



## Rudimentary investigation of the HIP process for tungsten target

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### ABSTRACT

Zr alloy and 316-Ti stainless steel (S.S.) were selected as cladding materials for a W target. The HIP processes were performed at 1200, 1300 and 1400 °C and 180 MPa. The observation and determination for micro-morphology of the interface, diffusion depth and composition as well as their micro-hardness were conducted. The results indicated that the bonding of W–Zr and W–S.S. was good under the testing conditions. No pores or micro-cracks in the interface were observed. Grain growth of W was not observed at 1200 and 1300 °C, but it was observed at 1400 °C. The diffusion of Zr–W on the interface of W–Zr was preferred during the HIP process. The diffusion layer was 6–13 μm in thickness for W–Zr, and 13 μm for W–S.S. A peak of the hardness was observed at the interface of W–Zr or W–S.S. A part of the stainless steel cladding melted after HIP using an oxygen absorber (Zr) at 1300 °C and 180 MPa. The conditions of 1200 °C and 180 MPa without using an oxygen absorber Zr are suitable for W–S.S. bonding, while 1300 or 1400 °C and 180 MPa are better for W–Zr bonding.

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### 1. Introduction

Two kinds of neutron production targets are currently studied for the future accelerator driven clean nuclear power system (ADS): liquid metal targets and solid metal targets. Tungsten is the most promising solid target material due to its high neutron yield and high strength at high temperature. The compatibility of tungsten and stagnant water without irradiation is good, however, tungsten will be attacked by flowing water [1–3], and machinable tungsten will be embrittled and corroded under irradiation [4]. In this case, another material with high irradiation and corrosion resistance and excellent neutron properties has to be used as the cladding of a solid tungsten target.

In order to increase the life time of a tungsten target, the cladding has to be able to transfer the heat from the W body to the coolant quickly and efficiently. Therefore, any pores or gaps between the W body and the cladding are not desired. The HIP seems to be the most promising and attainable method to eliminate pores or gaps between the W body and the cladding [2,3]. Zr alloy and 316-Ti stainless steel were selected as the cladding materials in this work.

### 2. Preparation of HIP samples

#### 2.1. Chemical composition of testing materials

The chemical composition of the Zr alloy cladding is in wt%: 98.88% Zr, 1.0% Nb, 0.12% O. For the 316-Ti S.S. cladding,

the composition is in wt%: 0.0404 C, 16.8 Cr, 12.5 Ni, 1.67 Mn, 0.73 Si, 3.75 Mo, 0.63 Ti, 0.0078 S, 0.016 P, Fe in balance.

#### 2.2. Size of samples

A polished W-bar of 8 mm in diameter and rotary swaged W-bars of 5 and 8 mm in diameter were used in this work. Both types of W-bars had a purity of 99.9%. Samples of 30 mm length were cut from both W-bars. The cladding of the polished and rotary swaged W-bar of 8 mm in diameter is Zr alloy cladding of 9.6 mm outer diameter and 0.75 mm wall thickness, and the cladding of the rotary swaged W-bar of 5 mm in diameter is S.S. cladding of 6 mm outer diameter and 0.4 mm wall thickness.

#### 2.3. Preparation of samples

The Zr alloy and S.S. claddings were chemically polished to remove oxide scale on the surfaces. The Zr alloy cladding was polished in a solution of 5% HF + 39% HNO<sub>3</sub> + 56% H<sub>2</sub>O at 30–39 °C for 1–2 min. The stainless steel cladding was polished in a solution of 4% HCl + 1% HNO<sub>3</sub> + 0.5% H<sub>2</sub>SO<sub>4</sub> + 5% CH<sub>3</sub>COOH + H<sub>2</sub>O at 80–100 °C for 2 min.

After inserting the W-bars into the corresponding Zr alloy and S.S. cladding tubes, the tubes were sealed by electron-beam welding (EBW). For the S.S. cladding, the EBW was conducted in a vacuum of  $2.66 \times 10^{-2}$  Pa at 60 kV with 6 mA, while for the Zr alloy cladding, it was conducted at  $6.66 \times 10^{-3}$  Pa, 60 kV with 8 mA

### 3. HIP process and parameters

HIP was conducted at Beijing General Research Institute for Non-ferrous Metal. The facility was QIH-6 type HIP machine made in USA.

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**Table 1**  
Parameters of the HIP process for the W–Zr alloy cladding

Temperature (°C)	Pressure (MPa)	Duration (h)	Rate of <i>T</i> increase (°C/h)	Oxygen absorber	Container	Feeding gas
1200	180	4	300	Sponge Zr	Al <sub>2</sub> O <sub>3</sub> crucible with a Zr lid	Ar (5N)
1300						
1400						

**Table 2**  
Parameters of HIP process for W–S.S. cladding

Temperature (°C)	Pressure (MPa)	Duration (h)	Rate of <i>T</i> increase (°C/h)	Oxygen absorber	Container	Feeding gas
1200	180	4	300	No	Al <sub>2</sub> O <sub>3</sub> crucible without lid	Ar (5N)
1300				Sponge Zr	Al <sub>2</sub> O <sub>3</sub> crucible with a Zr lid	



**Fig. 1.** Samples after HIP processing. (A) W–Zr sample, at 1300 °C, (B) W–S.S. sample, at 1200 °C, (C) W–Zr sample, at 1400 °C.

The prepared W–Zr and W–S.S. cladding samples were individually put into an Al<sub>2</sub>O<sub>3</sub> crucible which was placed in the furnace of HIP machine with a graphite heater. In order to prevent the oxidation of Zr alloy and S.S. cladding, argon (Ar) gas with purity of 99.999% (5N) was used as a feeding gas, and sponge Zr was put in Al<sub>2</sub>O<sub>3</sub> crucible as an oxygen absorber. The cladding samples were covered up with the sponge Zr, and a Zr piece was used as the lid of the Al<sub>2</sub>O<sub>3</sub> crucible.

The furnace was degassed by pumping to about 1–2 Pa, and then fed with the pure Ar gas. The degassing and feeding were repeated once more. Afterwards, HIP process was started by increasing the pressure up to 50 MPa, then increasing the temperature up to the desired temperature with a rate of 300 °C/h, and finally adjusting the pressure to 180 MPa. The HIP'ing pressure was held for 4 h.

The HIP process for the W–S.S. cladding samples was conducted at 1200 °C without an oxygen absorber.

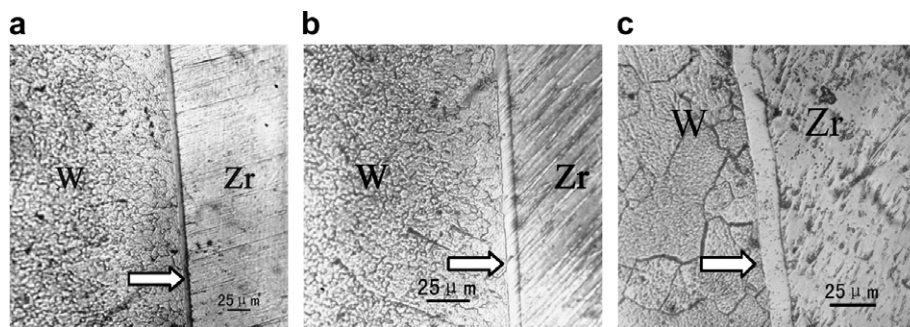
The parameters of the HIP process for the W–Zr alloy cladding are shown in Table 1, and those for W–S.S. cladding are listed in Table 2. The samples after HIP processing are shown in Fig. 1.

## 4. Results and discussion

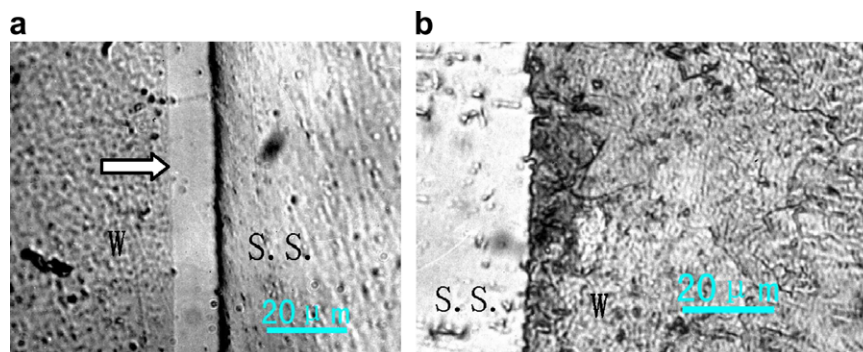
### 4.1. Micro-morphology of interface

#### 4.1.1. W–Zr cladding

The micro-morphologies of the W–Zr bonding interfaces are shown in Fig. 2. The optical microscopy observation indicates that there are no pores or micro-cracks in the W–Zr interfaces. At testing temperatures of 1200 °C and 1300 °C, grain growth in the tungsten is not observed, but it is obvious at the fabrication temperature of 1400 °C. There is a band with dark color on the Zr alloy side of the interface, the EDS analysis indicates that the main composition of the dark band is Zr, it proved that the dark band is resulted from the grinding during the preparation of the metallog-



**Fig. 2.** Micro-morphologies of the W–Zr bonding interfaces after HIP'ing at (a) 1200 °C (b) 1300 °C (c) 1400 °C.



**Fig. 3.** Micro-morphologies of the W–S.S. bonding interface after HIP'ing (a) at 1200 °C without Zr and (b) at 1300 °C containing Zr.

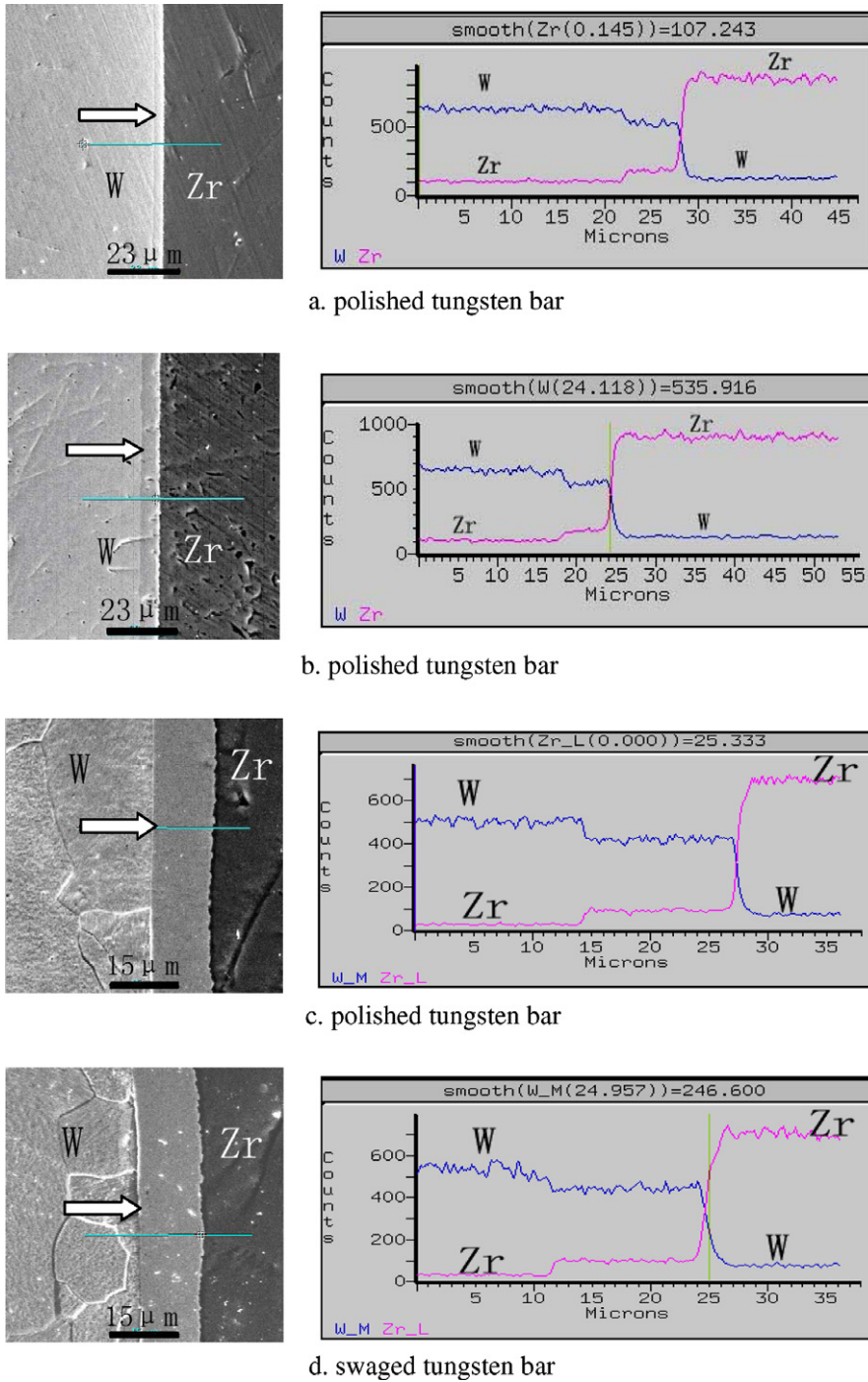


Fig. 4. Line scanning analysis of the composition and thickness of diffusion layers at the W-Zr bonding interfaces.

raphy specimens, because the hardness between W and Zr alloy is significantly different.

4.1.2. W-stainless steel cladding

The micro-morphologies of the W-S.S. bonding interfaces are shown in Fig. 3. The micro-morphologies observed by optical microscopy indicate that there are no pores or micro-cracks in the W-S.S. interfaces, and no grain growth of tungsten either at the testing temperatures of 1200 and 1300 °C. There is a dark band on the stainless steel side of the interface shown in 3(a), it may also be resulted from the grinding during preparation of the metallography specimens, because the hardness of W and stainless steel is rather different. After HIP'ing, the specimen containing an oxygen

absorber (Zr) and processed at 1300 °C showed that part of stainless steel cladding was melted. It may have resulted from the reaction between Fe and Zr, and the formation of FeZr<sub>2</sub> compound whose melting point is lower than 1000 °C [5]. Obviously, Zr should not be used as the oxygen absorber in the HIP process for W-S.S. cladding.

Table 3  
Diffusion layer depths at the W-Zr interface

HIP temperature (°C)	1200	1300	1400	1400 (Swaging W)
Diffusion layer depth (m)	6	9	13	13

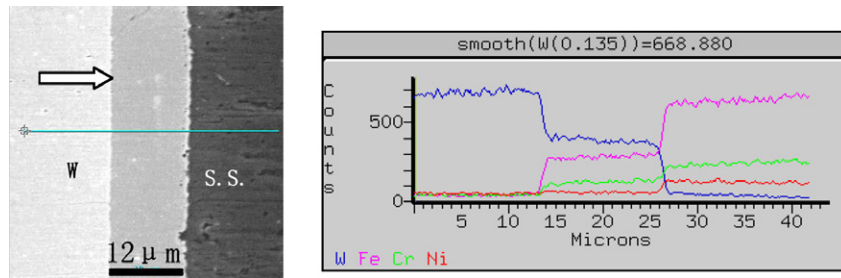


Fig. 5. Line scanning analysis of the composition and thickness of the diffusion layer at the W–S.S. bonding interface After HIP'ing at 1200 °C.

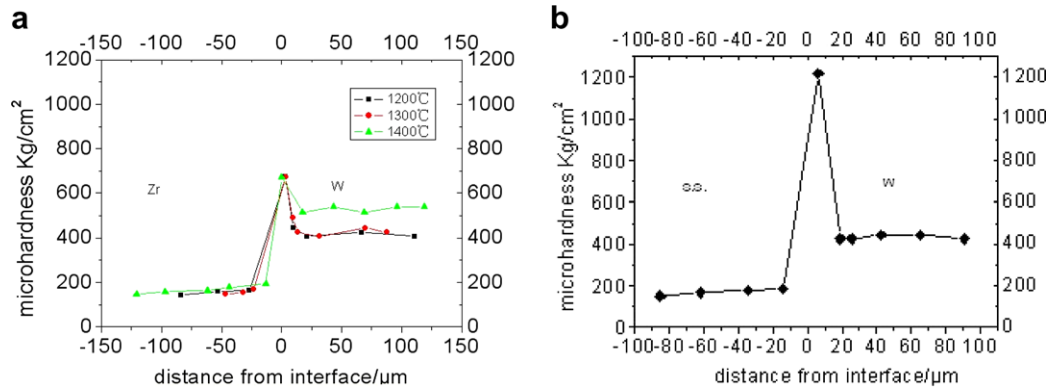


Fig. 6. Micro-hardness at the interfaces of the W-claddings: (a) W–Zr bonding and (b) W–S.S. bonding (1200 °C).

#### 4.2. Composition and thickness of diffusion layer at interface

The line scanning analysis of the composition of the diffusion layer on the interface of the W–Zr bond is shown in Fig. 4. It can be seen that the W content decreases while the Zr content increases on the W side of the interface, and the diffusion depth of W in Zr is very small. It means that the diffusion of Zr to W during the HIP process is preferred. The thickness of diffusion layers measured by line scanning is presented in Table 3.

It can be also seen from 4(c) and (d) that the oxide scale on the surface of the swaged tungsten bar has no influence on the diffusion layer thickness at 1400 °C.

The line scanning analysis of the diffusion layer on the interface of the W–S.S. specimen is shown in Fig. 5. It is clear that the diffusion of W and Fe is obvious. The diffusion depth is about 13 μm.

#### 4.3. Micro-hardness at interface

The measurement results of the micro-hardness for the W–Zr alloy and W–S.S. bonding are shown in Fig. 6. It shows a peak of hardness on the diffusion layer for both W–Zr and W–S.S. bonding. This may have resulted from the formation of the Zr–W and Fe–W intermetallic compounds respectively in the diffusion layers. It can be seen from the binary alloy equilibrium diagrams of Zr–W and Fe–W that  $W_2Zr$  can be formed below 2210 °C, and  $\delta FeW$  can be formed at 1190–1215 °C [5], and the hardness is very high.

### 5. Conclusions

In the present work, the bonding of W with Zr alloy or S.S. was studied after HIP processing at different temperatures. Metallography and hardness measurements were performed to inspect the quality of the bonding. The results showed the following:

(1) The bonding of W–Zr and W–S.S. was good under the testing conditions. No pores or micro-cracks were observed at the

interface; grain growth of W was not observed at testing temperatures of 1200 °C and 1300 °C, but was observed at 1400 °C. After HIP'ing at 1300 °C, a part of the stainless steel cladding melted in a W–S.S. sample containing an oxygen absorber Zr. This implies that Zr should not be used as an oxygen absorber for W–S.S. bonding during HIP'ing.

(2) The diffusion depth of W to Zr was small, while the diffusion of Zr toward W in HIP processing was obvious. The depth of diffusion layer under the present testing conditions were 6–13 μm, and increased with the HIP temperature. The diffusion of W to S.S. and Fe to W was almost equivalent for the bonding of W–S.S., the depth of diffusion layer was about 13 μm at 1200 °C.

(3) A peak of the hardness was observed on diffusion layers for both W–Zr and W–S.S. bonding. It might have resulted from the formation of the Zr–W and Fe–W metal compounds in the diffusion layers.

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