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Milling of magnesium powders without additives

Gülhan Çakmak [a,*], Tayfur Öztürk [b]

[a] Department of Metallurgical Engineering, Bülent Ecevit University, 67100, Zonguldak, Turkey
[b] Department of Metallurgical and Materials Engineering, Middle East Technical University, 06800, Ankara, Turkey

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ABSTRACT

Mechanical milling of ductile metals and alloys into fine powders is difficult to achieve due to particle agglomeration. Even though cryogenic milling and/or wet milling can lead to substantial reduction of particle size, the production of fine powders from ductile metals and alloys is a difficult process. The current study deals with the milling of Mg and examines alternative processing methods that would yield an efficient size reduction. Two methods were studied; milling with MgH_2 addition, and milling the powders pre-deformed by equal channel angular pressing (ECAP). The study showed that both methods are successful in preventing particle agglomeration resulting in a significant reduction in particle size. Equal channel angular pressing has the advantage that could be applied to all metals and alloys whereas milling with hydride could only be applied to metals forming their own hydride. It is concluded that, of the two methods, milling with hydride addition is a more effective method of size reduction.

1. Introduction

Powder particle size may be controlled at the production stage via adjustment of the relevant parameters, but for most purposes it is necessary to further process the powders to finer sizes. This is often achieved by milling. In ceramics and intermetallics, milling leads to an efficient size reduction yielding particles that are sub-micron in size. In ductile powders, however, the milling in addition to particle fragmentation, involves particle welding which often results in particle agglomeration [1]. Thus, ductile powders are often wet-milled so as to obtain an efficient size reduction [2]. Wet milling is not always desirable and it is necessary to employ alternative methods of size reduction. Such methods rely on the reduced ductility of powders, the most common method being the cryogenic milling [3]. For metals that form hydrides, milling under hydrogen atmosphere, i.e. reactive milling, is an efficient method of size reduction [4]. The use of additives, oxides, carbides or fluorides for the same purpose is also quite common, though in such cases there is a loss of some volume fraction.

Mg or Mg based alloys in the form of fine powders are essential for hydrogen storage purposes. Of necessity, such processes are to be carried out in dry conditions. In this study, two methods of dry milling are investigated: milling with MgH_2 addition and milling the powders pre-deformed by equal channel angular pressing (ECAP).

2. Material and method

Material used in this study was Mg powder (Alfa Aeser) with a median size (d_{50}) of 45.7 μm. MgH_2 (Goldschmidt) used as additive was 0.3 μm in size. Milling was carried out in a planetary mill (Fritsch-Pulverisette 7 premium line) at a speed of 700 rpm under argon atmosphere using a stainless steel vial. 15 mm diameter stainless steel balls were used with a ball-to-powder ratio of 10:1. In order to prevent heating, milling was stopped half an hour for every 30 min of operation.

A die used for ECAP processing of Mg powders is shown in Fig. 1. Here the channel angle is $\phi = 90°$ with a corner radius of $\psi = 20°$. In order to feed powders into the die-cavity, powders were embedded in a copper block. The block had a cross-section of 14×14 mm and with an 8 mm diameter bore. This bore, after having filled with Mg powders, was closed with a copper plug. The copper block was placed in the die cavity and pressed through the die with a punch. A passage through the die with the geometry given in Fig. 1 corresponds to a true strain of $\varepsilon = 1$ [5]. The sample was rotated 90° after each passage and re-fed to the die cavity. Following ECAP deformation, Mg powder which had been converted into a solid piece was removed from the copper block. The solid piece was crushed and milled for a short duration under the conditions given above.

Powders milled with MgH_2 addition and milled with pre-deformation with ECAP were characterized in terms of particle size distribution. A laser particle size analyzer was used for this purpose. Since Mg reacts with water, ethanol was used as dispersing medium.

3. Results and discussion

Of the two methods under investigation in this study, first milling with MgH_2 addition will be considered followed by milling of powders pre-deformed by equal channel angular pressing. For the former, Mg samples were prepared with MgH_2 addition of 5, 10 and 30% by volume. A batch of 10 g powder was prepared for each experiment. Milling was

* Corresponding author. Tel.: +90 372 257 4010 1411.
E-mail address: gulhan.cakmak@beun.edu.tr (G. Çakmak).

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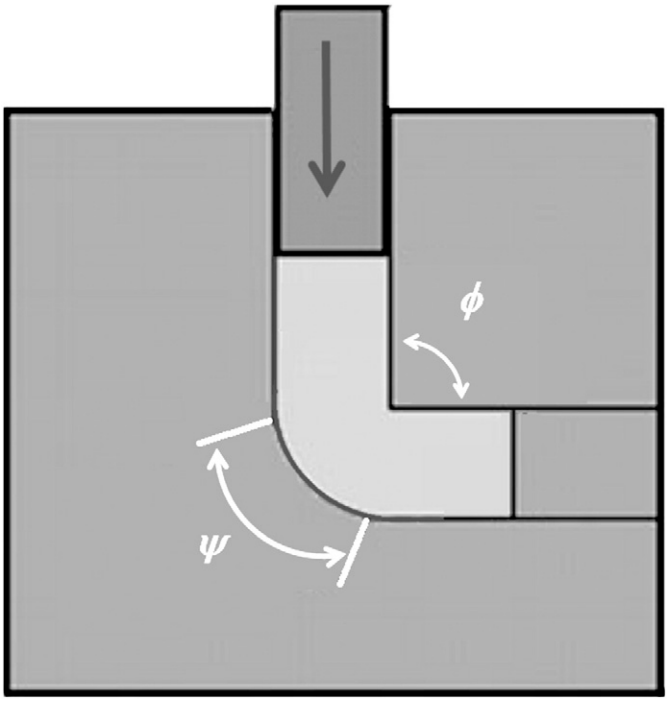

Fig. 1. Schematic representation of a die used for equal channel angular processing of powders. Channel angle $\phi = 90°$ and corner radius $\psi = 20°$.

carried out under argon atmosphere. Particulate structures obtained after 3 h of milling are shown in Fig. 2. Particle size distributions measured for the samples are given in Fig. 3(a). Numerical values derived from these distributions are reported in Table 1. Particle size values of a sample milled without additive are also included in this table. As seen in the table, milling with MgH_2 addition leads to particle sizes that are much finer than that milled without the additive. The addition of 5–10% by volume MgH_2 leads to median particle sizes of approx. 53 μm and 35 μm, respectively. This is reduced further to 26 μm with 20% by volume addition. The particle size with 30% MgH_2 addition is 9.8 μm. These values should be compared to 80 μm particle size obtained without the addition of MgH_2.

For the alternative method of pre-deformation with ECAP, samples were subjected to four passes through the die shown in Fig. 1. As a result of this deformation, the powders were compacted and consolidated to a solid piece. Consolidated Mg removed from the copper block was crushed and milled under the same condition as given above. Particulate structures obtained after milling of the samples are given in Fig. 4. As seen in the figures, particles resulting from pre-deformation are much finer than those subjected to direct dry milling, compare Fig. 2(a). The particles which have a median (d_{50}) size of approx. 40 μm after 2 h of milling are reduced to a size of 26 μm after 5 h of milling.

Results reported above show that both methods; milling with MgH_2 addition and milling of powders pre-deformed by ECAP lead to an efficient size reduction of Mg powders. Thus, MgH_2 addition in the order of 10–20 vol.% yields a particle size of 35–26 μm (30 vol.% MgH_2 yield 10 μm). For the same duration of milling, powders pre-deformed by ECAP results in a particle size of 26 μm. These values should be compared with an average particle size of 80 μm obtained with a direct dry milling of Mg powders. This reduction in particle size which occurs with pre-deformation should be attributed to the reduced ductility of powders brought about by ECAP deformation. It is well known [e.g. 6] that ECAP deformation leads to an increased dislocation density in deformed powders which tends to make powders more breakable.

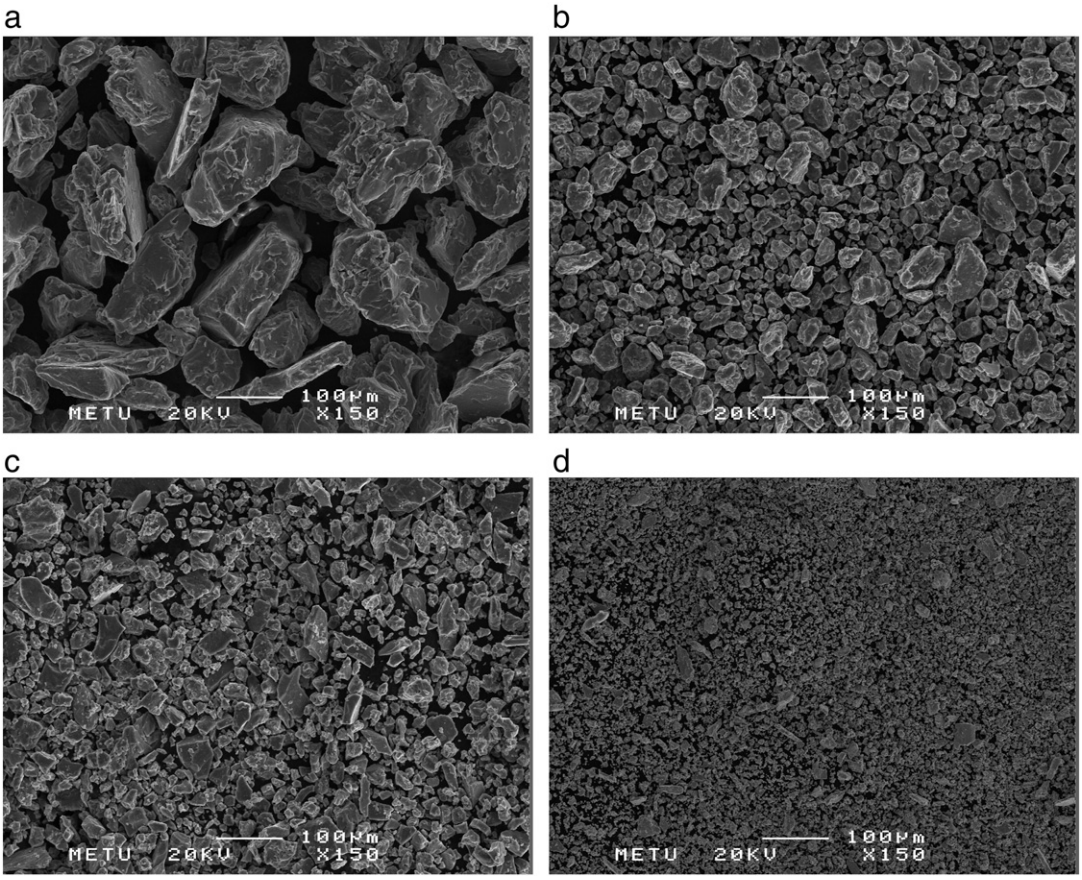

Fig. 2. Particulate structure of Mg powder milled with MgH_2 addition a) without additive, with MgH_2 additives of b) 5 vol.%, c) 10 vol.% and d) 30 vol.%.