The role of nano-particles in the field of thermal spray coating technology

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Abstract:

Nano-particles play not only a key role in recent research fields, but also in the public discussions about health and safety in nanotechnology. Nevertheless, the worldwide activities in nano-particles research increased dramatically during the last 5 to 10 years. There are different potential routes for the future production of nano-particles at large scale. The main directions envisaged are mechanical milling, wet chemical reactions or gas phase processes. Each of the processes has its specific advantages and limitations. Mechanical milling and wet chemical reactions are typically time intensive and batch processes, whereas gas phase productions by flames or plasma can be carried out continuously. Materials of interest are mainly oxide ceramics, carbides, nitrides, and pure metals. Nano-ceramics are interesting candidates for coating technologies due to expected higher coating toughness, better thermal shock and wear resistance. Especially embedded nano-carbides and -nitrides offer homogenously distributed hard phases, which enhance coatings hardness.

Thermal spraying, a nearly 100 years old and world wide established coating technology, gets new possibilities thanks to optimized, nano-sized and/or nano-structured powders. Latest coating system developments like high velocity flame spraying (HVOF), cold gas deposition or liquid suspension spraying in combination with new powder qualities may open new applications and markets.

This article gives an overview on the latest activities in nano-particle research and production in special relation to thermal spray coating technology.

Introduction

Nanoparticles have been produced in commercial quantities for many years. The most important representatives are carbon black (Cabot, Degussa, Columbia etc.), fumed silica (Cabot, Degussa) and pigmentary titania (DuPont, Millenium, Kerr-McGee, Ishihara), nickel and iron powders (BASF, INCO), nanostructured carbides, nitrides, borides and other non-oxide ceramics (Dow, H.C. Stark) [1].

Anselmann described in an overview article in 2001 many examples of nanoparticles and nanolayers already existing in commercial applications [2], like wet ground mica platelets used as fillers in plastics and cosmetics. Mono-, or polydisperse silica spheres can be covered with TiO_2 nano-particles (see *Figure 1*) to absorb UV light and adapt the refractive index or by Fe_2O_3 to produce magnetic particles [2].

Figure 1: Monodispersed silica spheres (left) and coated with TiO_2 particles of less than 10 nm (right) [2].

1. Thermal Spray Technology

Thermal spray coating technology uses powders according to the European standard EN 1274 [3] or equivalent ISO 14232 [4], or wires, rods and cords according to EN ISO 14919 [5]. These starting materials are introduced in different heat sources like flames or plasma to be melted and accelerated before being splashed at the surfaces of the substrates or components, respectively. Repeated transition of the spray gun across the substrate builds up a dense coating layer from a few ten microns up to millimetre thickness.

During the last century, various spray techniques have been developed, which are described in the European standard EN 657 [6] or equivalent ISO 14917 [7]. Among the numerous coating systems, High Velocity Oxy/Fuel (HVOF) spraying and Plasma Spraying (PS) seem to be the most promising candidates for the processing of nano-materials, due to their high processing speed or high thermal energy.

2. Nanomaterials for Thermal Spraying

2.1. Nanostructured Powders

The oldest Nanotechnology known in materials technology is probably precipitation hardening [8, 9]. The spraying of conventional coatings containing precipitation hardenable species [10-15] offered the possibilities of tuneable nanostructure even before the public discussion on nanotechnology started. The advantage of precipitation hardening lies in the classical utilization of well-established production technologies, but offering the advantage of designable sub-microstructure in a well known base material. These nanostructured materials showed already in the past increased mechanical properties in bulk steels [9, 10, 16] or in reactively thermally sprayed coatings [11, 12, 15].

Figure 2: Metallographic cross section of austenitic steel particles of type Fe17Cr10Mn6VMo containing alloyed Vanadium as most Nitrogen affine element to form secondary nitrides of nanometre size (see AFM picture (right) showing sticking out hard phases of type VN).

The advantage of this class of nano-structured materials is the applicability of conventional production methods like alloying, casting, forging, and of course thermal spraying.

The application of nano-scaled powders needs in general special adaptations of the following process depending on the types of nanoparticles used, whether they are wet or dry or agglomerated.

2.2. Nanopowders

There are different processing routes that can achieve the production of nanoparticles. Nevertheless, there are two distinct ways of approach. One route is called "top down" and can be reached e.g. by crushing and milling large pieces down to nanometre sized particles. The second route follows the so-called "bottom up" approach, e.g. by thermal evaporation of materials followed by condensation and controlled particle growth up to the desired size range. These routes offer tremendous control of the final bulk material properties since the chemical composition, the size and the interparticle spacing can be tailored up from the nano range. Different kind of passivation and functionalisation processes can successfully reduce the effect of undesired nanoparticle cluster growth and give access to 2- and 3-dimensional ordered aggregate structures and new functional materials [17].

One of the routes that offer high purity nanoparticles from precipitation processes are the wet chemical methods.

Wet chemical synthesis:

Nanoparticles with different morphologies can be produced by wet chemical routes. The key parameters controlling the precipitation process in the liquid are e.g. concentration of the solution, pH values and the washing media [14]. These parameters influence the phase transformation reactions and the particles recrystallisation processes. Using special surface controlling agents (SCAs) the nucleation, agglomeration and growth of the nanoparticles can be influenced, or even coating of nanoparticles by a functional layer of a few nanometer thicknesses can be realised [18].

Methods for nano-carbide production such as WC, SiC, B₄C

The traditional method of making cemented carbide powders is by crushing, grinding, and consolidation e.g. by sintering with different binders to micron scale particles. These powders can afterwards be used, i.e. further treated or fed, like any conventional powders. In addition, refractory metal-carbide composites, like WC-Co can be produced by wet chemical routes as well. Starting e.g. with an aqueous solution of CoCl₂ and mixing with H₂WO₄ in ethylenediamine to precipitate crystals of Co₃WO₄ and afterwards reductively decomposition to form nanoporous-nanophase W-Co powder [19]. The conversion to WC-Co is further done by reaction with CO₂-CO gas at pressure of 900 Torr and temperatures between 850 °C and 750 °C within approximately 30 minutes up to 5 hours, respectively. Other carburizing agents can be used such as carbon black (1'400 °C), or gaseous hydrocarbons (CH₄ or C₂H₄) at temperatures below 700 °C [20].

Other economical routes of mass production are e.g. Chemical Vapour Condensation methods.

Combustion Flame-Chemical Vapour Condensation (CF-CVC) was first described by Gleiter and co-workers in 1989 [21]. The concept consists of rapid vapour phase condensation synthesis to produce non-agglomerated nanoparticles by rapid condensation from the vapour phase in a reduced pressure environment. This goal can be achieved by a combustion burner or a hot wall reactor, where the chemical precursors are pyrolysed. The key parameters for a CF-CVC process are gas phase residence time, the temperature of the flames or the hot-wall reactor, and precursor concentration in the carrier gas [22, 23]. Recent studies showed the scalability of the Chemical Vapor Condensation process with production rates of up to 50 g/h by replacing the hot-wall reactor with a flat flame combustor, even operable at low pressures [22]. Nanopowders of SiO₂ produced by this process showed a surface area of 270 - 360 m²/g and nano-TiO₂ of 70 - 80 m²/g [22]. Singhal and co-workers have been seeking for different crystallographic orientations and minimised aggregation effects in flame synthesized V₂O₅ nanoparticles [24]. Theoretical understanding of the nanoparticle growth in gas phase processes was proposed by Kruis et al. in 1993 by a simple model describing the evolution characteristics of aggregates due to coagulation and sintering [25]. This model can be adapted to other thermal processes like plasma evaporation and condensation or other particle material species [26, 27].

State of the art nanoparticles produced by flame pyrolysis processes and available on market for a long time are e.g. SiO₂ or TiO₂ (Aerosil®, Cab-o-sil®). They have been used in the past even in thermal spray business mainly as fluidisation aids (in small mass amounts) for non- or poorly flowable thermal spraying powders. The high surface areas

act as “nano-ball-bearings” between the often blocky particles and the low mass content necessary to increase the flowability of the macro sized spray powders significantly (typically below 2 %-wt) is often below the detection limit. Another important gas phase process used in the past is the carbothermal reduction synthesis.

Rapid carbothermal reduction (RCR) synthesis of ultra fine carbide powders was one of the most efficient and promising production ways in the 1990's [28-30]. High surface area carbon powders of Boron, Tungsten or Silicon can be produced within less than one second at temperatures exceeding approximately 2'200 K [28]. Weimer and co-authors produced B₄C powders by the RCR process, that showed a high surface area between 12 and 32 m²/g and particle morphologies similar to other gas phase routes [28]. The most important steps of the formation of boron carbides by the carbothermal reduction process of boron oxide seems to be the phase change from solid to liquid to gaseous boron sub-oxides and the effect of reactive environment (like chemical composition, heating rate and final temperature) on the ending product.

The production of 5–12 nm carbon material (W₂C) with ultrahigh surface area (measured by BET method of about 3'234 m²/g), by the carbothermal hydrogen reduction (CHR) process at temperatures of 900 °C, was reported by Liang et al. [30]. However, the resulting carbide surface is often contaminated by polymeric carbon from the pyrolysis of the carbon-containing gases (like e.g. CH₄, C₂H₆, C₄H₁₀ or CO) and this polymeric carbon is difficult to be removed. An alternative way of nanoparticle production uses microwaves as energy source.

Microwave processing of nanoparticles at standard industry frequencies of 0.915 and 2.45 GHz is described by different authors [31-36].

The processing steps are similar to the other above mentioned gas phase processes: The precursors, typically chlorides, carbonyls or metal organic compounds, are introduced as vapours into a reaction tube - made typically of quartz glass - directly in front of a plasma zone created by high electromagnetic fields in the microwave cavity. In the reaction zone, the precursors will be at least partly dissociated and ionised and nanoparticles are formed on cooling. The particles leave the microwave plasma with high electrical charges of the same sign thus avoiding agglomeration [36]. Even coated nanoparticles can be produced using directly a subsequent second plasma process. Thus the interaction of the particles can be adjusted using a polymer based nano-coating such, that the cores are kept on well-defined distances [36].

2.3. Nanoparticle Suspensions

The preparation of stable suspensions is a prerequisite for any further processing [37]. Schuetz and Piesche proposed a mathematical model for calculating floc size distributions for coagulating particles (dispersed quartz) in aqueous suspension (demineralised water with sodium hydroxide NaOH and calcium chloride CaCl₂) with turbulent flow, which showed the significant influence of the local energy input in the coagulation reactor [38].

Kebinski et al. showed that the heat flow in suspensions with nano-sized particles, so called nanofluids, can be increased by factors of hundreds or thousands compared to conventional heat-transfer fluids, which could not only be explained by Brownian motion, but rather by ballistic phonon transport [39]. This effect would give reason to the advantage use of nanoparticle suspensions for thermal spraying, as this seems to be the only way, how the heat of an external heat sources, like in plasma processes, can easily be transported within the suspension.

3. Thermal Spraying of Nanomaterials

One of the problems to overcome with nanoparticles is the momentum needed to fully penetrate a flame or plasma for further treatment, i.e. melting, accelerating and splashing on the component. As the diameter of the particles decreases, the aerodynamic diameter is becoming the dominating factor. The inertia of the nanoparticles gets small and therefore they will follow nearly perfectly any flow field and never fully enter the plasma to be further treated in.

This problem can be solved either by a preliminary step of soft particle agglomeration or by mixing the nanoparticles in a suspension. The liquid of the suspension then reacts as a carrier and may even contribute additional energy to the process.

The suspension, containing the nanograins, is often injected radially in the plasma jet followed by a vaporisation process. The control of the injection conditions of suspensions containing nanograins of alumina in a DC plasma was described e.g. by Wittmann [40]. During in-plasma flight, the liquid is immediately vaporised and melting,

agglomeration and sintering of the nanograins starts. Poirier et al. suggested first a model of aerosol flame spraying and impact processes of submicron particles [41, 42].

Bouyer, Gitzhofer, and Boulos presented in 1996 a parametric study of suspension plasma sprayed hydroxyapatite. Another promising application was the preparation of perovskite powders and coatings by RF-suspension plasma spraying [43] for solid fuel cell applications. The coatings obtained by suspension spraying are showing mainly an open porous structure (*Figure 3*).

4. Results and Discussion

Worldwide, many research groups are working on the use of nanoparticles for thermal spraying. Most of them are following the liquid suspension route [40, 41, 44-56], where the liquid has to be continuously pumped to the thermal spray torch. A very precise injection into the plasma is important for the following pyrolysis and nanoparticle formation process. A prerequisite using liquid suspensions is the stabilization of the suspension against sedimentation and agglomeration. The materials used in suspensions are mainly oxides like calcium phosphate (hydroxyapatite) delivering porous coatings for biomedical applications, Alumina or Zirconia. The coatings produced are showing often a nano- up to submicron porosity, which in case of porous layers for fuel cells or biomedical applications can be advantageous due to their high exchange surface areas.

Figure 3: Nanoporous coatings obtained by liquid suspension plasma spraying of ZrO_2 [57].

The use of precipitation hardenable materials was mentioned earliest [11, 12, 16, 58]. Their use facilitates production technologies like thermal spraying, because no special feeding mechanism is needed. The use of reactive plasma species like N_2 or C_2H_4 induces the precipitation process forming nitrides (*Figure 4*) or carbides, respectively.

Figure 4: Left: Thermal sprayed coating using prealloyed and precipitation hardenable powders type Fe17Cr10Mn6VMo during reactive vacuum plasma spraying. Dark spots on the right picture represent the locations of Nitrides after wet chemical etching.

5. Conclusions

Nanoparticles play already a key role in many technological fields like fillers and pigments in paints and lacquers. Within thermal spraying technology so far, the use of nanoparticulate starting materials is mainly concentrated at universities and research laboratories level.

Nevertheless, promising coating properties shown at laboratory scale may be interesting candidates even for mass production. The question of costs and availability of starting materials needs to be addressed and clearly answered, as well as the aspect of process efficiency and environmental concerns.

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