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Production of fine calcium powders by centrifugal atomization with rotating quench bath

Liang Tian [a,b,*], Iver Anderson [a,b], Trevor Riedemann [b], Alan Russell [a,b]

[a] Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI, 48109, USA
[b] Ames Laboratory, U.S. Department of Energy, Iowa State University, Ames, IA 50011, USA

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ABSTRACT

Recently, a novel Al/Ca composite was produced by severe plastic deformation of Al powders and Ca granules for possible use as a high-voltage power transmission conductor. Since the strength of such composites is inversely proportional to the Ca filament size, fine Ca powders (less than ~250 μm) are needed to achieve the desired high strength for the powder metallurgy production of an Al-matrix composite reinforced by nano-scale Ca filaments. However, fine Ca powders are not commercially available. Therefore, we have developed a method to produce fine Ca powders via centrifugal atomization to supply Ca powder for prototype development of Al/Ca composite conductor. A secondary goal of the project was to demonstrate that Ca powder can be safely prepared, stored, and handled and could potentially be scaled for commercial production. Our results showed that centrifugal atomization can yield as much as 83 vol.% Ca powder particles smaller than 250 μm. The mean particle size sometimes matches, sometimes deviates substantially from the predictions of the Champagne & Anger equation likely due to unexpected secondary atomization. The particle size distribution is typical for a ligament-disintegration atomization mode. Scanning electron micrographs showed that the morphology of these Ca powders varied with powder size. Spark testing and auto-ignition tests indicated that the atomized powders were difficult to ignite, providing confidence that this material can be handled safely in air.

1. Introduction

The authors are engaged in a long-term project to develop an Al/Ca metal-metal composite (MMC) for use as a next-generation high-voltage power conductor [1]. This composite is produced by a powder metallurgy method utilizing blended Al and Ca powders. Fine Al powder is readily available from commercial vendors; however, fine Ca powders (a few hundred microns and smaller) are not available commercially. The strength of an Al/Ca composite increases rapidly with decreasing Ca filament thickness by a Hall-Petch relation. For this reason, initial Ca powder particles with size below 250 μm are necessary to achieve the desired high strength in a transmission conductor. Once achieved, the Al/Ca composite could reduce the construction costs and increase the reliability and efficiency of high-voltage power transmission systems [2]. This paper reports on the development of a centrifugal atomization method to produce fine Ca powders that could be scaled up to meet the future needs of commercial-scale manufacturing of Al/Ca composite conductors.

Centrifugal atomization is a well-established method for the production of fine metal powders [3–5]. This method has been successfully used to produce various metal powders such as Sn, Pb, Al, Mg, Zn, Ti, Ni, and their alloys [4,5]. Compared with liquid or gas atomization techniques, centrifugal atomization can produce highly spherical metal powders with low impurity content, narrow particle size distributions, and high production yields [4,5]. Centrifugal atomization with a rotating disk uses centrifugal force to disintegrate a molten metal stream poured directly onto the middle of a rotating disk, cup, or wheel [3] that is spinning about a vertical axis. For rotating-disk centrifugal atomization, successful atomization can be achieved only if the molten metal forms a thin liquid film on the surface of the rotating disk [3–5]. This requires excellent wettability of the molten metal on the rotating disk so the melt will flow freely on the disk's surface [3,5]. This liquid film will be accelerated by viscous drag between the liquid metal and rotating disk and will reach the full rotational speed by the time it experiences atomization at the disk edge [6]. These fine atomized droplets would be expected to solidify during flight by convective cooling before hitting the atomizer chamber wall or quenching medium [6].

* Corresponding author at: 2018 Gerstacker Bldg, 2200 Bonisteel, Ann Arbor, MI 48109, USA.
E-mail address: lilangt@umich.edu (L. Tian).

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1.1. Atomization disk characteristics

The design characteristics of a rotating disk atomizer have a significant effect on the performance of the atomizer [3–6]. Improper disk design can cause deviations from optimal atomization conditions, resulting in undesired powder characteristics such as large mean particle size; irregular, non-spherical particle shape; wide particle size distributions; and excessive chemical contamination [3]. An ideal atomization disk has the following characteristics: very good wetting by the liquid metal but minimal erosion/dissolution in the melt, low heat capacity and thermal conductivity, and a smooth profile that redirects the falling liquid to horizontal flow pattern [3].

The good wetting between rotating disk and molten metal ensures that the liquid would be atomized at the disk edge, as previously discussed [3,5]. Poor wetting (or a sub-optimum melt superheat) may result in a metal skull forming on the disk, which reduces the effective radius of the atomization disk and raises the droplet size since the atomization occurs at the edge of the skull instead of the disk edge [7]. Moreover, the skull may detach from the atomization disk during operation, which could damage the device and degrade the atomizer performance [5]. Some investigators reported that a moderately rough surface finish on the atomization disk promoted physical bonding between the disk and skull, preventing skull detachment [3]. Wetting conditions also can be improved through design modifications of the disk. For example, a thin coating of other metals on the disk, such as Ta [3] and Sn [5], has been used to enhance wettability of a stainless steel atomization disk. Likewise, excellent wetting can be achieved between liquid metal and a coating with a composition similar to the molten metal [5]. The surface profile of an atomization disk can be designed to promote the wetting. Compared with flat disks, cup-shaped disks use centrifugal force to push the liquid metal against the disk surface to improve the wetting and to permit full liquid acceleration to be achieved [5]. In Ref [5], two methods other than promoting perfect wetting were taken to address the skull formation problem: a motor coupler was used to reduce heat conduction through the motor shaft, and a hot-air gun was used to preheat the atomization disk and maintain the disk at elevated temperature throughout the operation.

It is desirable for an atomization disk to have low heat capacity and low thermal conductivity because this allows rapid heating of the thin disk by the superheated melt, which minimizes solid metal buildup and the thick skull effect on mean particle size [3]. The break-up of the solid skull during atomization would also change the particle size distribution and powder morphology [5,6]. Therefore, in order to eliminate fractions of irregular particles, the thickness of this solid skull layer should be minimized [5,6]. The chemical inertness of the atomization disk to the molten metal stream is necessary to maintain high purity in the metal being atomized [3]. A smooth surface profile on the atomization disk would prevent liquid splashing and direct the molten metal stream evenly to form a uniform thickness liquid metal film, narrowing the particle size distribution [3].

1.2. Atomization modes

Three atomization modes are typically available at the rotating disk edge as shown in Fig. 1: direct droplet formation, ligament disintegration, and film disintegration [3–6]. The transitions between these three modes depend on liquid metal flow rate, the liquid metal's physical properties, and the speed of rotation and diameter of the atomization disk [5]. Direct droplet formation is favored at low liquid flow rates and over a range of disk rotating speeds. Increasing the flow rate continuously would lead to a transition to the ligament disintegration mode first and then to film disintegration [5]. Champagne and Angers proposed an empirical formula to predict the critical flow rates of the melt corresponding to the transitions between these three modes, though the mechanisms of the transitions are not clear [8]. Considering the complexity of melt flow on an atomization disk and the speed of the disk edge, the atomization mode on the disk edge is difficult to identify as a specific mode without clear high speed image [5]. Therefore, mathematical models based on the flow behavior of liquid melt must be experimentally verified before they can be relied upon to predict particle shape and particle size distribution [5,9].

Ideally, centrifugally atomized powders have an approximately spherical shape. However, irregular particles have been observed frequently in centrifugally atomized Al and Mg alloy powders [10,11]. Various factors cause these irregular morphologies. First, during flight of liquid metal droplets, the cooling rate of fine droplets can be sufficiently fast to prevent complete spheroidization before solidification [6]. Besides, the liquid metal film may extend beyond the atomization disk edge to allow more time for cooling and delay the spheroidization. The spheroidization of these droplets is caused by the intrinsic interface perturbation coming from stagnant gas and liquid interaction according to Lord Rayleigh's instability theory [12]. The growing of the most favorable single harmonic perturbation is a kinetic process [13]. It takes more time for large droplets to complete spheroidization than it does for smaller droplets [13]. Thus, large droplets tend to develop irregular shapes, while small droplets are spherical, which has been experimentally observed by Xie et al. [5].

Second, if the atmosphere in the atomizer chamber contains oxygen, rapid oxidation of the liquid metal would form stiff oxide coatings around droplets, preventing their spheroidization and further disintegration [3,5,6]. This leads to irregular particle morphologies and distorted particle size distributions. An inert gas atmosphere design has been adopted to produce spherical powders of oxide-forming metals with less chemical contamination [3].

Third, irregular powder morphologies can also be caused by secondary atomization via impingement on the chamber wall [5,6]. Small droplets cool faster due to their higher surface-to-volume ratio and fully solidify before hitting the chamber wall, so their spherical shape is maintained. Large liquid droplets have relatively slower cooling rates than small liquid droplets and they could remain partially liquid and break up into flake or splat shapes upon striking the chamber

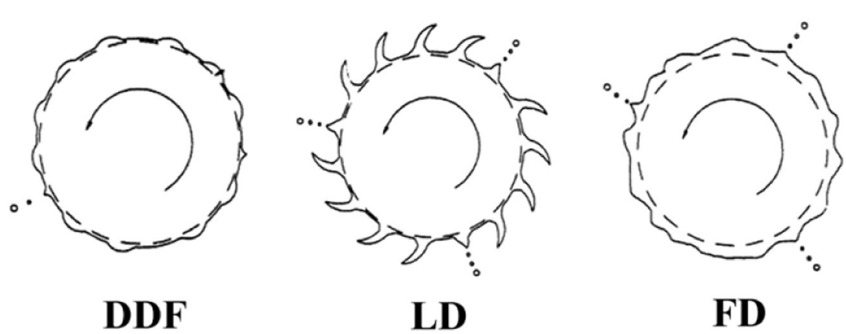

Fig. 1. Three atomization modes at the edge of atomization disk: direct droplet formation (DDF); ligament disintegration (LD); film disintegration (FD) [3].