



Surface reaction of titanium beryllide with water vapor

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Abstract

Beryllium is one of the candidate materials for the neutron multiplier in the tritium-breeding blanket. However, there are some problems related to the application of beryllium as a neutron multiplier, which include compatibility with structural materials, tritium inventory and reaction of beryllium with water vapor and oxygen in a LOCA accident. Titanium beryllides such as Be_{12}Ti are known to have advantages over beryllium from the perspectives of higher melting point, lower chemical reactivity and lower swelling. Thus, these materials are promising alternatives of beryllium. The authors investigate here the reaction of titanium beryllides with water vapor at high temperatures, and it is found that Be_{12}Ti is by far more tolerant to water vapor than beryllium. To clarify the high tolerance of Be_{12}Ti to water vapor, the surface of Be_{12}Ti used in the experiment was investigated by means of digital microscope, SEM, XRD and electron spectroscopy for chemical analysis (ESCA). These analyses suggest that some oxidized state of beryllium was formed on the surface of Be_{12}Ti exposed to water vapor. In particular, the results of the ESCA analysis gave some clues to understanding the higher tolerance of Be_{12}Ti to water vapor at high temperatures.

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1. Introduction

Beryllium is one of the candidate materials for the neutron multiplier in the tritium-breeding blanket. Beryllium in this capacity would be placed in the high neutron flux and temperature environment, which leads

to problems related to compatibility with structural materials, tritium inventory and reaction of beryllium with water vapor and oxygen. Titanium beryllides such as Be_{12}Ti have advantages over beryllium from the perspectives of higher melting point, lower chemical reactivity and lower swelling. Thus, these materials are promising alternatives of beryllium as a neutron multiplier. With regard to the reaction with water vapor, beryllium is highly reactive at high temperatures [1–3]. Here, the authors investigate the reaction of titanium

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beryllides with water vapor at high temperatures [4]. The experimental results reveal that Be_{12}Ti is highly tolerant to water vapor. To clarify the mechanism leading to such high tolerance, the surface of Be_{12}Ti used in the experiment was examined by means of digital microscope, scanning electron microscope (SEM), X-ray diffraction analysis (XRD), electron spectroscopy for chemical analysis (ESCA).

2. Experimental procedure

A sample disk of Be_{12}Ti (7.9 mm in diameter and 1.4 mm in thickness) was prepared by NGK INSULATORS LTD. The sample disk was placed in the reactor made of quartz. Argon gas containing H_2O was introduced to the reactor. Water vapor was generated by introducing H_2/Ar gases into a CuO bed at 350°C . The concentrations of H_2 and H_2O in outlet stream of the reactor were measured with a quadrupole mass spectrometer. More details of the experiment are given in literature [4]. After the experiments described above, the surface of the sample was observed using various techniques.

3. Results and discussion

The authors first tested the chemical stability of Be_{12}Ti under argon gas flow containing 0.096% of H_2O . The reactor temperature was raised up to 940°C at the constant rate of $10^\circ\text{C}/\text{min}$. The result of the experiment reveals that a breakaway reaction, which takes place on Be metal ($\text{Be} + \text{H}_2\text{O} \rightarrow \text{BeO} + \text{H}_2$), did not take place on the surface of the Be_{12}Ti disk. The sample was exposed to water vapor for 220 min. The authors next investigated the chemical stability of Be_{12}Ti under water vapor of 1%. Fig. 1 shows changes in the concentration of H_2 in outlet stream of the reactor when the argon gas containing 1% of H_2O was introduced. The generation of H_2 started at a temperature near 600°C . The concentration of H_2 in the outlet stream of the reactor reached a peak at 1000°C and then began to decrease. The generation of H_2 terminated at 10 h. Again, more details of the experimental results have been reported in previous literature [4].

After exposure to 1% of H_2O , the color of the sample surface turned to white, indicating that the sample

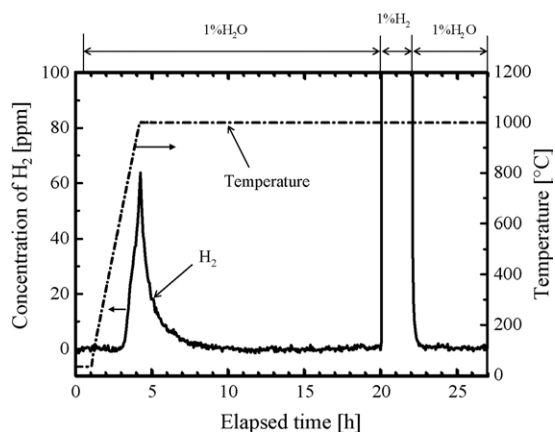


Fig. 1. Change in the concentration of H_2 in the outlet stream of reactor with a Be_{12}Ti disk exposed to 1% $\text{H}_2\text{O}/\text{Ar}$ gas (flow rate: $84.9\text{ cm}^3/\text{min}$ (standard temperature pressure)).

surface was oxidized. The surface roughness of the sample was measured using the digital microscope, which suggests that the roughness increased after exposure to water vapor. The sample surfaces were observed by SEM as well. The surface of the sample exposed to 0.096% is characterized by the presence of lots of white spots with the size of several tens of microns. Such spots are not observed on the surface of the sample exposed to 1% of H_2O . However, the photographs taken with higher resolution indicate that the surface of the sample exposed to 1% of H_2O is rougher. Once again, more details of the above observation have been reported in literature [5].

Questions remained as to why Be_{12}Ti is so highly tolerant to oxidation by water vapor. Thus, the authors performed the XRD analysis using a X-ray diffractometer (RINT-1400 by RIGAKU Inc.). Fig. 2 shows X-ray diffraction patterns. The diffraction pattern of the sample as received (Fig. 2a) shows several diffraction peaks, most of which were attributed to Be_{12}Ti using the JCPDS file. The other peaks were attributed to disproportionated phases such as $\text{Be}_{10+x}\text{Ti}$. Fig. 2(b) shows the diffraction pattern of the sample exposed to 0.096% of H_2O . Comparison of the results in Fig. 2(a and b) suggests a small difference in the patterns at 41° , which was attributed to beryllium oxide. Fig. 2(c) shows the diffraction pattern of the sample exposed to 1% of H_2O , indicating that one high peak arose at 41° . Additionally, new lower peaks arose. The new

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