INFLUENCE OF MILLING ATMOSPHERE ON THE HIGH-ENERGY BALL-MILLING PROCESS OF PRODUCING PARTICLE-REINFORCED ALUMINUM MATRIX COMPOSITES

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Abstract

The present article deals with the influence of different milling atmospheres (air, argon, nitrogen) on the high-energy ball-milling process when milling an Al alloy with SiC particles. The investigations show that the reaction of the ground material with air, when rinsed with air, changes the milling properties of the aluminum powder significantly. Unlike with inert atmospheres, the use of a process control agent (PCA) is therefore no longer necessary.

1 General Introduction

Aluminum reinforced with hard particles is expected to show improved mechanical properties in comparison to the unreinforced alloy. A fine dispersion of particles in the metal matrix and an appropriate interface state are required. In this context, the powder-metallurgical production of MMCs has advantages over casting methods. High temperatures, as in the processing in the molten state, can be avoided. So, the diffusion and chemical reaction between the matrix and hard particles is limited or prevented. A suitable method for producing particle-reinforced aluminum alloys is the high-energy ball milling (HEM) in combination with a subsequent consolidation. The milling process is influenced by a lot of parameters such as the milling atmosphere. In order to avoid undesirable reactions of the milling material, inert gases are applied frequently [1, 2]. Selective phase transformation through a reactive ambient medium such as air is another way. In the case of aluminum powder, oxides developing on the powder surface can turn into finely dispersed reinforcement particles during the milling.

In our previous publications [3-6], we discussed materials which were milled by means of a closed chamber with a constant small amount of process control agent (PCA). This work deals with the influence of rinsing with gas, and varying the amount of PCA on the HEM process and the resulting powder for the material pairing present.

2 Experiments

Spherical Al powder of a diameter ≤ 100 μm (EN AW 2017) was used as feedstock material for the matrix. SiC of submicron- and micron-grain size was chosen as reinforcement particles. In this work, the milling behavior with and without SiC particles was investigated to determine the influence of the milling atmosphere on the formation of the composite powder and the composition of the matrix material. The starting point was the milling atmosphere, i.e. the residual air still in the chamber after charging, which was described in previous publications [4-6]. A supply and discharge of gases during the process did not take place. In contrast, the other test setups worked with gas rinsing, where the milling chamber was initially flooded and continuously rinsed during milling. To control the gas flow, a bubble counter was used. Nitrogen and argon were used as inert gases in addition to air. The high-energy ball milling was carried out with a Simoloyer CM08 mill (Zoz) with steel equipment. The milling parameters used in all experiments are listed in Tab. 1. A PCA was added to limit the welding of the particles and to avoid unwanted adhesions on the rotor, the balls and the chamber wall. The use of stearic acid is very common [7–9]. In the framework of this contribution, 0 and 0.5 wt.-% stearic acid were applied as PCA in addition to 0.13 wt.-% as used in our previous publications. Thus, the influence of stearic acid on the ground material and possible interactions with the milling atmosphere were examinable. The prepared powder cross-sections were first characterized by means of light microscopy (LM, Olympus PMG 3). The main focus of the investigations was on the particle-reinforced powders because the degree of dispersion and the distribution of the SiC particles in the matrix material can be very well represented by light microscopy. However, the size and shape of the composite powders are also important characteristics from which conclusions can be drawn about the influence of the milling atmosphere and the PCA. The samples were further analyzed by scanning electron microscopy (SEM, Zeiss Leo1455VP) and energy dispersive X-ray microanalysis (EDXS, EDAX Genesis).

3 Results and Discussion

The composite powder formation is described in detail in [4, 5]. Initially, the spherical aluminum particles are deformed into flat particles. Simultaneously, the reinforcements attach to the surface of these flakes. In the next stage, the effect of cold welding starts and leads to the formation of larger composite particles with lamellar structure. The resulting structure is a mixture of alternating reinforced and unreinforced lamellas. Free particles are no longer existent at that stage. A steady deformation of the composite particles causes an increase in mixing and thus an improvement of the dispersion degree. The chronological procedure of the composite powder formation depends on the milling parameters. Strong welding effects due to high rotational speeds lead to premature formation of large, poorly mixed particles. They also lead to an increase in the composite powder grain size fraction.

The described process can be significantly influenced by the amount of PCA. In the early milling state, a large proportion of PCA leads to an increased flattening of the metallic powder without attaching of the SiC particles. The effect of cold welding is hindered by the PCA until it is degraded. Thus, the composite powder formation is delayed. The effect is particularly evident when milling aluminum powder without SiC particles. It results in extremely flattened particles (Fig. 1).

During milling at residual air atmosphere (closed vessel) without PCA, the cold welding causes a strong coarsening of the powder (Fig. 2). This limits the possible milling time and thus the desired fine distribution of hard particles inside the powder particles. Increasing the amount of PCA reduces the effect of cold welding and so limits the powder coarsening. When using an inert milling atmosphere, similar effects can be observed.

A completely different behavior of the process is detectable when using air rinsing (Fig. 3). The influence of air reduces the cold welding to a favorable level. Therefore, the PCA has no significant influence under these conditions. The composite powder formation works very well even without PCA (Fig. 3a). Adhesions to the milling tools do not take place. The formation of oxide on the surface of the aluminum particles is assumed to inhibit the tendency to cold welding similar to the PCA. In the literature, alumina is occasionally used as PCA [10].

It is likely that the atmosphere and the PCA cause changes in the composition or contaminations of the ground material. For large amounts of stearic acid (up to 5 wt.-%), an influence was already detected by means of thermogravimetric measurements [11]. Own TGA investigations on powders after milling with 0.5 wt.-% of stearic acid have not yielded any useful results.

On the basis of EDXS measurements, the oxygen concentration of the powder was tested. Analyses were performed on powder cross-sections in the core of the powder particles. Tab. 2 clearly shows the influence of the milling atmosphere on the oxygen content in the composite. As expected, the oxygen content is multiplied by using air rinsing compared to inert gas rinsing. It has to be noted that the PCA also inserts oxygen into the ground material. The extent of this effect depends on the milling conditions. This is particularly evident in milling tests with a closed milling chamber. Due to the high pressure in the chamber, the evaporation of the stearic acid only takes place at higher temperatures. Additionally, the gaseous stearic acid cannot leave the milling chamber, which would be possible with gas rinsing. In this respect, for the closed as well as the rinsed experimental setup, different amounts of PCA are involved in the milling process. It is assumed that the oxygen content in the case of milling with a closed chamber originates from both the residual air and the stearic acid. This has been confirmed by the comparison of the oxygen concentrations of 2.4 and 2.9 wt.-% for the PCA amounts 0.13 and 0.5 wt.-% respectively.

4 Summary

The influence of the milling atmosphere and the amount of PCA on the high-energy milling of an aluminum alloy with SiC particles (0.2 to <2 microns) was studied. As expected, the comparison of the inert gases nitrogen and argon shows no significant differences with respect to the grinding behavior. However, the use of rinsing air results in different behavior. Whereas the use of inert gases and a closed milling chamber principally only works with a PCA in order to limit excessive adhesions on the milling tools as well as powder coarsening, this can be omitted because of the separating effect of the in-situ-formed alumina. Accordingly, air rinsing shows the clearest changes in the increase of the oxygen content during milling without SiC.

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Fig. 1: Optical micrograph of milled AA-2017 without reinforcements and 0.5 wt.-% stearic acid after 4 h of milling time.

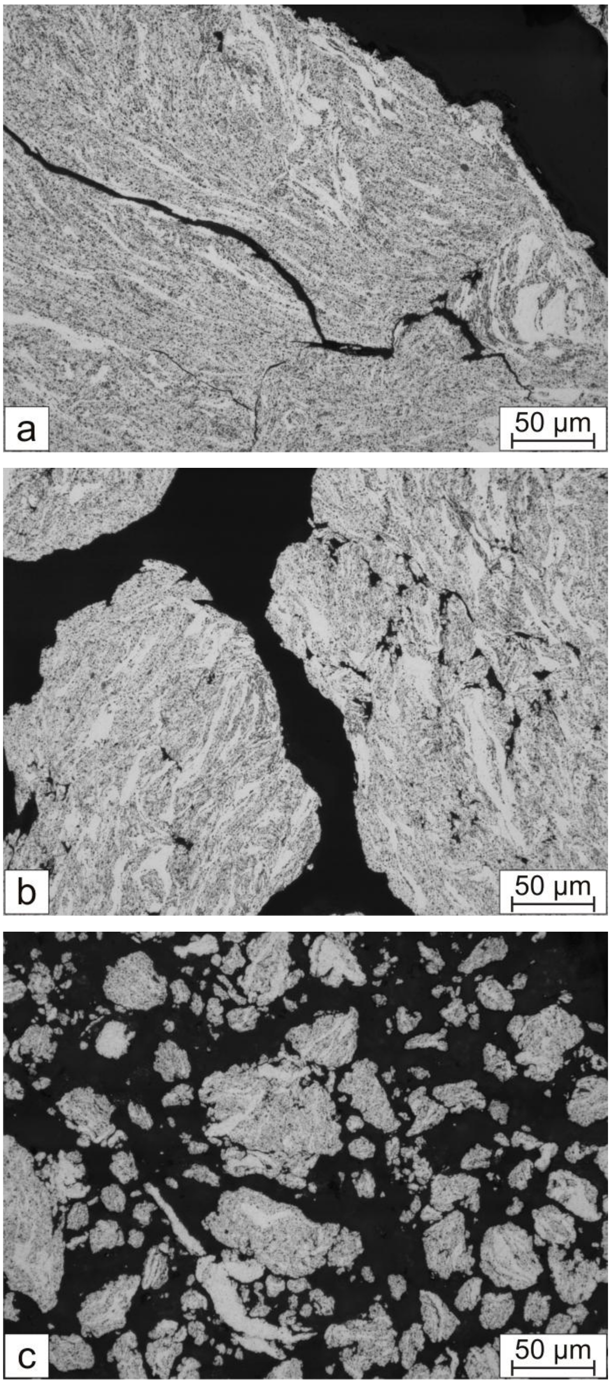

Fig. 2: Optical micrograph of milled AA-2017 with 10 vol.-% SiC under residual air:
a) without PCA, after 3 h of milling time;
b) with 0.13 wt.-% stearic acid after 4 h of milling time;
c) with 0.5 wt.-% stearic acid after 4 h of milling time.

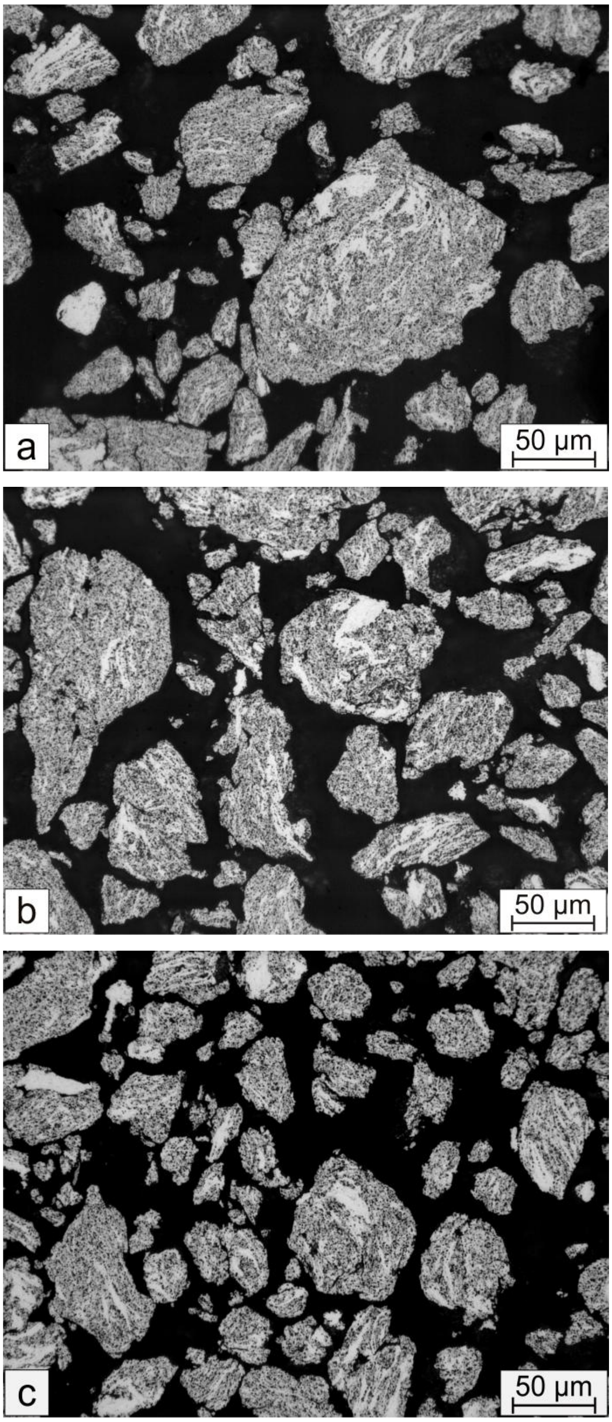

Fig. 3: Optical micrograph of milled AA-2017 with 10 vol.-% SiC under rinsing air:
a) without PCA, after 4 h of milling time;
b) with 0.13 wt.-% stearic acid after 4 h of milling time;
c) with 0.5 wt.-% stearic acid after 4 h of milling time.

Tab. 1: Milling parameters.

Parameter (Simoloyer ® CM08)	Value
Mass of steel balls	8 kg
Ball diameter	4.6 mm
Ground material / powder mass	0.8 kg
Rotor speed	400 - 700 1/min (cyclic)
Milling time	4 h

Tab. 2: EDXS measurements: oxygen concentration in milled AA-2017 powder, depending on the atmosphere at constant grinding parameters and 0.13 wt.-% PCA.

Milling atmosphere	Oxygen content [wt.-%]
Closed chamber (residual air)	2.4
Air rinsing	4.8
Argon rinsing	0.9
Nitrogen rinsing	0.9

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