tensile properties of 97W-2Ni-1Fe heavy alloy has been studied in this work. The heat treatments have been conducted in the vacuum for the tensile specimen cut from an ASDEX Upgrade target at different temperatures (600 °C, 1100 °C and 1350 °C) with various durations (15 min, 60 min and 120 min). After the heat treatment, the tensile properties were remarkably improved. The increase of the interfacial strength between tungsten grains and matrix phase as well as growth of grains are considered as the main reasons for the increase of elongation after heat treatment. The experimental results reveal that electrical discharge machining reduces the total elongation of the specimens. Furthermore, no big impact on the tensile properties is found after low energy deuterium plasma implantation.

Keywords: tungsten heavy alloy, plasma-facing component, heat treatment, tensile test

1. Introduction

Tungsten is the main candidate material for the plasma facing components (PFCs) in the fusion reactor due to its excellent physical properties, such as high thermal conductivity, highest melting point of all metals, low sputtering yield etc. The main function of the most heavily loaded PFC, the so-called divertor target, is to exhaust the power and particles stemming from the hot main chamber plasma [1]. Following the massive temperature increase in the target resulting from the particle bombardment, the stress in the target is critical to the plasma facing material. Deep cracks in the tungsten divertor target have been frequently observed in the high heat flux tests of actively cooled ITER divertor mock-ups [2] as well as in the tokamak ASDEX Upgrade which uses adiabatically loaded bulk tungsten divertor tiles [3]. The crack formation is considered to be attributed to the brittleness of tungsten below its ductile to brittle transition temperature (DBTT) [4].

To avoid loss of structural integrity due to brittle cracking in the divertor target, it was proposed [5] to use tungsten heavy alloy (W-Ni-Fe) instead of pure tungsten as the target material in ASDEX Upgrade. Tungsten heavy alloys are two- phase composites consisting of dispersed tungsten grains in a ductile phase, because of which they can have high density, high strength and high ductility at low temperature at the same time [6]. Furthermore, the tungsten heavy alloys are considerably cheaper due to the facilitated sintering process and they show improved machinability compared to pure tungsten. During the 2017 ASDEX Upgrade campaign, the divertor targets made of tungsten heavy alloy showed no macroscopic failure as also expected from the finite element analysis [7], while macroscopic cracks have been found in almost all the divertor targets made of pure tungsten in the previous campaign [3].The mechanical properties of tungsten heavy alloy are strongly dependent on its manufacturing process [8, 9, 10]. Many investigations have been focused on the cause of loss of ductility in the tungsten heavy alloy with different tungsten composition. It turned out that, the heat treatment would be the most practical way to improve the mechanical properties of the tungsten heavy alloy.

In order to understand and improve the material behaviour of tungsten heavy alloy in the ASDEX Upgrade divertor, the impact of heat treatment on tensile properties of 97W-2Ni-1Fe heavy alloy is investigated in this work. The tensile specimens were produced from the as delivered divertor target for ASDEX Upgrade and have been heat treated in vacuum under various conditions before the tensile tests at room temperature. Microstructural and morphological analyses were conducted in order to elucidate the mechanism of variation of tensile properties due to previous heat treatment. Furthermore, the impact of deuterium implantation as expected under plasma conditions on the tensile properties has been investigated.

2. Experimental

The tungsten heavy alloy investigated in this work was supplied by HC Stark Hermsdorf GmbH, named HPM 1850. HPM 1850 consists of 97 wt.% W, 2 wt.% Ni and 1 wt.% Fe. The mixed powders of tungsten,

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nickel and iron were pressed and then liquid phase sintering was applied. No additional heat treatment was conducted. The tensile specimens were prepared out of an as-received tungsten heavy alloy divertor target tile (15 × 77.27 × 227.62 mm^3) from HC Stark Hermsdorf GmbH for the ASDEX Upgrade divertor.

A universal testing device (TIRAtest 2820, TIRA GmbH) with a 20 kN load cell was used to perform the tensile tests. To avoid exceeding the maximum load capacity, the standard sample geometry listed in the ASTM E8/E8M was not considered. Instead, specimens with a smaller cross-section were used. The tensile tests were performed with a crosshead speed of 10 µm/s. The strain was measured by 2D Digital Image Correlation (DIC) with a 20 Hz camera, the results of which are in line with conventional strain gauge measurement. The DIC is a non-contact measurement technique, which is considered to give a more accurate result. By applying DIC, two feature areas in the gauge section are first selected. These feature areas are successfully tracked in different frames during the test, see Figure 1. The relative vertical displacement is thus calibrated by pixels.

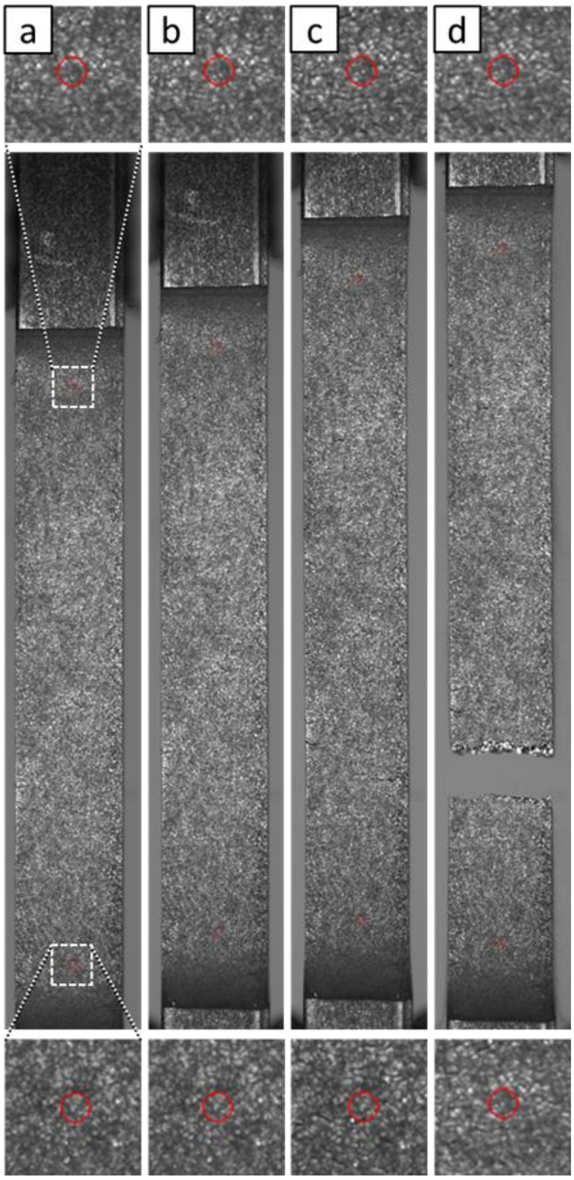

Figure 1 Example images as used by DIC, a) Strain = 0% (initial), b) Strain = 1.43%, c) Strain = 10.7% (last frame before fracture), d) First frame after fracture

The heat treatment of the specimens was conducted in a quartz tube furnace under a vacuum of ca. 10^{-4} Pa at different temperatures. The period of heat treatment considers only the time when the temperature had reached the target temperature. The rates of heating and cooling were roughly 5 °C/min. All tensile tests were conducted at room temperature.

To ensure suitability and reliability of the smaller testing samples, three types of specimen (see Figure 1) were prepared: 1) rectangular specimen proportional to the standard in ASTM E8, 2) cylindrical specimen proportional to the standard in ASTM E8 and 3) SS-J-type tensile specimen [11]. The specimens were prepared with Electrical Discharge Machining (EDM) and milling. During the manufacturing, one cylinder sample was broken. The SS-J-type tensile specimen showed a strong bending after milling. The rectangular specimens are the easiest to manufacture and they showed also rather stable results in the tensile tests, see Figure 3. The total elongation of other specimens was similar or smaller than that of rectangular specimens. Therefore, rectangular type is selected for the further investigation in this work. If not specifically mentioned, the rectangular specimens were finalized by milling from a rectangular block (60 × 5 × 3 mm^3), which was cut by EDM from the target tile.

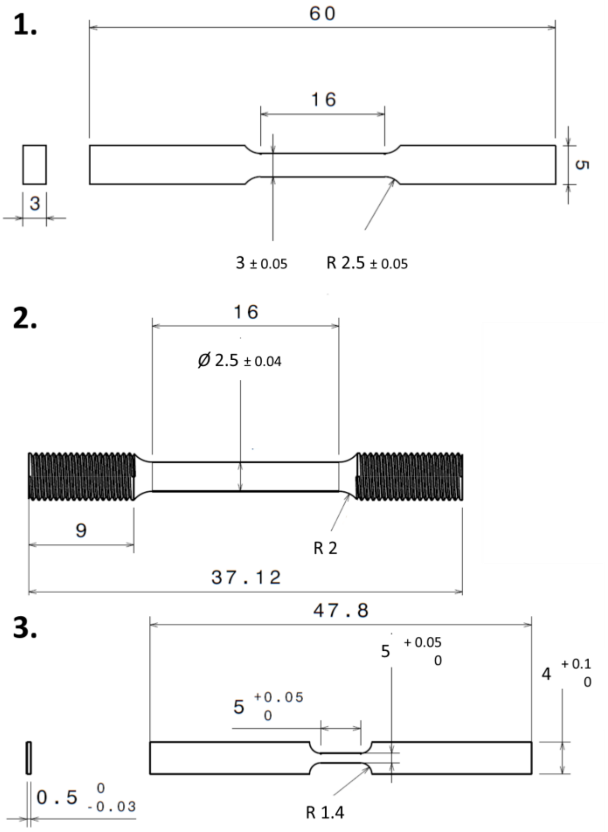

Figure 2 Dimensions of three types of specimens (unit: mm).

3. Results

Figure 3 shows the engineering stress-strain curves of three specimens without heat treatment. The three curves are quite similar indicating a good homogeneity of the material. Yielding has been observed, however, the total elongation is only ca. 0.5%, which is quite different from the values in the delivery note (total elongation of 3.5%). It should be noted that the samples for generating data in the delivery note are produced in a different way. The impact is discussed in the next section. Figure 4 shows the engineering stress-strain curves of the specimens after 60 min heat treatment at 1100 °C. The stress-strain curves are quite similar for all six tested samples, and the total strain lies between 9 % and 12 %. Compared to the specimens without heat treatment, the total strains increase dramatically by almost of a factor of 20. Two of the six specimens (total strains: 9.2 % and 10.3 %) have been shock cooled with oil, and no essential difference of total elongation was found compared with other specimens (cooling rate: 5 °C/min).

Tensile tests were also conducted for specimens, which were heat treated for various durations at different temperatures. For each temperature and duration, three specimens were tested. Figure 5 and Figure 6 show the total elongation and tensile strength for different heat treatment durations (i.e. 15, 60 and 120 min) at 1100 °C and 1350 °C, respectively. The total elongation increases slightly with increasing heat treatment duration at 1100 °C, while for the heat treatment at 1350 °C the largest total elongation is obtained for the specimen which had been heat treated for 60 min. The tensile strengths were not as sensitive as the total elongation to the heat treatment duration and temperature in the above-mentioned testing range. The tensile strength ranged from800 and 900 MPa and generally increased slightly with the heat treatment duration. Figure 7 and Figure 8 show the total elongation and tensile strength of specimens after 60 min heat treatment at different temperatures, respectively. The specimens without heat treatment are also plotted here as a reference. The total elongation increased dramatically with increasing heat treatment temperature, whereas the change of the tensile strength was more moderate. The maximum value of about 900 MPa is already in the range of tensile strength of stress-relieved tungsten and is even larger than those (340 – 540 MPa) of the pure tungsten specimens cut from the bulk tungsten divertor tile from ASDEX Upgrade using the same machining and heat treatment [7].

No local necking has been observed. The hardening behavior of all tested samples is similar.

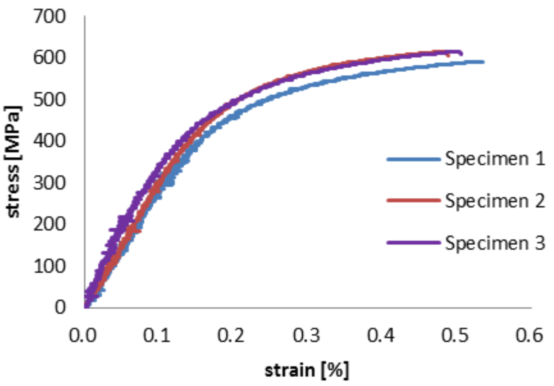

Figure 3 Engineering stress strain curves of specimens without heat treatment. Different colors stand for different specimens.

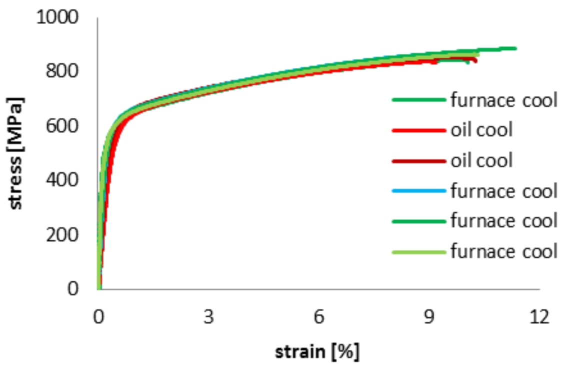

Figure 4 Engineering stress strain curves of specimens after heat treatment for 60 min at 1100 °C.

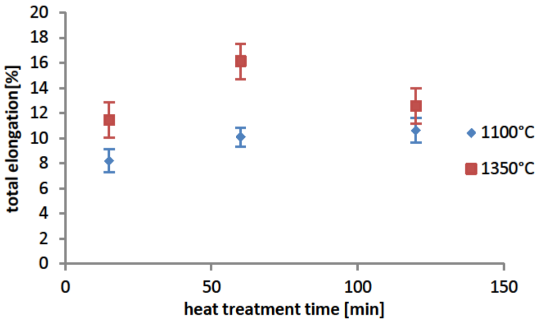

Figure 5 Total elongation of specimens for different heat treatment durations at 1100 °C and 1350 °C.

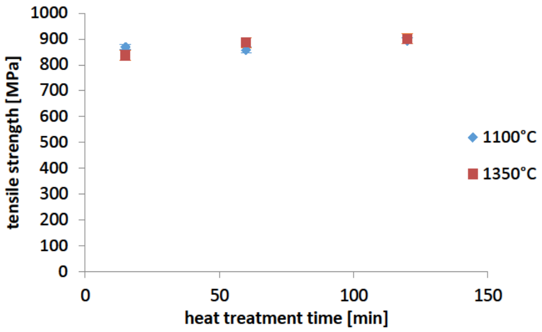

Figure 6 Tensile strength of specimens for different heat treatment durations at 1100 °C and 1350 °C.

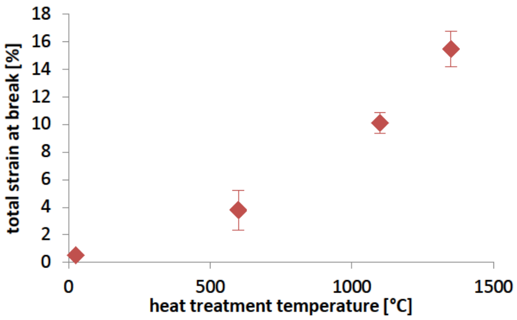

Figure 7 Total elongation of specimens after 60 min heat treatment at different temperatures.

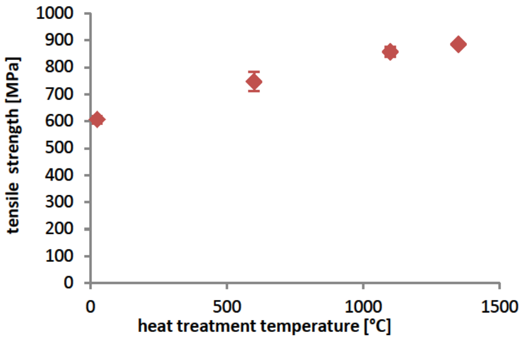

Figure 8 Tensile strength of specimens after one-hour heat treatment at different temperatures.

4. Discussion

The results of the tensile tests revealed that heat treatment strongly increases the total elongation of tungsten heavy alloy taken from the as-received target. This improvement is considered to be attributed to two aspects: 1. recovery of defects and/or damages caused by machining (e.g. EDM); 2. improvement of the material strength.

To investigate the impact of the first aspect, a study of the impact of EDM is conducted. A block of tungsten heavy alloy (60 × 15 × 20 mm) was cut from the same target by EDM. Subsequently, it was heat treated at 1100 °C for 60 min.

Heating and cooling were slowly thus no additional residual stress is expected for different geometries. In [9], the heat treatment duration was suggested to be proportional to the square of the linear dimension, which indicates 60 min of heat treatment for the large block (linear dimension: 95 mm) would be comparable to ca. 30 min of heat treatment for the tensile specimens (linear dimension: 68 mm). Based on the data in Figure 5, specimens after 50 min of heat treatment would have a slightly smaller total elongation than that of the specimens which have been heat treated for 60 min at 1100 °C. Therefore, the tensile property of the block is assumed to be similar as or slightly worse than that of the tensile test specimens, which have been heat treated at 1100 °C for 60 min, see Figure 4.

Three specimens were cut from the block using only EDM. The elongations were 4.5%, 3.6% and 4.3%. Another three specimens were cut from the block by EDM and then again heat treated at 1100 °C for 60 min. The elongations are 6.3%, 7.3% and 9.2%.

After EDM, the average elongation reduced to 4.1% from the previously measured 10.1% (see Figure 7), and increased again to 7.6% after the second heat treatment. Therefore, it is concluded that EDM reduces the total elongation, which can be partly recovered by heat treatment. As possible mechanism residual stress and/or hydrogen embrittlement introduced by the machining were discussed in [12, 13] and [14], respectively. The latter will be investigated and discussed later in this section.

The impact of the second aspect is easier to be proven. In the delivery report of the tungsten heavy alloy target, a tensile test result of the specimen manufactured in the press form (i.e. without any machining) together with the production of tungsten heavy alloy target shows a total elongation of 3.5%, which is obviously smaller than that of the heat treated specimens. This indicates that the material properties of tungsten heavy alloy are improved by heat treatment.

The manufacturer confirmed that the delivered tungsten heavy alloy had been sintered under the hydrogen atmosphere. In the literature, the hypothesis of hydrogen on the mechanical properties was discussed in [9] by Yoon et al. It was reported that the heat treatment in non-hydrogen atmosphere can recover the degradation of the interfacial strength caused by impurity segregation of hydrogen. Further it was observed that in tensile tests, cracks appeared to propagate through the matrix without heat treatment of the specimens, while after heat treatment in vacuum, more transgranular cracks occurred [9]. The elongation increased after heat treatment in vacuum, however, no increase was found after heat treatment in hydrogen. Similarly, J. Das et al. [15] have reported that, predominantly tensile fracture in the matrix or in the interface changed to transgranular cracks in tungsten grains after heat treatment.

A similar behavior was also found in this work. Figure 8a shows the SEM micrograph of the fracture surface for a specimen without heat treatment. Figure 9b and 8c show the fracture surface surfaces of the specimens which have been heat treated for 60 min at 1100 °C and 1350 °C, respectively. In Figure 9a, the de-cohesion of the matrix from the tungsten grains is identified to be the dominant fracture mode, whereas more intragranular cracks are observed in Figure 9b and 8c. Moreover the number of intragranular cracks increases from Figure 8b to Figure 8c. Obviously, the interfacial strength has been increased by the heat treatment depending on the maximum temperature. The manufacturer confirmed that the delivered tungsten heavy alloy had been sintered under the hydrogen atmosphere.

In the investigations discussed above, the hydrogen was introduced during the sintering process or the heat treatment. In order to investigate the issue from a fusion specific aspect, tensile specimens were exposed to low temperature deuterium plasma to evaluate the effect of embrittlement under hydrogen implantation. For this purpose, three specimens were manufactured from the same target as the specimens described above. Two of the three (one was kept as a reference) specimens, which had been heat treated at 1100 °C for 60 min, were exposed in the well-characterized low-temperature plasma experiment PlaQ [16] for the deuterium implantation. The exposure was conducted for 90 hours at 227 °C (500 K) with a deuterium fluence of 3×10^{25} D/m^2. The background pressure of the neutral gas was of 1 Pa and the average ion energy was 15 eV/D. After the exposure in PlaQ, the total elongation of the two specimen was 6.6% and 8.2%, and the reference specimen (without exposure) showed a total elongation of 7.0%. As a consequence, no obvious impact of hydrogen embrittlement is found in tungsten heavy alloy specimen after deuterium implantation under the above listed conditions. However, as the mechanism of deuterium retention and transport in the tungsten heavy alloy is not well understood yet, the impact of deuterium implantation could be quite different under other experimental conditions.

Nevertheless, using the hypothesis of hydrogen embrittlement to interpret the increase of elongation by increasing the heat treatment temperature from 1100 °C to 1350 °C would be difficult, since heat treatment temperatures up to 1000 °C would be sufficient to remove the effect of hydrogen embrittlement [9]. In this work, it is considered that the growth of tungsten grain to be one of reasons for this increase. The recrystallization temperature of pure tungsten is ca. 1300 °C [17]. The size of tungsten grain in the tungsten heavy alloy increases after heat treatment at 1350 °C, while the heat treatment at 1100 °C does not change the grain size. The average size of tungsten grains in the specimens without heat treatment and after heat treatment of 60 min at 1100 °C is nearly the same, (roughly 55 µm), while it is ca. 77 µm in the specimens after heat treatment of 60 min at 1350 °C. The joint of two neighboring grains which share a tungsten-tungsten interface might also be initiated more easily during the high temperature heat treatment resulting in a decrease of solid-solid interfacial energy and area [18]. Eroglu and Baykara showed that if powder of finer tungsten grade was applied during the manufacturing, tensile properties of tungsten heavy alloy were reduced, as a higher degree of contiguity of tungsten grain resulted in early failure in tungsten heavy alloys in a more brittle manner [19].

As mentioned in the last section, the hardening behavior of all the tested specimens is quite similar. The strain hardening rate seems not to be affected by the heat treatment. The strain hardening rate is quite low, so the increase of the tensile strength is not as strong as the increase of the total strain, see Figure 7 and Figure 8.

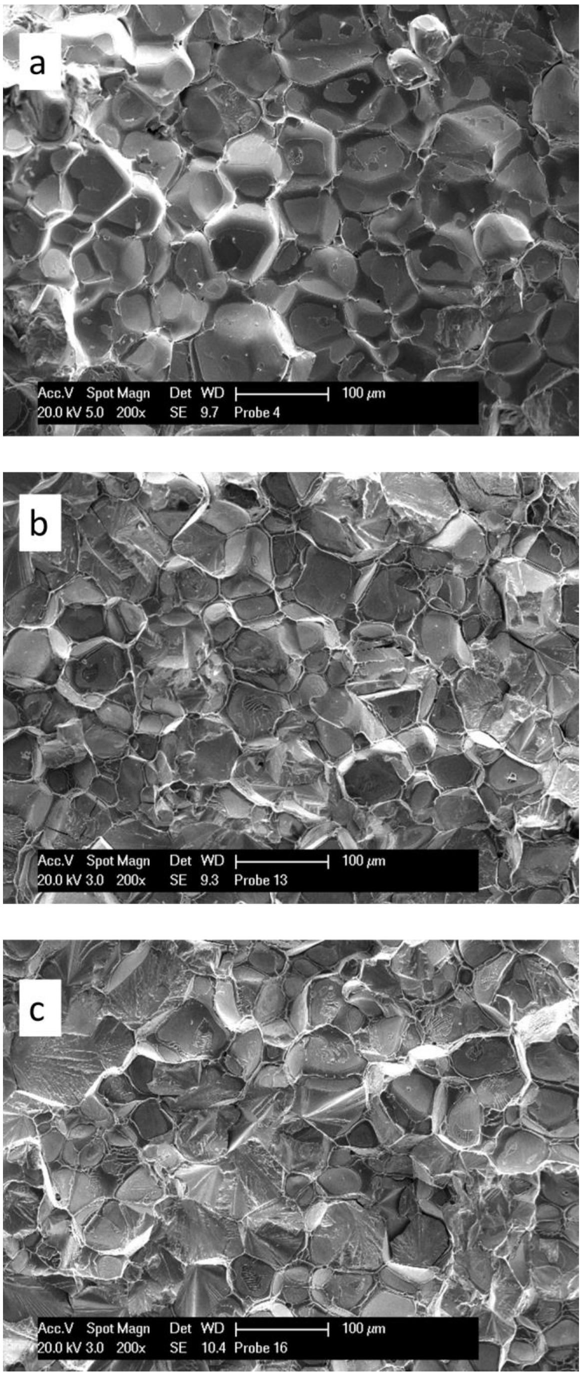

Figure 9 SEM images of fracture surfaces for the specimen (a) without heat treatment, (b) after heat treatment at 1100 °C for 60 min, (c) after heat treatment at 1350 °C for 60 min.

5. Conclusion

In this work, the impact of heat treatment on tensile properties of 97W-2Ni-1Fe heavy alloy has been investigated. The heat treatments have been conducted in vacuum at three temperatures (600 °C, 1100 °C and 1350 °C) with various holding times (15 min, 60 min and 120 min). Additionally, heat treated specimens were subjected to a low energy plasma for 90 hours at 227 °C. The results can be summarized as follows:

1. Heat treatment increases the total elongation of as received 97W-2Ni-1Fe heavy alloy significantly.
2. With increasing the heat treatment temperature, the total elongation increases.
3. The total elongation increases slightly with increasing heat treatment duration at 1100 °C, while for the heat treatment at 1350 °C the largest total elongation is obtained for the specimen which has been heat treated for 60 min.
4. Cooling rates do not change the total elongation significantly after the heat treatment at 1100 °C for 60 min.
5. Machining with EDM reduces the total elongation of the 97W-2Ni-1Fe heavy alloy, which can be partly recovered by heat treatment.
6. Deuterium implantation for 90 hours at 227 °C by low temperature plasma does have no noticeable impact on the tensile behavior.

These results support the superior mechanical behavior of the W heavy alloy tiles observed in the divertor of ASDEX Upgrade [7], and provide some confidence that this behavior is sustained under plasma exposure.

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