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Fabrication of a tantalum-clad tungsten target for KENS

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Abstract

Since the cold neutron source intensity of KENS (the spallation neutron source at High Energy Accelerator Research Organization) was decreased into about a third of the designed value because a cadmium liner at the cold neutron source deformed and obstructed the neutron beam line, the target-moderator-and-reflector assembly (TMRA) has been replaced by a new one aimed at improving the neutron performance and recovering the cold neutron source. The tantalum target has also been replaced by a tantalum-clad tungsten one. In order to bond the tantalum-clad with the tungsten block, a hot isostatic press (HIP) process was applied and optimized. It was found that gaseous interstitial impurity elements severely attacked tantalum and embrittled, and that the getter materials such as zirconium and tantalum were effective to reduce the embrittlement.

1. Introduction

The spallation neutron source is very useful for material science and technology and for nuclear engineering. KENS [1] is the first pulsed neutron source for material science in the world, which is located at the High Energy Accelerator Research Organization. It uses a solid target of tantalum bombarded by high-energy proton beam from the 500 MeV synchrotron accelerator and cooled by water. Although the facility operates at only 3 kW beam power, its cold neutron production is comparable with other large pulsed spallation sources because of its high-performance target-moderator-and-reflector system. We have waited for a chance to increase the neutron intensity of KENS for a long time and this year will accomplish this by replacing the whole target-moderator-and-reflector assembly (TMRA) which has been used for 20 years by a new one with tungsten target.

Tungsten is the most promising solid target material in the viewpoint that (1) neutron yield of tungsten is the highest among neighboring nuclides, (2) tungsten is a material for high-temperature usage and (3) it has high strength in compression. Accordingly, tungsten is/was used for a spallation neutron target at some facilities such as LANSCE [2] and even KENS. In the APT project [3], tungsten was selected for the target and elaborately studied at LANL. However, the only disadvantage of tungsten is that tungsten has a high-corrosion rate in water under radiation or high temperature. This can be avoided by cladding tungsten with a corrosion-resistant material such as titanium, stainless steel, Zircaloy-2, niobium, tantalum and gold. ISIS has developed [4] a tantalum-clad tungsten target block by means of the HIPing at 1400°C and a high pressure of 1450 MPa. However, such conditions are not easily attained with an ordinary HIP.

We have started the R&D work for determining the optimum material for a long-lived and high-neutron yield target for future spallation neutron sources for material science. In this work, tantalum has been selected as one of the hopeful candidates of a cladding material, because it has the largest neutron yield among the corrosion-resistant materials mentioned above,

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strong toughness to both high temperature and stress, and good ductility. Additionally, tungsten and tantalum are compatible due to their complete solid solubility. The issue developing tantalum-clad tungsten target is to find the proper bonding method. The HIP process seemed to be the most promising and attainable method. We would like to apply the optimum HIP conditions with respect to temperature, pressure and impurity gas control by using small samples. Then, we applied it to fabrication of the target block and succeeded to find the desirable HIP method after some trials.

In this report, we describe the fabrication of the target block together with the preceding R&D work to find the optimum condition of the HIP process.

2. History of KENS and new TMRA designs

KENS was constructed in 1980. It operates at 3 kW with a proton beam of 500 MeV in energy and 6 μA in beam-current. Proton pulses with 50 ns width bombard the target at 20 Hz. Three kinds of targets were used: a tungsten target from January 1980 to November 1985, depleted uranium target from December 1985 to November 1996 and tantalum target from December 1996 to June 2000. The tungsten target was changed to depleted uranium to increase the neutron intensity by about 50%. The first uranium target had been used for 10 years but discontinued because Xe-135 was detected in the cover gas of the reservoir tank. The second and third uranium targets had very short lives of 2 months and one day, respectively. Therefore, a tantalum target was substituted for the uranium one. The thermal neutron source was composed of a target, an ambient moderator with a size of 100 mm × 100 mm × 50 mm, a beryllium reflector with an overall size of about 600 mm and thick B_4C de-coupler as shown in Fig. 1. Initially, a polyethylene moderator was used but was replaced by a water moderator in 1988. The cold neutron source of KENS was also composed of a solid methane moderator of 100 mm × 150 mm × 50 mm, a beryllium reflector and a cadmium de-coupler. An aluminum case forming a part of the cold neutron beam line near the moderator was also covered with a cadmium liner. The nominal cold neutron flux generated by it was several times lower than those of ISIS liquid-hydrogen moderator in spite of about one-fiftieth of the proton beam intensity of ISIS.

In 1998, absolute measurement of neutron fluxes was made at various neutron beam lines by a gold foil activation method and by a fission detector. From its results, the sub-cadmium neutron flux of the cold neutron moderator was found to be about a third of the nominal value, while the epi-thermal neutron fluxes were nearly equal to the nominal value. The neutron spectrum of the cold-neutron moderator was also measured by a time-

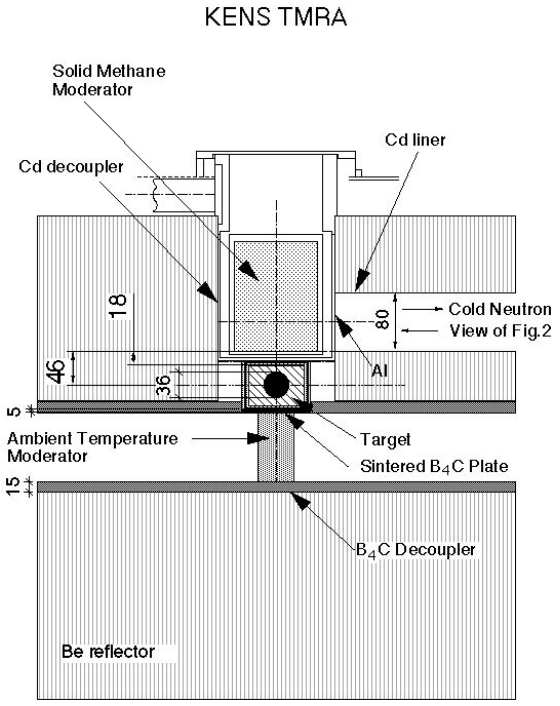

Fig. 1. TMRA of KENS.

of-flight method using a ^{235}U fission detector. The measured neutron spectrum in the energy region below the cadmium cut-off energy was depressed from a single Maxwellian distribution with a $1/E$ tail, which was obtained by the neutronics model calculation with the Monte Carlo method. It had been considered that the flux reduction and the anomalous spectrum might have resulted from a part of cadmium-liner coming off the aluminum case and shielding the beam line. In order to confirm it, inspection of the cadmium-liner was made in November and December in 1998. We found that the interior of the cadmium-liner was uneven because of heavy swelling, and the color appeared more or less white, but dark yellowish parts were seen around rivets. The swelling must have occurred due to repeated heating up to several tens of degrees in centigrade by 20 Hz proton pulses, damage by total neutron fluence of about 2×10^{19} n/cm^2, and chemical reactions with air and nitrogen oxides.

Initially we looked at methods of repairing the liner, but could not without proper remote-handling equipments because of the high-radiation levels and the possible hazards due to the chemical poison. Therefore, we decided to replace the whole TMRA by a new one. The new TMRA was designed to improve neutronics performance, by means of neutronic calculations with the Monte Carlo method using the LCS code system. It is composed of a tungsten target, ambient moderator of

water poisoned with gadolinium, cold moderator of solid methane, and a composite reflector of small amount of beryllium and large graphite. The same cadmium and B_4C de-couplers are used as in the old TMRA, while thicknesses of B_4C slab are optimized to give higher and sharper thermal neutron pulses than the old ones.

As the target material, tungsten was selected to improve neutron intensity by about 20% over the case of tantalum one. However, tungsten is corrosive in water, especially under circumstances of radiation. On the other hand, tantalum has a high-corrosion resistance in water and higher neutron generation rate than other structural material such as stainless steel, zircalloy and niobium. Accordingly, we selected it as a cladding material of tungsten block and have improved the hot isostatic press (HIP) process to join them.

3. Fabrication of target

The target is composed of four blocks and a housing as shown in Fig. 2. The front block has a hole for inserting the thermocouple. The blocks are of 29.18 mm × 56.16 mm × 76.89 mm size and covered with 0.6 mm thick tantalum-clad. Spacers to make gaps of 2 mm between the blocks separate each block. The coolant water flows through the gaps between blocks at the flow rate of 60 l/min. The tungsten block was made by machining from a rolled tungsten block of 19.2 g/cm^3 density and 99.99% purity by an electro-discharge machinery (EDM) or spark erosion technique. The hole for the thermocouple was made by the plunge EDM technique. The tantalum-clad is composed of a case and two caps. The case was made of a sheet of 0.8 mm thick tantalum of 99.987% purity by using a model that was formed by fitting to the actual tungsten block. The edge of the sheet was bonded by TIG welding. The cap with 2.2 mm depth was made by EDM and end-mil work against a sheet of 3 mm thick. The tungsten block was inserted into a case and was covered up by caps. The edges of cap and cases tentatively welded at four spots joining them. Then, the clad was perfectly sealed by welding a whole of edges by about 1 mm depth from its tip by the electron-beam-welding. The color check was made to confirm the welding result. In case of the block having the hole for the thermocouple, tantalum sheath for the hole is made of slim pipe with 1.6 mm inner diameter and 0.8 mm thickness by filling the bottom edge with small pellet. The pipe and the pellet were bonded by the TIG-welding.

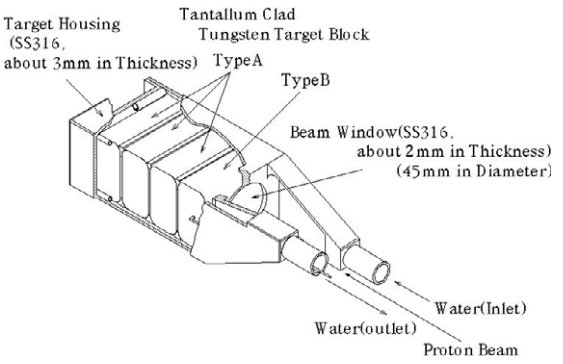

Fig. 2. Target housing and target blocks.

The HIPing was made to bond the tantalum-clad with the tungsten block. HIP condition was determined by testing it with the small samples. We found that the HIP condition is very important to bond both different materials of tantalum and tungsten that are well known as refractory materials for high-temperature usage. Especially, tantalum was very sensitive to gaseous interstitial impurity elements absorbed at high temperature. The details of test and the results of HIPing are described in the Section 4. After the HIPing, the protuberances were cut down and the surface of the block was polished to obtain the designed thickness and roughness.

4. HIP procedure

4.1. Without impurity-gas-getter material

The HIP condition was initially investigated by using the small samples of 13 mm in diameter and 13 mm in length. For the test, we prepared two kinds of samples by using a drawn tungsten rod and a rolled tungsten sheet. The former is named a rod type and the latter a disk type. The thickness of tantalum-clad was 1 mm. HIPing was made by using a furnace with a graphite heater at Tokyo Tungsten and feeding a pure argon gas of 99.999% purity (5N). Temperature of the furnace was increased at 10°C/min up to 800°C. Then a compressor was started and operated for 120 min to increase a pressure up to 1300 kgf/cm^2 in the furnace. The temperature is increased again at the same rate as before up to the desired temperature and pressure for HIP. HIP condition was kept for 180 min. Then, the temperature was decreased from −5°C/min to 800°C, during which the pressure was decreased to 1,300 kgf/cm^2 at 800°C, and at −10°C/min to room temperature. Three kinds of condition such as (1600°C, 1600 kgf/cm^2), (1800°C, 1800 kgf/cm^2), and (2000°C, 2000 kgf/cm^2), were examined. As a result of HIP, it was found that the cavities and/or pores in tungsten block disappeared and that the good bonding was obtained. The color check showed that cracks were found in tantalum-clad layer in case of the highest temperature and pressure condition of (2000°C, 2000 kgf/cm^2). The thickness of the diffusion layer in the

vicinity of the boundary of tungsten and tantalum was measured by electron probe X-ray micro-analyzer (EPMA) method detecting characteristic X-rays from both materials. The diffusion layer thickness monotonously increases from 3.5 μm at 1600°C to 15.6 μm at 2000°C. There are not so large differences between rod and disk types and positions of samples. Then, we selected the temperature 1800°C, 1800 kgf/cm^2 and keeping time of 3 h as an optimum condition of HIPing so as to obtain sufficient diffusion thickness without any cracks.

The above condition was applied to the target block of KENS with a larger graphite heater. After the HIPing, the welded edges of the both sides of the block were cut down by EDM method. We found that the caps and the trunk of tantalum were bonded well by the HIPing. However, we found also many small patterns as like a tortoise shell on the surface of the block. The Vickers hardness of the tantalum surface was very high 500 HV (Vickers micro-hardness in unit of kg/mm^2) to 1200 HV compared to about 100 HV of pure tantalum. Fig. 3 shows SEM micrographs of cross-section of tantalum and tungsten layers. The tantalum layer is covered with a yellowish layer of about 100 μm. Many cracks were observed on the surface and some cracks penetrate into tungsten layer as shown in Fig. 3. Fig. 4 shows that tantalum becomes very brittle and has many small cracks. The content of the yellowish surface layer was identified as TaC by the X-ray diffraction analysis. The hardness of the HIPed block is near to those of TaC i.e., 1800 HV [5]. Thus, we have concluded that carbon from the graphite heater attacked tantalum and made chemical compound of TaC. The chemical reactions were accelerated under too high temperature.

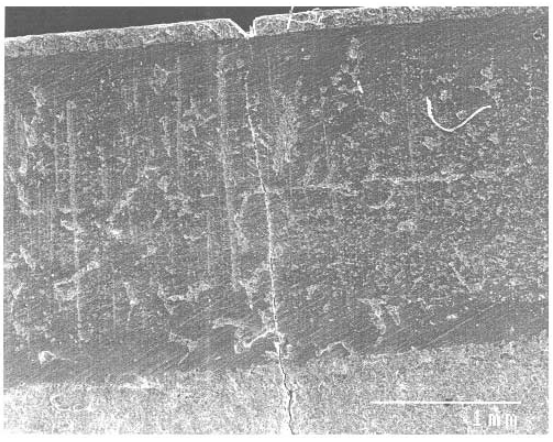

Fig. 3. SEM photo showing a surface layer of TaC and a crack penetrating through a tungsten layer of the target block HIPed using the graphite heater keeping at 1800°C and 2000 kgf/cm^2 for 3 h.

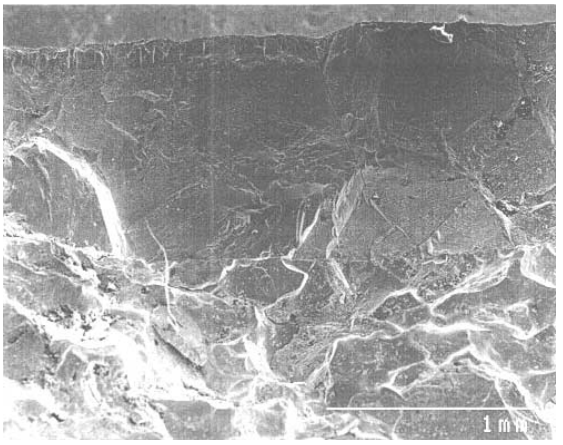

Fig. 4. SEM photo showing a brittle tantalum layer of the target block HIPed using the graphite heater keeping at 1800°C and 2000 kgf/cm^2 for 3 h.

4.2. HIP process with getter material

Further investigation of HIP procedure was made by using a small sample and a large furnace at KOBELCO to be used for processing the KENS target block under the highest pressure of 2000 kgf/cm^2. The contents of the test are summarized in Table 1.

In Case 1, we selected a large molybdenum furnace in order to avoid the influence of carbon. HIPing was made at 1400°C, feeding argon gas of 4N. However, the surface of the processed sample lost a metal brightness and became dark gray. Dark gray layer was easily peeled off and brittle. X-ray analysis identified it as Ta_2O_5 and TaC. Thus, it can be said that tantalum is very sensitive to gaseous interstitial impurity elements such as carbon, nitrogen and oxygen under high pressure even at relatively low temperature of 1400°C. In order to avoid such influence, we have to carefully control argon gas to remove gaseous-interstitial-impurities.

In Case 2, we used the molybdenum furnace at 1500°C and argon gas of 5N. The sample was enveloped with the zirconium foils and additive tantalum slab in order to absorb oxygen in the argon gas. After the HIP process, the sample did not lose a metallic brightness of tantalum and tantalum envelope kept a characteristic of high ductility.

In Case 3, we used the graphite furnace at 1600°C and argon gas of 6N. The sample was enveloped with more zirconium foils and additive tantalum than Case 2. However, the sample color looked like gold caused by the production of TaC.

From the test mentioned above, we adopted the condition of 1500°C, 2000 kgf/cm^2 and impurity-getter material as an optimum one for HIPing the KENS target blocks. Additionally, the temperature-increasing

Table 1
HIP conditions and impurity-getter materials

Case	Furnace condition temperature, heater, size[a], Ar gas	Getter materials
Case 1	1400°C, Mo, 200∅ × 500 H, 4N	None
Case 2	1500°C, Mo, 100∅ × 280 H, 5N	Ta (100 μm × 2) Zr (125 μm × 2) Zr (20 μm × 8) Ta (100 μm2)
Case 3	1600°C, C, 100∅ × 280 H, 6N	Ta (100 μm × 2) Zr (125 μm × 2) Zr (20 μm × 20) Ta (100 μm × 2)
KENS	1500°C, Mo, 100∅ × 280 H, 6N	Ta (100 μm × 2) Zr (100 μm × 1) Ta (100 μm × 1) Zr (20 μm × 5) Ta (100 μm × 1)

[a] Diameter and height.

rate was decreased to 400°C/h not to give a severe influence of thermal stress on the block. In HIP process, two blocks enveloped with getter materials of zirconium foils and tantalum slabs were put in the same molybdenum furnace, which was used for test at 1500°C. Then the supersonic diagnostic was performed the HIPed blocks to check the bonding between Ta/W and Ta/Ta. The HIPing gave good results to the blocks with no hole for thermocouple. However, the supersonic diagnostic for the block with a hole suggested that there were some defects around the hole. Additionally, imperfect bond was found after polishing the surface of the block. Then, we re-made the block carefully. The second block with a hole gave better ultrasonic image. Finally, we have adopted the second made block. Fig. 5 shows the four blocks presently fabricated.

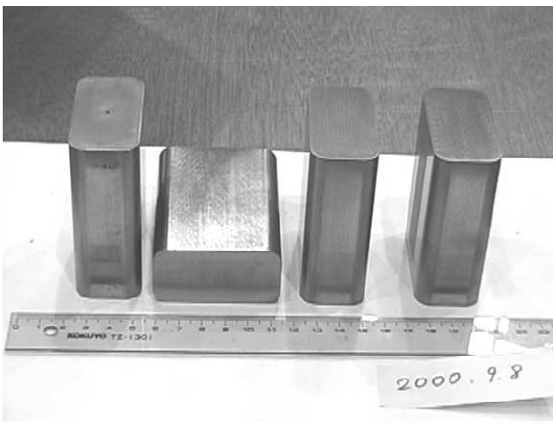

Fig. 5. Polished tantalum-clad tungsten target blocks HIPed using the molybdenum heater keeping at 1500°C and 2000 kgf/cm^2 for 3 h.

5. Results and discussions

The HIPing with gaseous-interstitial-impurity getter material became successful and applied to the KENS target block. However, some problems were also found for the block with the hole for thermocouple. In this section, the main results and discussions are given.

5.1. Vickers hardness and X-ray diffraction analysis

In HIP process, tantalum is very sensitive to gaseous-interstitial-impurities. The result of the HIP process can be judged by measuring the Vickers hardness of tantalum.

In Case 1 processed at 1400°C without zirconium foils nor additive tantalum slab, the Vickers hardness ranged 400–500 HV. In the cap regions at the top and bottom, in the lateral side, the hardness decreased from 750 HV to 400 HV with a depth. The hardness was scattered around 500 HV in tungsten region.

In Case 2, the hardness is nearly constant as shown in Fig. 6: about 130 HV for tantalum and about 500 HV for tungsten. The former is slightly larger than the value of pure tantalum of about 100 HV because of hardening due to working to make the sample. Accordingly, this condition will not give appreciable influence on metal characteristics.

In Case 3 given in Fig. 7, the hardness decreases with a depth from surface of tantalum: 800–400 HV in the bottom zone and 650–160 HV in the lateral side. In the

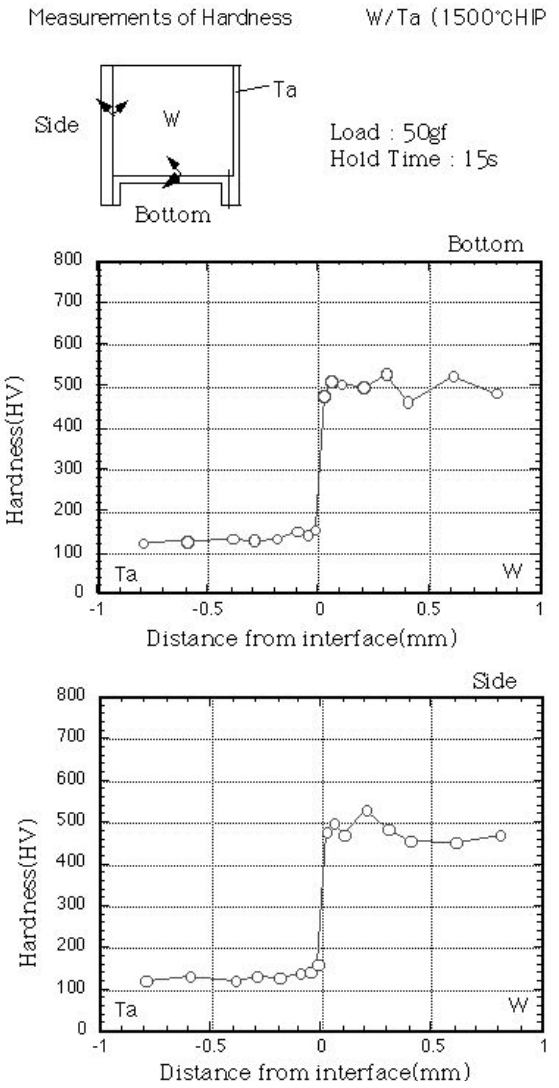

Fig. 6. Vickers hardness distribution of the W/Ta interface for sample HIPed at 1500°C with molybdenum furnace.

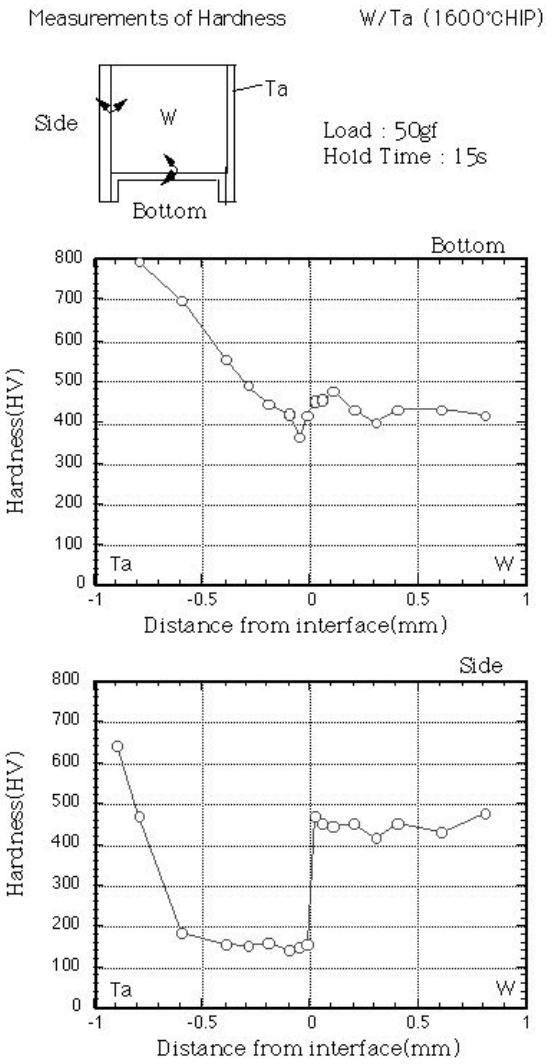

Fig. 7. Vickers hardness distribution of the W/Ta interface for sample HIPed at 1600°C with graphite furnace.

tungsten region, hardness of about 440 HV is slightly less than those of Cases 1 and 2. X-ray diffraction analysis of tantalum layer was made for the sample before and after polishing the surface. It was found that the surface layer is composed of Ta (100), TaC(63) and Ta_2C(17) before polishing and that the interior layer of 638 Hv is composed of Ta (100), TaC(7) and Ta_2C(5). The value in parenthesis is to relative peak intensity of compound to tantalum. The result means that decreasing of hardness with depth reflects a decreased production of chemical compounds of TaC and Ta_2C.

For the two target blocks without a hole, the hardness of tantalum was measured to be 82.3 HV in average. The value is smaller than 90.9 HV for the original plate. It means that the HIPing caused recrystallization and grain growth in tantalum layer.

5.2. Optical microscope observation

It is observed that there are many micro-cracks in tantalum in case of 1400°C. However, there are no micro-cracks in the case of 1500°C. Fig. 8 shows the observation. Grain size of tantalum becomes larger as the temperature is increased. The significant grain growth is observed in tungsten in the case of 1600°C. In general, the recrystallization grain growth softens metallic materials. However, for the group VIa metals such as tungsten and molybdenum it causes embrittlement due to weakening of grain boundaries [6], but not for the

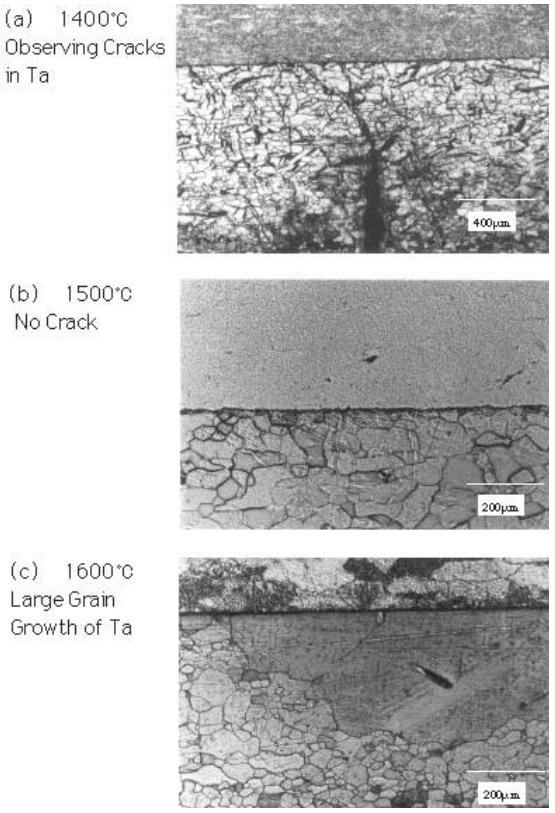

Fig. 8. Optical micrographs of W/Ta Interface. W (upper) and Ta (lower).

group Va metals such as tantalum and niobium. The embrittlement is known as recrystallization embrittlement. Accordingly, the lower temperature is better to avoid the recrystallization and grain growth of tungsten. It should be noted that from the viewpoint of ductility for the group Va metals the recrystallization and grain growth is not bad providing that the detrimental effect of gaseous-interstitial-impurities is negligibly small.

5.3. Diffusion layer of Ta/W boundary

Fig. 9 shows the Ta/W diffusion layer thickness as a function of the HIP temperature. The results measured at Tokyo Tungsten are well fitted by a straight line on the semi-logarithmic graph. It means that the thickness can be described with a single exponential function such as $\exp(-a/T)$; a is constant.

From this figure, the diffusion layer of KENS target processed at 1500°C is assumed to be 3.5 μm.

5.4. Oxygen contents absorbed in getter material

We wrapped the tantalum-clad tungsten specimen with zirconium foils and tantalum slab used as an

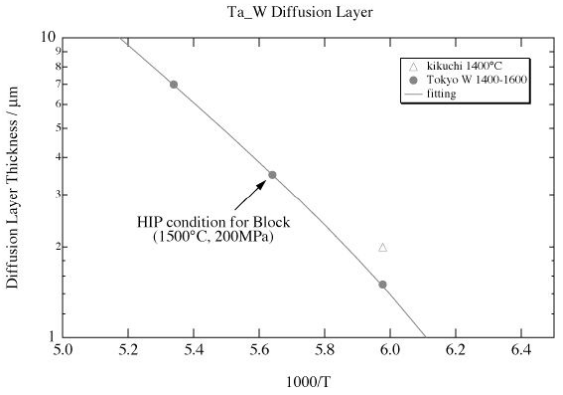

Fig. 9. Diffusion layer thickness of Ta/W interface.

impurity element getter material as listed in Table 1. The getters led to good results. It is very interesting to quantitatively compare the ability of zirconium or tantalum to absorb oxygen as getter materials used for the KENS target block. On the other hand, the most inner tantalum slab must remain the characteristic of the tantalum-clad. Accordingly, the oxygen contents were chemically analyzed in the tantalum and zirconium which were adjacent to each other near the surface and the most inner tantalum slab used for the HIP process of the KENS target block. The measured results of oxygen contents are as follows:

In the surface Ta slab:	0.182 w/% (1820 ppm)
In the surface Zr foil:	0.285 w/% (2850 ppm)
In the inner Ta slab:	0.0102 w/% (102 ppm).

Oxygen contents in the surface tantalum slab seem to be more than an expected value, compared with the value in the surface zirconium foil. The reason is that the tantalum slab enveloping the zirconium foils directly touched the argon gas and absorbed more oxygen than inner zirconium foil. Perhaps, the zirconium is a more effective getter material than the tantalum, because the same weight of zirconium will absorb several times of oxygen than tantalum and the price is much lower.

Oxygen content in the inner tantalum slab is fairly similar to 79.8 ppm of the original tantalum sheet. It is possible that the tantalum slab should absorb by about 10 ppm during work to envelop the target block and actual oxygen absorption during HIPing seems to be quite small; about 10 ppm. Accordingly, tantalum-clad of the block was scarcely affected by impurity oxygen.

5.5. Ultrasonic diagnostic

Ultrasonic diagnostic can be used to determine the bonding state of the tantalum-clad with tungsten block.

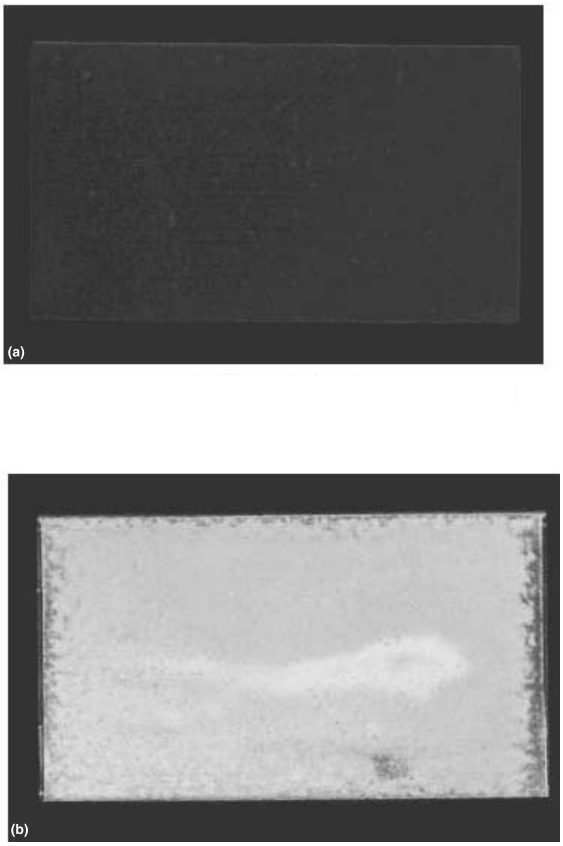

Fig. 10. Ultrasonic echo from the layer near Ta/W interface: (a) block with no hole for thermocouple; (b) first made block with a hole for thermocouple.

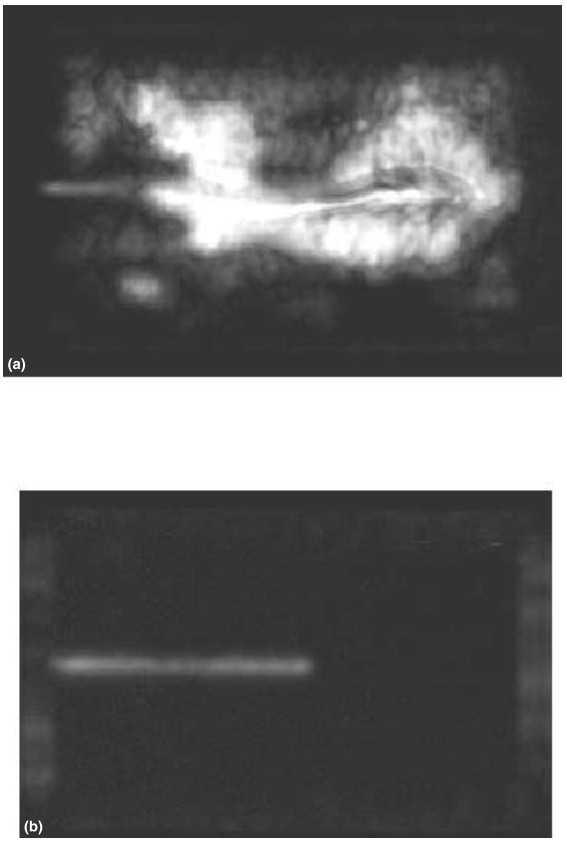

Fig. 11. Ultrasonic echo from the depth of about 14 mm in block with a hole for the thermocouple: (a) first made block; (b) second made block.

Fig. 10 shows the two-dimensional pattern of ultrasonic echo from the layer near tantalum/tungsten interface. The intensity for the block with no hole is very uniform. Accordingly, the HIP process can be said to give a good bonding. However, the echo pattern in the case of the block with a hole has a white shade of a quite unusual shape as shown in Fig. 10(b). Fig. 11 is the echo from the depth of about 14 mm where the pipe was imbedded in the block. It is seen in Fig. 11(a) that there are some kinds of defects around the hole. It is important to clarify the kinds of the defects and the reasons for the occurrence of them. Fig. 11(b) shows that the echo from the secondary made block with a hole. It displays the beautiful pattern of the hole.

6. Conclusion

The tantalum-clad tungsten target block for KENS was successfully fabricated by means of the presently developed HIP process. The optimum HIP condition was determined with the small sample. It should be noted since there are problems such as gaseous interstitial impurity elements in argon gas severely attacking tantalum-clad under high pressure, we have to not only use the very high-purity argon gas but also control the circumstances around the objective so as to make the detrimental effect of the impurity elements negligibly small. Getter material is very effective to meet the aim. Zirconium is more convenient than tantalum, considering oxygen absorbing rate and cost.

Ultrasonic diagnostic clarified that bonding between tantalum and tungsten was good as well as bonding between tantalum layers. However, it is important to clarify the reasons producing the defects in the block with a hole to insert thermocouple for developing the more intense spallation neutron source.

The diffusion layer thickness of the target block is assumed to be 3.5 μm. The value seems to be not so large, but we think that it and the 0.6 mm thick tantalum-clad are enough to protect the tungsten from corrosion by water in KENS operated at 3 kW of beam power. Adding the pressure and keeping the adequate

condition for more time in the HIP process will increase the diffusion layer thickness.

Target blocks have been assembled into the target housing of KENS and installed in the TMRA in September and it will be irradiated in early November in 2000.

Acknowledgements

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