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Invited review paper

New developments in spark production of nanoparticles



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ABSTRACT

The paper selects a number of recent developments in spark production of nanoparticles that are important for production of nanopowders and nanoparticulate materials. It explains the method, including recent improvements, and refers to theoretical considerations as well as practical experience in controlling the main particle parameters determining the product properties, namely size and composition. The paper focusses on particles below 10 nm, where the spark method works best. Values for feasible production rates and energy efficiencies are estimated using published data. Spark mixing is identified as a feature that renders great potential to the method, especially for catalysis but also for other purposes, as it opens myriads of new possibilities in the form of material combinations. The most important condition for this potential to turn into industrial application is the capability of scaling up. The basic principles that allow mixing are treated, methods are reviewed and examples for applications are given. These include the creation of new phases that only exist in the nanoparticulate state. A new technique allowing an increase of the production rate of a single electrode pair by a factor of 10^2 – 10^3 is introduced. It allows production nanoparticles typically 5 nm in size at a rate of 1 g/h, and this rate can arbitrarily be increased further by operating multiple sparks in parallel. The energy requirement is in the order of 3 kWh/g. The paper stems on adoption and interpretation of published articles as well as on new developments that are presented for the first time.

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1. Introduction

1.1. Objective of this publication

The present paper selects some published and unpublished developments in spark production that are, in our view, of principle importance in view of production of nano-powders and materials based on or containing nanoparticles. Much emphasis is laid on a unique feature of the spark method: the potential of mixing virtually any inorganic substance with any other inorganic substance(s) on a scale of nanometers or smaller. We refer to this feature as spark mixing. We are convinced that it is mainly this feature that renders great potential to the method, both for future industrial and research purposes. Spark mixing will, in our opinion, lead to the discovery and fabrication of completely new materials that cannot be produced effectively by any other method. In particular, this technique is poised to lead to a revolution in the field of heterogeneous catalysis, where the mixing capabilities allow the design of primary catalyst particles of a virtually unlimited number of compositions. We believe it will play an important role in replacing expensive noble metals by less expensive species like mixed transition metal oxides. Apart from homogeneous (atomic) mixtures, the flexible combination of different components mixed on a nanoscale is bound to lead to new possibilities. One prerequisite for practical application is the capability of producing sufficient quantities. The absence of scaled-up production is a major reason for the absence of any breakthrough in this field until now. Another task to be solved is creation of robust, hierarchical structures: porous on a nanoscale and preferably with varying pore dimensions from nano- to micrometers. The whole task is structuring all the way from atomic, via nanometer to micrometer dimensions, called micro- meso- and macrostructure in the field of catalysis. For catalysis, these structures do not need to be regular or follow specific geometrical patterns, as long as automatic ordering on the atomic scale provides the desired catalytically active sites. The random process of spark mixing, including nanoparticle agglomeration in the aerosol phase or in surface deposition, is believed to render an important contribution to this field.

The present paper therefore dedicates much attention to atomic and nanomixing and to unpublished crucial steps in scaling up. Existing approaches of creating porous, high surface area structures are briefly treated as well. The mixing part concentrates on published work, which has mainly been carried out in the group of the authors at Delft University of Technology, without wanting to diminish the value of the contributions of others. New considerations about feasible mass production rates and energy efficiency, based on published data, are presented. To begin with we describe the method in Section 2, and refer to particle size control – a crucial feature, as particle size determines the properties of the product just as the chemical composition does – in Section 3. For further reading on spark preparation of nanoparticles, we refer to a recent review by Mueller et al. [1]. This review from Lund University focuses on existing spark generator construction concepts, considerations concerning the discharge itself and classical particle growth principles. It explains numerous applications.

1.2. Historical sketch of spark production of nanoparticles

The phenomenon of sparks occurring between electrodes and ablating them has been known for a long time, for instance as an

undesired effect on spark plugs in gasoline engines. The evaporation per spark has been related to the energy dissipated [2] back in 1950. Continuous discharges were applied for producing particles in gasses in the early 20th century [3], but, to our knowledge, sparks were not applied for the purpose of nanoparticle production until first reported by Burtscher and Schmidt-Ott in 1984 [4]. A detailed description of the method by Schwyn, Garwin and Schmidt-Ott followed in 1988 [5]. This paper already demonstrates that particles as small as 1 nm in size can be produced by the method, and the production of particles ranging in size from the single atom to 100 nm by spark ablation have been reported since then in a large number of publications and PhD theses. The method was commercialized as a source of carbon particles in 1993 [6]. A notable publication about spark production of particles in liquids appeared in 1987 [7]. The review by Mueller et al. [1] and its references are recommended as further sources of literature on the method.

1.3. Features of spark production of nanoparticles

Spark production of nanoparticles is based on ablation of electrodes by inducing spark discharges between them. The particles produced have much in common with those produced by laser ablation [8–10], a method that is well established as a means of producing nanoparticles in quantities not exceeding milligrams for research purposes. The great attention that the spark has drawn as a competing principle primarily stems from the fact that the costs are much lower, because no laser is required. Secondly, nanoparticle production by spark discharge is scalable. The European project “Buonapart-E”, started in 2012 and joining 21 European partners, has scaling up of the spark method as an objective. The project was granted mainly on the grounds of the process being environmentally friendly, as no chemical precursors are required and no waste is produced.

In its original form the method applies no chemicals except the substance or mixture of substances that are supposed to compose the particles. Other notable features of the method are the feasibility of very high purity, enabling non-oxide particle production even for magnesium-based materials [11]. The particles are usually crystalline, and their presence in the aerosol phase after production allows reactive modification such as oxidation and size selection. The particles can, if desired, be brought into liquids, e.g. by bubbling [12]. Where purity is not a major concern, sparking in the liquid is also a possibility [7].

2. The method of spark production of nanoparticles

Fig. 2.1 shows the basic topology of a spark generator. Repeated sparks are produced between electrodes, and a gas flow, usually an inert gas, carries the formed particles away from the production volume. Mueller et al. [1] give an overview of the configurations applied to date. Numerous general studies on the phenomenon of spark discharge have been published [13]. A review on atmospheric pressure discharges connected with nanoparticle production has been written by Borra [14], but the knowledge on the complex process from plasma formation to nanoparticle formation is limited. Each spark consists of a plasma between two electrodes (Fig. 2.1(a)) during the time of typically a few microseconds, the temperature of which is around 20,000 K [15] and higher [16]. This

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