Powder Technology 321 (2017) 494–498

Contents lists available at ScienceDirect

Powder Technology

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

Synthesis of ultrafine beryllium powder of high purity by pyrolysis of di-t-butylberyllium etherate

Jiqiang Zhang, Yudan He, Long Chen, Lei Jin, Kai Li, Bingchi Luo, Wenqi Li, Weidong Wu, Jiangshan Luo *

Research Center of Laser Fusion, Science and Technology on Plasma Physics Laboratory, China Academy of Engineering Physics, Mianyang, Sichuan 621900, People's Republic of China

ARTICLE INFO

Article history:
Received 19 March 2017
Received in revised form 13 August 2017
Accepted 17 August 2017
Available online 18 August 2017

Keywords:
Ultrafine powder
Beryllium
High purity
Pyrolysis

ABSTRACT

Ultrafine beryllium (Be) powder of high purity was successfully synthesized by the pyrolysis of high-purity di-t-butylberyllium etherate (t-$Bu_2Be\cdot Et_2O$) in elevated solvent. Scanning electron microscopy (SEM) shows that the powder is near-spherical. The most probable size of Be powder slightly decreases from 1.8 μm to 1.2 μm when the pyrolysis temperature increases from 202 °C to 214 °C and obviously decreases to 0.4 μm as further increases to 230 °C. X-ray diffraction (XRD) indicates that the powder has a single phase of α-Be with average crystallite size of 19.8 nm–21.5 nm. The total content of metal impurities in the powder is 6.8 ppm–94.0 ppm by inductively coupled plasma (ICP) determination.

1. Introduction

Beryllium possesses unique properties, and it has been widely applied in many important engineering areas, such as nuclear energy systems and propulsion systems of aviation and aerospace [1–3]. As-cast beryllium contains large columnar grains and microcracks. It exhibits almost no ductility at room temperature, and is extremely difficult to fabricate [4]. Microcrystalline and nanocrystalline Be is significantly stronger than coarse-grained Be. So beryllium metal is almost entirely processed by powder metallurgy. The quality of Be powder (particle size, particle shape, purity) may play a very important role in the production of microcrystalline and nanocrystalline Be. Disk milling and impact attrition milling are the two most commonly used methods in the production of Be powder, and the latter is more sophisticated than the former. In this paper, a method for synthesis beryllium powder was developed: pyrolysis of high-purity t-$Bu_2Be\cdot Et_2O$ in solvent (dodecane or tridecane). Be powder with smaller particle size and lower impurities was synthesized by this method.

2. Experimental

2.1. Synthesis of Be powder

High-purity (99.999 wt%) t-$Bu_2Be\cdot Et_2O$ was used as precursor [5–7]. All the operations for the synthesis of beryllium powder were conducted under an inert atmosphere in a glove box. The content of oxygen and hydrosphere in the glove box was about 1.0 ppm and 0.1 ppm respectively. Fig. 1 shows the scheme for the synthesis of Be powder. High-purity BeH_2 powder was firstly prepared by pyrolysis of high-purity t-$Bu_2Be\cdot Et_2O$ in elevated solvent. Then free Be was synthesized from overpyrolysis of BeH_2 at temperatures exceeding 215 °C. Lastly, solvent was removed by distillation and Be powder was obtained.

Experiment apparatus was composed of a glove box, a heating resistance collar, a 3-neck reaction flask, a 1-neck reaction flask, a reflux condenser, an oil bubbler, a dropping funnel, an electronic temperature detector, a vacuum pump, a magnetic stirrer etc. The 3-neck reaction flask was connected to the reflux condenser, the drop funnel and the electronic temperature detector respectively. The vent of reflux condenser was connected to the oil bubbler outside the glove box by a tube. At beginning, the 3-neck reaction flask was charged ~300 g of solvent. The solvent was heated to a desirable temperature and then the temperature was maintained during pyrolysis process. Stirring speed was increased to 500 r/min and ~80 g of t-$Bu_2Be\cdot Et_2O$ was added to the elevated solvent at rate of ~1 g/min. After addition was completed, pyrolysis and gas evolution slowed. White BeH_2 slurry was obtained. Then the BeH_2 slurry was heated to 215 °C–230 °C in the solvent and the temperature was maintained over a period of ~5 h. Overpyrolysis of BeH_2 occurred and Be slurry was obtained. Lastly, solvent was removed by distillation and dried Be powder was obtained.

Seven samples with different pyrolysis temperatures of 194 °C, 198 °C, 202 °C, 206 °C, 210 °C, 214 °C, 230 °C were obtained. Besides, another three samples (powder 1#, powder 2#, powder 3#) synthesized by t-$Bu_2Be\cdot Et_2O$ of different purity (t-$Bu_2Be\cdot Et_2O$ 1#, t-$Bu_2Be\cdot Et_2O$ 2#, t-$Bu_2Be\cdot Et_2O$ 3#) at same temperature of 202 °C were obtained.

* Corresponding author.
E-mail address: jiangshanluo@caep.cn (J. Luo).

http://dx.doi.org/10.1016/j.powtec.2017.08.050
0032-5910/

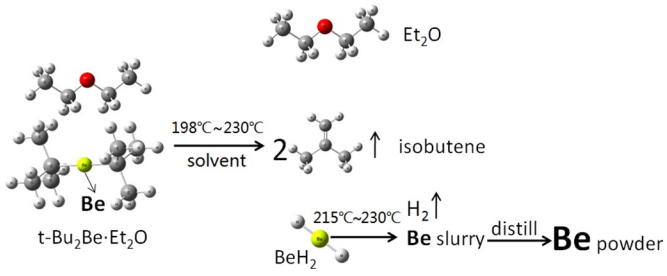

Fig. 1. The scheme for the synthesis of Be powder.

2.2. Characterization

The obtained powder was characterized by X-ray diffraction (XRD, Empyrean with Cu Kα-radiation λ = 0.15406 nm at 40 kV and 40 mA), scanning electron microscopy (SEM, ZEISS ULTRA 55, acceleration voltage: 15.00 kV). The bulk chemical compositions were obtained by chemical digestion of the Be powder followed by characterized in inductively coupled plasma mass spectrometry (ICP-MS, PerkinElmer Nexlon 300×), and inductively coupled plasma atomic emission spectrometry (ICP-AES, IRIS Intrepid II). High-resolution ICP-MS was used to determine ultra-trace impurities (ppb), and ICP-AES was used

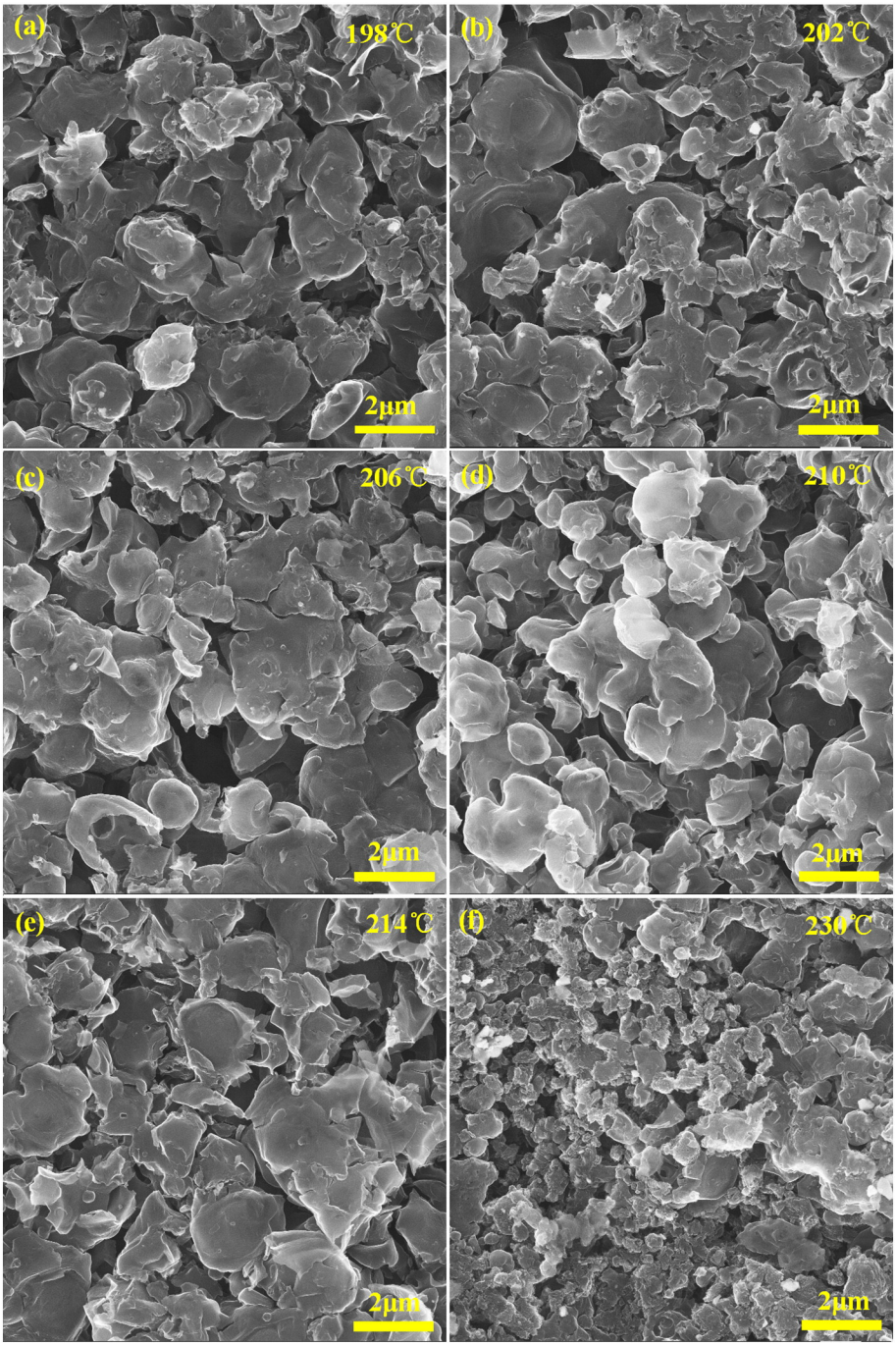

Fig. 2. SEM image of Be powder.