Available online at www.sciencedirect.com

SCIENCE DIRECT

Acta Metall. Sin. (Engl. Lett.) Vol. 19 No. 6 pp411–417 Dec. 2006

ACTA
METALLURGICA SINICA
(ENGLISH LETTERS)

www.ams.org.cn

COMPARISON ON REFINEMENT OF IRON POWDER BY BALL MILLING ASSISTED BY DIFFERENT EXTERNAL FIELDS

L.Y. Dai[1,2], *B. Cao*[1], *and M. Zhu*[1]*

1) School of Mechanical Engineering, South China University of Technology, Guangzhou 510640, China

2) Marine Engineering Institute, Jimei University, Xiamen 361021, China

Manuscript received 22 March 2006; in revised form 1 August 2006

The cryogenic milling and milling in conjunction with dielectric barrier discharge plasma (DBDP) have been separately set up. The combined effect of low temperature and plasma on ball milling has been investigated by examining the refinement of particle size and grain size of iron powder using scanning electron microscopy, X-ray diffraction, and small angle X-ray scattering. It was found that the mean size of iron particles could reach 104nm only after 10 hours of ball milling in conjunction with DBDP, whereas a minimum average grain size of 8.4nm was obtained by cryomilling at −20℃; however, it is difficult to refine the particle size and grain size under the same milling condition in the absence of DBDP and cryogenic temperature.

KEY WORDS *ball milling; cryomilling; dielectric barrier discharge (DBD); nanoparticle*

1. Introduction

Fabrication and microstructure of nano-iron powders have generated considerable interest among researchers because of their unique application in magnetic recording, as magnetic fluid, catalyst, in wave absorption, as biomedical vector, and in site remediation[1–5]. The methods commonly employed to obtain iron nanoparticles include physical process and chemical process, such as inert gas condensation[6], high-energy ball milling[2,7], sol-gel method[8], and micro-emulsion method[9]. The properties of iron nanoparticles also strongly depend on the preparation methods because the microstructural features are sensitive to the processing parameters[10].

*Corresponding author. Tel.: +86 20 87112762.

E-mail address: memzhu@scut.edu.cn (M. Zhu)

High-energy ball milling is one of the important means to prepare nano-powders[11]. When metal powder is milled in conventional ball milling, much time is required for obtaining nanoparticles. It is an feasible way to improve the efficiency of normal ball milling by cryomilling[12] and by applying external fields[13]. Cryomilling process may remove or reduce the dynamic recovery and recrystallization encountered in milling by reducing the milling temperature, and it allows substantial refining of materials, such as Fe and Al powder, with high ductility, low recrystallization temperature. The efficiency of milling can also be significantly enhanced by the combination of mechanical energy and other physical energies when milling is assisted by external fields[14]. Recently, a vibratory ball mill assisted by dielectric barrier discharge plasma (DBDP) has been invented by the authors of this study. The details of this technique have been described elsewhere[15,16]. DBDP is a typical nonequilibrium, high-pressure (≥ 0.1MPa) ac gas discharge[17]. It can be excited between two electrodes, at least one of which should be covered with a dielectric material, by applying a high-voltage alternating current on the electrodes. Local spark or arc discharge cannot generate in the plasma zone during dielectric barrier discharging, as a result of which the steel balls, vials, and the powders are prevented from being sintered or being damaged when DBDP is induced into the milling process.

In the present study, the influence of the application of external fields on ball milling by investigating iron powders milled separately under conventional milling, cryomilling, and milling in conjunction with DBDP, has been reported. An attempt has also been made to understand the mechanisms of the refinement of the iron particles and grains during the vibratory milling assisted by different external fields.

2. Experimental

Fig.1 is the schematic illustration of the vibratory mill used in this study. The cryomilling was realized by circulating the refrigerant in the interlayer chamber of a double-deck vial. The refrigerant was provided by a low-constant temperature bath (DC-2020, Shanghai Hengping Apparatus Plant). For DBDP-assisted milling, a high-frequency discharge device was used to generate DBDP inside the milling vial[16].

Iron powder with purity of 98% and average particle size of 10μm was milled in various modes up to 60h in a stainless steel cylinder under protection of high-purity argon atmosphere (0.1MPa). The balls were vibrated within the cylinder at a double amplitude of 10mm and a frequency of 25Hz. The weight ratio of the ball to the powder was 50:1. The temperature for cryomilling was controlled at −20℃. The power supply used in DBDP-assisted milling generates 24kV of high-voltage alternating current of high

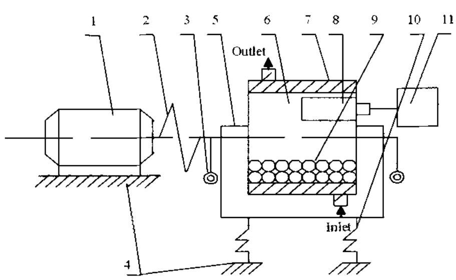

Fig.1 Schematic illustration of the vibratory mill assisted by external fields (1.electromotor; 2.elastic joint; 3.vibration exciter; 4.baseplate; 5.framework; 6.vial; 7.interlayer chamber; 8.electrode; 9.steel balls; 10.spring; 11.DBDP power).