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Compatibility between $Be_{12}Ti$ and SS316LN

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Abstract

Beryllides have good properties and are one of the candidate materials for the neutron multiplier of the DEMO reactor in which good performance is required at 600–800 °C. Therefore, a good compatibility with structural materials is expected for the beryllides. In this study, a compatibility test between $Be_{12}Ti$ and SS316LN was carried out as first step to evaluate the compatibility between beryllides and structural materials. The thickness of the reaction layer between $Be_{12}Ti$ and SS316LN at 800 °C after 1000 h was approximately 30 μm, whereas that of beryllium metal was 300 μm. At 600 °C after 1000 h, the thickness of the reaction layers as to $Be_{12}Ti$ and Be was less than 10 and 100 μm, respectively. The compatibility between $Be_{12}Ti$ and SS316LN was evaluated and $Be_{12}Ti$ was perfectly better than that between beryllium metal and SS316LN at high temperature (600–800 °C). The advantage of beryllides as a neutron multiplier in the Demo reactor was proved.

1. Introduction

Beryllium is used in the tritium breeding blanket as a neutron multiplier. It is expected that the operation temperature range of this blanket is 450–750 °C. Under the conditions of high temperature and long heating period, the chemical interaction between beryllium and structural material is also very important. From many investigations, it appeared that the reaction between beryllium and austenitic stainless steel occurred above 600 °C and BeNi is formed at 600 °C [1].

Flament et al. [2,3] carried out compatibility tests between beryllium and 1.4914 ferritic steel without nickel in vacuum. It was indicated that the reaction between beryllium and 1.4914 ferritic steel was less in comparison with SS316. Hofmann and Dienst [4] carried out compatibility tests between beryllium and AISI316 under argon atmosphere. It was indicated that a chemical reaction occurred above 600 °C, and that beryllium diffused into SS316 and BeNi was observed by AES and IMA. However, the identification of reaction products was not conducted. Terlain and Herpin [5] carried out compatibility tests between beryllium and 1.4914 ferritic steel. A small chemical interaction was observed at 650 °C after 1500 h annealing, thickness of the single phase reaction layer was 8 μm.

Kawamura et al. [6] investigated the compatibility of beryllium with ferritic steel (F82H). The reaction layer was formed on the F82H side above 600 °C. The reaction layer thickness on SS316 was about 1.5 times larger than that in the F82H at 800 °C after annealing for more than 300 h. The reaction products were formed on the F82H side. The reaction product, Be_2Fe was identified at 600 °C by X-ray diffraction (XRD), and Be_2Fe and $Be_{11}Fe$ were identified by XRD at 800 °C. A reaction product between beryllium and chromium was not observed. The total reaction layer increased linearly with the square root of annealing time. From the quantitative analysis of the reaction layer, it was divided into two different layers in which the atomic concentrations of beryllium and iron were almost constant. It was observed that the segregation was formed near the reaction layer at 800 °C annealing. It was divided into two types; the first was a tungsten segregation zone near the reaction layer, and the other was a chromium segregation zone in F82H bulk.

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On the other hand, beryllides have good properties (high melting point, etc.) and are one of the candidate materials for the neutron multiplier of the DEMO reactor in which good performance is required at 600–800 °C. Therefore it is expected that beryllides have good compatibility with the structural materials. However, there are no data for the compatibility between beryllides and the structural materials.

In this study, the compatibility test between $Be_{12}Ti$ and SS316LN was carried out as first step to evaluate the compatibility between beryllides and structural materials, especially the compatibility of $Be_{12}Ti$ and beryllium with SS316LN.

2. Experimental

2.1. Specimens

2.1.1. $Be_{12}Ti$

$Be_{12}Ti$ specimens were fabricated by the HIP process from beryllium and titanium powder, were machined to disks that were approximately $\varnothing = 8$ mm, $t = 2$ mm and were mirror-polished on the surface. These $Be_{12}Ti$ specimens were made by NGK Insulators Ltd. The detailed fabrication process is shown in Fig. 1. The chemical composition of $Be_{12}Ti$ specimens is shown in Table 1. The main impurity of the $Be_{12}Ti$ specimen was beryllium oxide (1.32 wt%). The surface of the beryllide specimens was washed ultrasonically with acetone.

2.1.2. Beryllium metal

The beryllium specimens were hot-pressed beryllium disks made by NGK Insulators Ltd. The specimen

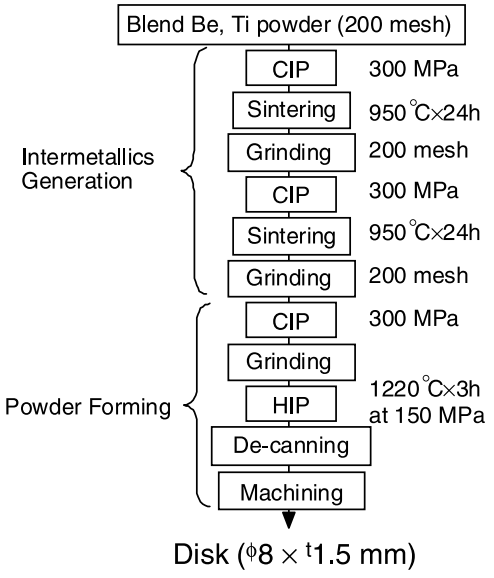

Fig. 1. Detailed fabrication process of $Be_{12}Ti$.

Table 1
Chemical compositions of the materials

Material (wt%)	Element															
	Be	BeO	Mg	Al	Si	Ti	Cr	Fe	Co	Ni	Cu	W	C			
$Be_{12}Ti$	Balance	1.32	0.001	0.014	0.025	33.4	0.003	0.044	0.047	<0.001	0.004	< 0.01	0.026			
Be	Be	BeO	Mg	Al	Si	Fe	C									
	>99.0	<1.0	<0.06	<0.06	<0.06	<0.08	<0.06									
SS316LN	Fe	C	Mn	Ni	Cr	Mo	N	P	S	Si	Ta	Ti	Co	B		
	Balance	0.0225	1.80	12.25	17.50	2.50	0.07	0.025	0.008	0.50	0.01	0.15	0.05	0.001		

dimensions were 10.0 mm diameter and 1.0 mm thickness. The purity of the beryllium specimens was almost 99 wt%. The chemical composition of beryllium specimens is shown in Table 1. The main impurity was beryllium oxide. Hot-pressed beryllium was wrapped with mild steel (SS41), rolled at 900 °C, annealed at high temperature, and then polished until shining condition by 1 μm diamond paste on buff cloth. The surface of beryllium specimens was washed ultrasonically with acetone.

2.1.3. SS316LN

The chemical composition of the SS316LN specimens is shown in Table 1. The specimen dimensions were 10.0 mm diameter and 1.0 mm thickness. The SS316LN specimens were polished until shining condition, and cleaned by ultrasonic washing with acetone.

2.2. Testing procedure

The outline of the capsule is shown in Fig. 2. Diffusion couples for this test consisted of the $Be_{12}Ti$/SS316LN or Be/SS316LN. They were inserted into a Zry-2 capsule with a tungsten spring to maintain the contact pressure (32.4 N/contact area) at the interface. A helium leak test was carried out on the assembled capsule, and the leakage rate was less than 1.0×10^{-9} Pa m^3/s. High purity helium gas (99.9999wt%) was sealed into the capsule by TIG welding. The compatibility tests were carried out at 600, 700 and 800 °C by annealing in an electric furnace under vacuum conditions. Annealing times were 100, 300 and 1000 h. As for beryllium metal, the same tests were carried out for $Be_{12}Ti$. After annealing, the interaction in the diffusion couple was evaluated.

As for the evaluation, the contacting surface of each specimen was observed visually. XRD was performed to identify the reaction products on the near surface region. The microstructure of the reaction layer was observed in the cross section by scanning electron microscopy (SEM). The composition of the layer was identified by electron probe microanalysis (EPMA). Here, the accelerating voltage was 15 kV and the electron beam diameter was about 1 μm.

3. Results and discussion

3.1. Observation of appearance

From the visual observation of the specimens after annealing, the marks of the chemical interaction were investigated visually. The results are shown in Fig. 3. It was observed that the reaction marks of the contacting surface of $Be_{12}Ti$/SS316LN were heavier than those of Be/SS316LN at any temperature and annealing time.

3.2. Identification of reaction products

On the $Be_{12}Ti$ and Be sides, reaction products were not found by XRD at 600, 700 and 800 °C up to 1000 h annealing conditions. The results of XRD on the SS3116LN side are shown in Fig. 4.

BeNi was identified above 600 °C as reaction product on the surface of the SS316LN side in contact with $Be_{12}Ti$. However, $Be_{11}Fe$ was not identified up to 800 °C. BeNi and Be_2 Fe were identified at 600 and 700 °C as reaction products on the surface of the SS316LN side in contact with Be (see Fig. 4). At 800 °C, $Be_{11}Fe$ was identified instead of BeNi and Be_2Fe though Be–Ni phases are more stable than Be–Fe phases. It is considered that Be–Ni might not be detected by XRD because $Be_{11}Fe$ layer forms as thick as it shuts X-ray off.

3.3. Observation of the reaction layer

On the $Be_{12}Ti$ and Be sides, reaction layers were not found by SEM observation at 600, 700 and 800 °C up to 1000 h annealing conditions. The results of the SEM observation are shown in Fig. 5. The thickness of the reaction layer was measured. The results are shown in Fig. 6. The thickness of reaction layer between $Be_{12}Ti$

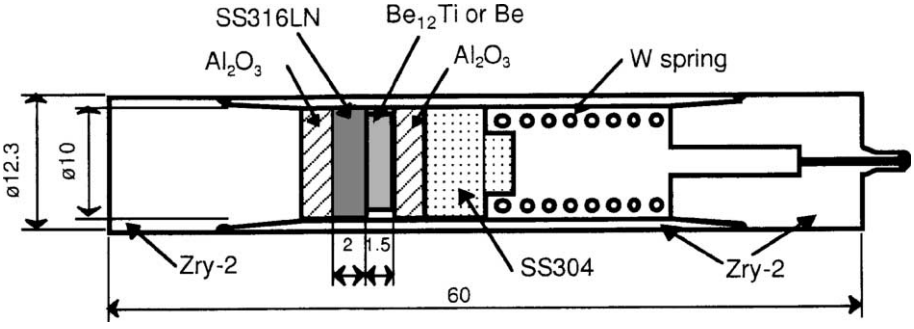

Fig. 2. Outline of the capsule, dimensions in mm.

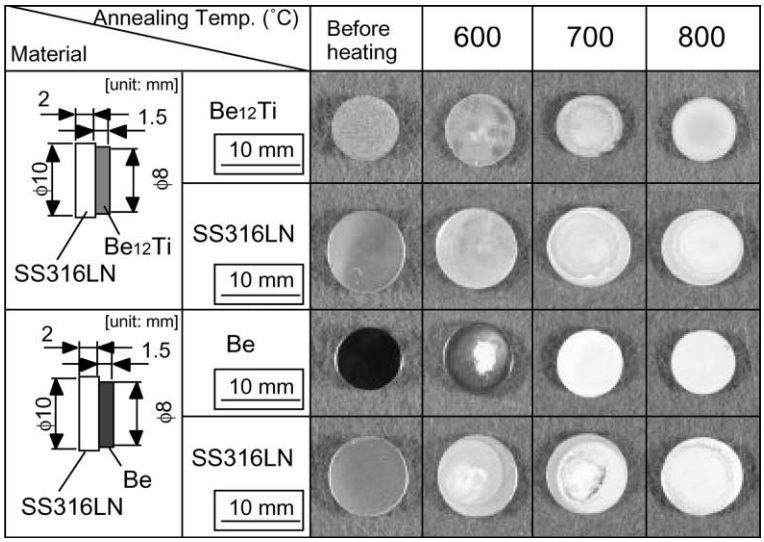

Fig. 3. The results of visual observation for the contacting surface after annealing at 1000 h.

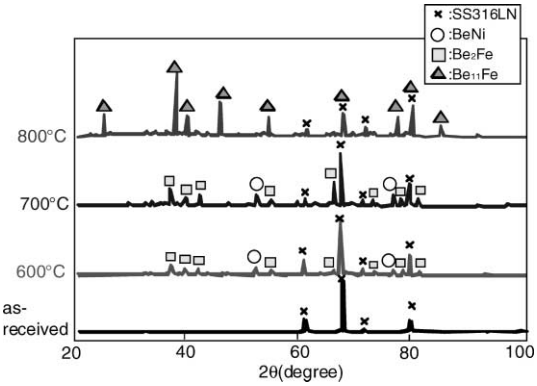

Fig. 4. XRD profile on the surface of SS316LN after annealing.

and SS316LN at 800 °C after 1000 h was approximately 30 μm, whereas that of beryllium metal was 300 μm. At 600 °C for 1000 h, each thickness of reaction layer as to $Be_{12}Ti$ and Be was less than 10 and 100 μm, respectively. It was obvious that the compatibility between $Be_{12}Ti$ and SS316LN was much better than that between Be and SS316LN.

3.4. Analysis of layer structure

A quantitative line analysis was performed for the typical phase that was observed by SEM. The results were shown in Fig. 7. It was observed that a large variation of Be content varied only in 30 μm and a small variation was observed up to 50 μm. The Fe content was low in the region where Be was high. In case of Be/SS316LN, the Be and Fe contents varied up to 330 μm, and Be became 0% and Fe became 80% that was the original content of SS316LN in the thicker region. From the distribution of each element, it was considered that three layers existed.

To determine the material of each layer, a quantitative point analysis was performed. As for the reaction layer of $Be_{12}Ti$/SS316LN, the first layer with gray color on the surface was Be_2Fe, and small black particles, gray phases and the matrix in the second layer were Be_2Fe, BeNi and SS316LN, respectively. As for the reaction

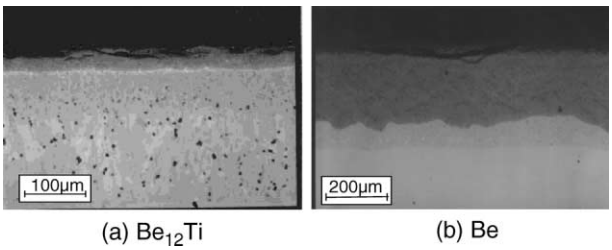

Fig. 5. SEM observation of the reaction layer in SS316LN at 800 °C after 1000 h.

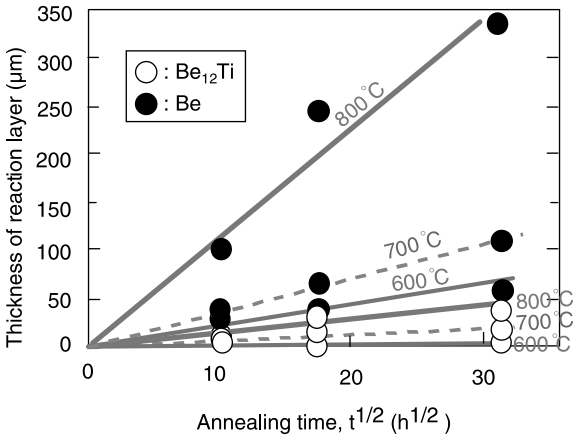

Fig. 6. Change of the thickness of the reaction layer with annealing time.

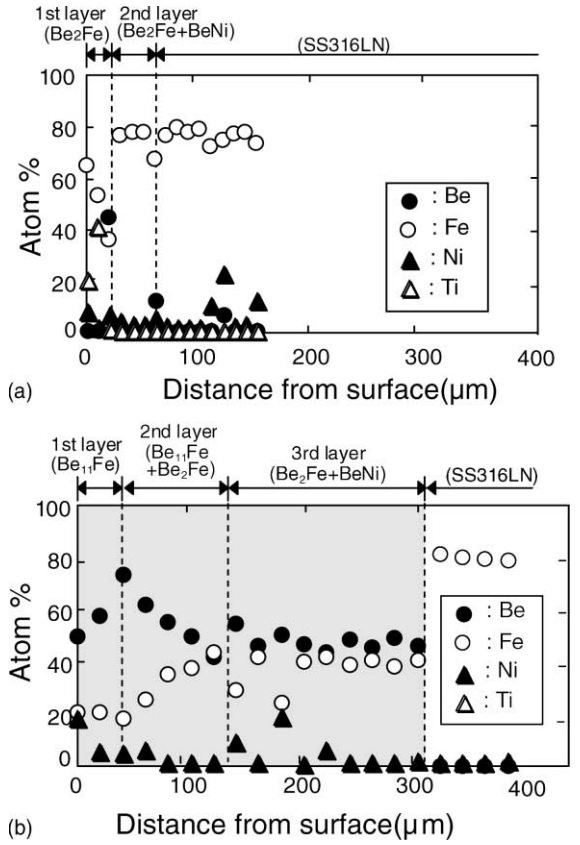

Fig. 7. Distribution of Be, Fe, Ni and Ti in the reaction layers at 800 °C after 1000 h.

layer of Be/SS316LN, the first layer with black color was $Be_{11}Fe$, and the gray part of the second phase was Be_2Fe. Small black particles, gray phases and the matrix in the third layer were Be_2Fe, BeNi and SS316LN, respectively.

From the analysis and the discussion, the structure of the reaction layer was made clear, it is described in Fig. 7. The reaction layer on $Be_{12}Ti$ consists of two layers that were Be_2 Fe and Be_2Fe + BeNi. The reaction layer on Be consists of three layers that were $Be_{11}Fe$, $Be_{11}Fe$ + Be_2Fe and Be_2Fe + BeNi. No $Be_{11}Fe$ formation was observed in case of $Be_{12}Ti$. There are concentration distributions from 60 to 80 at.% in the $Be_{11}Fe$ layer 80%, and the cause was not clear.

It was considered that the binding energy between Be and Ti in the lattice of $Be_{12}Ti$ was higher than that in Be. And it was hard for Be element to diffuse into SS316LN. And it was hard to form $Be_{11}Fe$ since the concentration of Be in the surface is not high enough for the $Be_{11}Fe$ formation.

4. Conclusion

The compatibility tests and the following results showed the big advantage of $Be_{12}Ti$ as the material for high-temperature use:

- The thickness of the reaction layer between $Be_{12}Ti$ and SS316LN was much smaller that that between Be and SS316LN. The thickness at 800 °C was one tenth of that for beryllium.
- The structure of the reaction layer between $Be_{12}Ti$ and SS316LN showed characteristic differences from that for Be. $Be_{11}Fe$ was not observed in the layer between $Be_{12}Ti$ and SS316LN.

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