

Particle Morphology Change and Its Coagulation during Ball-Milling of Cu Powder

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Introduction

It is widely known that during dry milling of a premix of metal powders the particle morphology and size change due to the particle coagulation and fracture caused by impact of milling balls⁽¹⁾. The authors showed that the mechanical alloying process of Ti-Cu⁽²⁾, Ti-Al⁽³⁾ and Al-Nb⁽⁴⁾ powder mixtures which resulted in eventual formation of alloys or intermetallics were described as follows: 1)formation of flaky particles of elemental powders, 2)formation of disk-shaped composite particles having layered structure, 3)formation of globular equiaxed particles with randomly oriented fine lamellar structure, 4)formation of alloy or intermetallic fine particles on the globular equiaxed particle surface. These reports revealed that particles with various sizes and shapes which characterize the milling processes were formed during milling. Especially, on the know of the efficient manufacturing condition of intermetallic compound in the mechanical alloying, globular equiaxed particles formation mechanism from elemental particles is very important since the intermetallic fine particles formed equiaxed particle surfaces. However, very few reports treated the formation mechanisms of those particles with various shapes and sizes during milling of metal powders.

It is also widely known that the particles are cold welded on ball surfaces and mill container wall and form thin composite layers during milling^(1,5,6). It is presumed that the thin layers are work-hardened and break off and consequentially form composite particles⁽³⁾. Therefore, the formation of the thin layers, which is called 'coating', is presumed to play an important role in the formation of the particles with various shapes and sizes.

In this study, a tumbler-ball milling was carried out to investigate the particle morphology change and its coagulation which are characteristic to mechanical alloying of ductile powders, using Cu powder as a model material. The reason why only the elemental Cu powder was milled is to exclude effects of alloy formation on the morphology change and coagulation. The formation mechanisms of the particles with various shapes and sizes were investigated from the mechanical point of view. Particle coagulation during milling was analyzed by Gillespie-Langstroth coagulation-and-deposition rate equation⁽⁷⁾ which is generally used for Brownian particle coagulation and deposition.

Experimental procedures

An electrolytic Cu powder(99.9%)with an average size of 48 μ m was milled in argon by a laboratory tumbler-ball mill. The Cu powder was annealed at 673K for 1h in a H₂ atmosphere to remove oxide on the surface before milling. The powder was charged in a stainless steel(SUS304) mill container(70mm inner diameter and 135mm inner length) with 12.7mm hardened steel balls(SUJ2) in an argon glove box. The ball charge was 505 of the full charge of the mill container. The powder charge filled 10% of void space in the ball charge(powder/ball weight ratio, 1/63). The powder was milled at a rotational speed of

11.4rad/s. After a given period of run, all the milled powder and several milling balls were taken out of the container and were observed by an optical microscope and SEM. The number of particles included in unit mass of milled powder was counted by using the optical microscope to analyze the fracture and coagulation. The milled powder and milling ball were mounted in an epoxy resin and polished to expose cross sections. The cross sectional microstructure of the particles and milling ball were observed by the optical microscope and the micro Vickers hardness was measured on the cross sections of the particles. Particle size and morphology were quantitatively estimated by measuring projected area diameter and morphology factor(OSF) of the cross section of particles.

Conclusion

Particle morphology change from elemental particles to globular equiaxed particles and its coagulation which is an important role during mechanical alloying of ductile materials were investigated by using Cu powder as a model material.

The obtained results are as follows:

- (1) Morphology of the Cu powder particles changed with milling as follows; elemental particles→disk-shaped particles→globular equiaxed particles. This morphology change process is in accord with the results of observation in the mechanical alloying of ductile metal powder mixtures. And it is considered that these morphology changes are due to the purely mechanical factor.
- (2) It is believed that the disk-shaped particles were formed by break off of the thin layers made on ball surfaces and the mill container wall by the impacting action of balls in the early stages.
- (3) The globular equiaxed particles grew by folding and unusual coagulation of the disk-shaped particles.
- (4) The globular equiaxed particles grew by agglomeration of the disk-shaped particles and globular equiaxed particle, of agglomeration of globular equiaxed particles.
- (5) The Gillespie-Langstroth coagulation-and-deposition rate equation well described the particle coagulation and coating processes during milling.

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