Journal of Alloys and Compounds 585 (2014) 63–68

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

Preliminary synthesis and mechanical property of titanium beryllide pebbles with different chemical compositions

Jae-Hwan Kim *, Masaru Nakamichi

Breeding Functional Materials Development Group, Division of Blanket Research and Development, Fusion Research and Development Directorate, Japan Atomic Energy Agency, 2-166 Oaza-Obuchi-Aza-Omotedate, Rokkasho-mura, Kamikita-gun, Aomori 039-3212, Japan

ARTICLE INFO

Article history:
Received 30 August 2013
Received in revised form 15 September 2013
Accepted 16 September 2013
Available online 25 September 2013

Keywords:
Advanced multiplier
Plasma sintering
Beryllides
Pebble
Rotating electrode method

ABSTRACT

Beryllide is well-known as the most promising candidate for advanced multipliers in demonstration fusion reactors. However, due to its brittleness, it is difficult to fabricate a pebble-type beryllide. Here, we report the synthesis and analysis of beryllide pebbles with different chemical compositions. In order to clarify the effect of the content of Ti on the fabrication of beryllide pebbles, a rotating electrode method was applied using a plasma-sintered beryllide rod jointed by two beryllide blocks and powder. The beryllide pebbles were successfully fabricated accompanied by phase changes induced by re-melting during the rotating electrode method. The SEM images of the rods and the cross-section of the pebbles demonstrated that the phases of the plasma-sintered beryllide rods with 6 at.%, 7 at.%, and 7.7 at.% Ti were transformed to the peritectic $Be_{12}Ti$ phase with a $Be_{17}Ti_2$ phase due to re-melting whereas pebbles with 3 at.% and 5 at.% Ti showed similar phases as the plasma-sintered beryllide rods. Additionally, the dependence of the Ti amount on crush load depicted that the pebbles with the larger area fraction of Be indicated the higher crush load as well as longer displacement because the Be contributed to increase of ductility in the pebbles.

1. Introduction

Beryllium and its intermetallic compounds have been extensively investigated as a neutron multiplier of fusion reactor blankets. In a fusion reactor, one neutron interacts with beryllium to generate two neutrons resulting in an improved tritium breeding ratio (TBR). This neutron multiplier is supposed to be loaded in the blanket as pebble type because of higher packing density as well as reduction of cracking due to thermal stress. To prevent degradation of the neutron multiplication, the packing density and spherical size of the neutron multiplier are important factors and are generally 68% and in the range of 0.2–2 mm, respectively. For granulation of beryllium, conventional methods such as the rotating electrode method (REM), Mg reduction method (MRM), and gas atomization method have been suggested [1]. Among them, the REM is the most attractive from the viewpoints of impurity control and mass fabrication [2]. Although many studies concerning designs of the component of the blanket have been conducted, few reports on beryllide pebbles have been published. This is because it is considerably difficult to produce beryllide pebbles due to their brittleness.

As an advanced neutron multiplier in DEMO reactors, pebble type of the beryllides has been investigated in Japan and the EU in the DEMO R&D of the International Fusion Energy Research Centre (IFERC) project as part of Broader Approach (BA) activities from 2007 to 2016. Our group [3–5] has reported the successful fabrication and property evaluation of not only disk-type but also rod-type beryllide simply by using a plasma sintering method prior to granulation of beryllide using the REM. Recently, Nakamichi et al. [6,7] reported that not only trial fabrication of beryllide pebbles with 7.7 at.% Be but homogenization to $Be_{12}Ti$ was successfully carried out.

In the present study, we investigated the effect of the content of Ti on the synthesis and mechanical property of beryllide pebbles using plasma-sintered beryllide rods and the rotating electrode method.

2. Materials and methods

For the granulation of beryllide with a diameter of approximately 1 mm, a conventional rotating electrode method (REM) was applied [6]. Prior to the REM, all specimens were prepared by a plasma sintering method [8,9]. To investigate the effect of the content of Ti on the fabrication of beryllide pebbles, the powder composition was varied among 3 at.%, 5 at.%, 6 at.%, 7 at.%, and 7.7 at.% Ti. Considering that it is necessary to shorten the sintering time and lower the sintering temperature for mass production, the plasma sintering temperature used in the experiments was 1073 K while the sintering times were 2 min and 30 s with heating and cooling rates of 100 and 200 K/min, respectively. After the sintering, small rods with lengths

---

* Corresponding author. Tel.: +81 071 71 6537; fax: +81 175 71 6502.
E-mail address: kim.jaehwan@jaea.go.jp (J.-H. Kim).

0925-8388/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.jallcom.2013.09.103

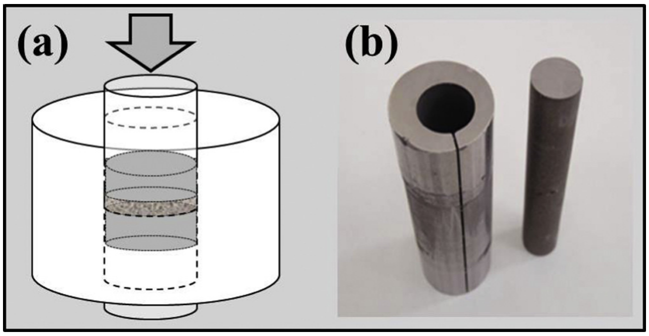


Fig. 1. (a) Schematic of the graphite punch and die used for the fabrication of beryllide rods by jointing and a (b) specimen cut by wire discharge.

Table 1
Conditions applied in the rotating electrode method.

Classification	Conditions
Arc current	80 A
Atmosphere	He gas (0.1 MPa)
Rotational speed	6000 rpm
Rod diameter	10 mm

and diameters of about 25 mm and 20 mm, respectively, were polished and jointed to 60 mm long rods by a plasma sintering method applying the same sintering conditions, as shown in Fig. 1(a).

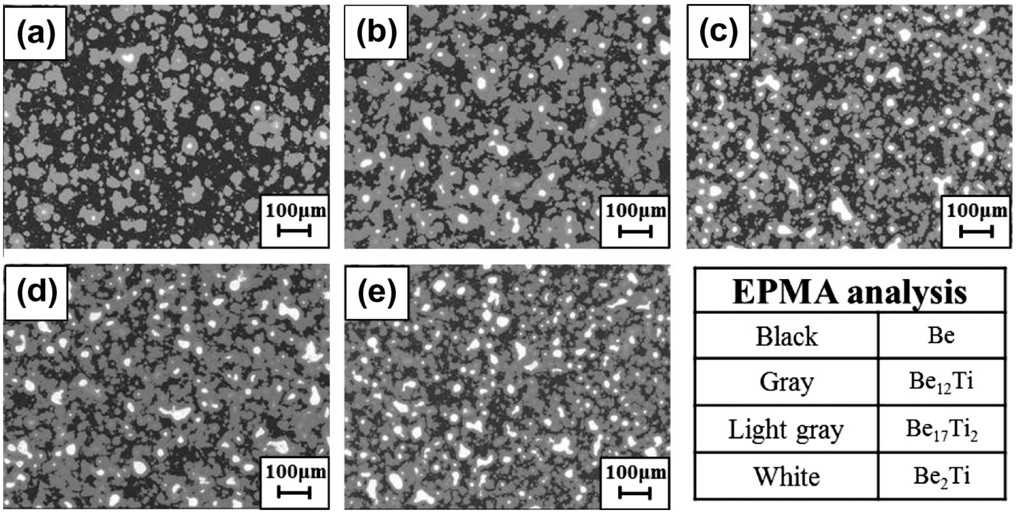
After the joint, the final rod was machined by wire discharge (FANUC, irobot-0), as shown in Fig. 1(b), and set on the rotating electrode machine [6]. The conditions of the REM are given in Table 1. To compare the phase compositions before and after the granulation, scanning electron microscope images of the surfaces and cross-sections were obtained by using an electron probe micro analyzer (EPMA, JXA-8530F, JEOL, Japan). Additionally, X-ray diffraction measurements (XRD, Rigaku, UltimaIV, Japan) were carried out for qualitative analysis. In order to understand the mechanical property of the beryllide pebble, compression test (Autograph, AG-X10kNX, Shimadzu, Japan) was carried out with test speed of 0.5 mm/min, and for the sake of clarity, 20 pebbles were examined and averaged.

3. Results and discussion

Fig. 2 shows the SEM images using back scattered electrons of the plasma-sintered beryllides with different contents of Ti. The EPMA analysis clearly prove that the samples are composed of Be, $Be_{12}Ti$, $Be_{17}Ti_2$, and Be_2Ti phases corresponding to the black, gray, light gray, and white areas, respectively. For the plasma-sintered beryllide rod with 3 at.% Ti, a large portion of Be remained. As the content of Ti increased, however, the $Be_{12}Ti$, $Be_{17}Ti_2$, and Be_2Ti phases increased while the Be phase decreased. Although variations of the Be, $Be_{12}Ti$, and $Be_{17}Ti_2$ phases were dominated by the lever rule, the Be_2Ti phase was exceptional. This is because both the sintering temperature and time were not sufficient to synthesize $Be_{12}Ti$, which is a stoichiometrically stable phase. The XRD results of the plasma-sintered beryllides are shown in Fig. 3, demonstrating that with increasing Ti content, the area fraction of the Be phase decreased while those of the $Be_{12}Ti$ and Be_2Ti phases increased. However, the $Be_{17}Ti_2$ phase was not identified as a result of its small portion in the XRD profile. Fig. 4 indicates the variation of each phase for beryllides used as the electrode for the REM, demonstrating that with increasing Ti content, the Be phase decreased while the other phases increased. This result is in good agreement with the XRD results. Although unexpected phases such as the Be_2Ti phase were formed in the plasma-sintered beryllide, undoubtedly, each beryllide rod retained its stoichiometric chemical composition. It was expected that using the plasma-sintered beryllide including other phases in addition to the $Be_{12}Ti$ phase provide ductility to the beryllide electrode rods such that breakage of the rods due to the arc heating shock experienced during the rotating electrode process can be prevented.

Granulation using a commercial rotating electrode machine was conducted, as shown in Fig. 5(a). The appearance of beryllide pebbles after the REM using plasma-sintered beryllide rods is shown in Fig. 5(b). The pebbles were approximately 1 mm in diameter but there was some scattering of the sizes.

For mass production, the fabrication yield of the pebbles is as an important factor. Based on the results obtained by dividing the pebble weight by the rod weight, the yield was approximately 80%. This result includes whole pebbles with not only different sizes but also crushed pebbles resulting from collisions of the pebbles with the inside of the container. Since a commercial machine was used in this study, however, the crushed pebbles can be modified by increasing the container size. Further studies of the yield of beryllide pebbles are ongoing as few studies on the fabrication of beryllide pebbles have been reported in the past couple decades. To investigate the phase distribution of beryllide pebbles, both the surface and cross-section of the pebbles were observed by SEM with back scattering electrons.



EPMA analysis	
Black	Be
Gray	$Be_{12}Ti$
Light gray	$Be_{17}Ti_2$
White	Be_2Ti

Fig. 2. SEM images of the specimens used as the electrode in the rotating electrode method, showing the different area fractions for each phase depending on the content of Ti: (a) 3 at.%, (b) 5 at.%, (c) 6 at.%, (d) 7 at.%, and (e) 7.7 at.% Ti after sintering for 5 min at 1073 K.