The Pulsation Reactor Process - Large Scale Production of Nano-Sized Metal Oxide Powders in a Single Step

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Abstract

IBU-tec GmbH & Co. KG – a German company with core competency in materials and process development is specialized in developing mixed oxide nano powders. The proprietary pulsation reactor technology is an innovative thermal process capable of producing tailor made nano particles in a single step. This special development is therefore a promising alternative to multi step production systems.

Keywords: pulsation reactor, mixed oxide, composite material, thermal process

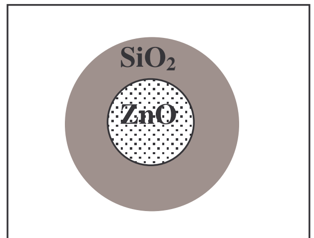


Figure 3: Composite material, combines UV-protection and scratch resistance.

Introduction

The production of non agglomerated nano particles in large amounts at reasonable production costs summarizes some of the most important issues in the nano business.
Traditionally the sol-gel process has been a flexible and powerful tool when it came to tailoring nano particles. Size distribution, pore sizes or surface area could be relatively easily controlled by adjusting reaction parameters such as concentration, porogens or pH. On the other hand the upscaling of the production is not always easy and can influence the reproducibility and several steps (synthesis, washing, drying, calcination) are necessary.
Alternatively we offer a very fast single step production in a pulsation reactor. This has several advantages. The energy needed for the thermal process can be kept relatively low since the average residence time per particle is less than 1 second. The flexibility in educts (solutions, aerosols or solids) offer the possibility to optimize the costs and consider environmental issues (water as solvent). Enhanced powder characteristics (catalytic highly active) due to the special production process are possible.

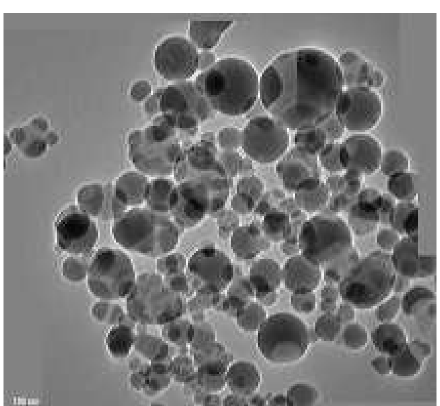


Figure 2: Powder produced in a pulsation reactor

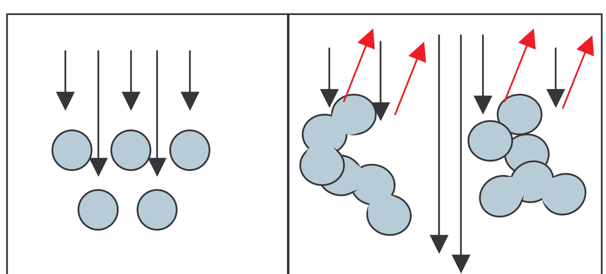


Figure 3: comparison of non-agglomerated (left) and agglomerated (right) particles as UV-light protection.

The Pulsation Reactor

The major backdrop of large production in thermal processes is quite often the difficulty in producing non agglomerated nano particles. Agglomeration during a thermal process is afterwards generally hardly made undone. But agglomerated powders show several disadvantages (scheme 3) compared to non agglomerated nano particles. The total size of the particle increases and transparency decreases. Further more less amount of non agglomerated powder is needed to obtain an efficient UV protection. In the following we show that it is possible to produce non agglomerated nano metal oxide powders via a pulsation reactor.

The principle of a pulsation reactor is similar to that of a pulse jet.

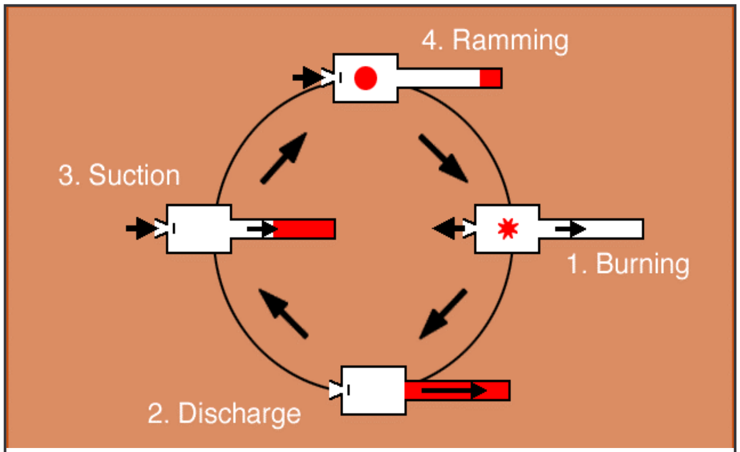


Fig. 4: Principle of a pulsation reactor.


Figure 5: Resonance tube.

The pulsation creates a "flameless" burning process which guarantees that the temperature inside the reactor is very homogenous this ensures monodispers powder characteristics at high throughput (figure 4).

A second result from the pulsation is a high energy impact. Therefore it is possible to synthesize materials at much lower temperature than normally needed. The combination with very short residence times is the key to catalytic highly active materials.

The technology of a production facility (figure 5 and 6) illustrates the possibilities to inject either liquid, gaseous or solid raw materials into the combustion chamber or the resonance tube.

A shock cooling in the filter stops the powder reaction at a definite time and unwanted chemistry can be more easily controlled. A well chosen combination of chemical know-how, educt injection, process conditions and filter system can minimize agglomeration of the final product.

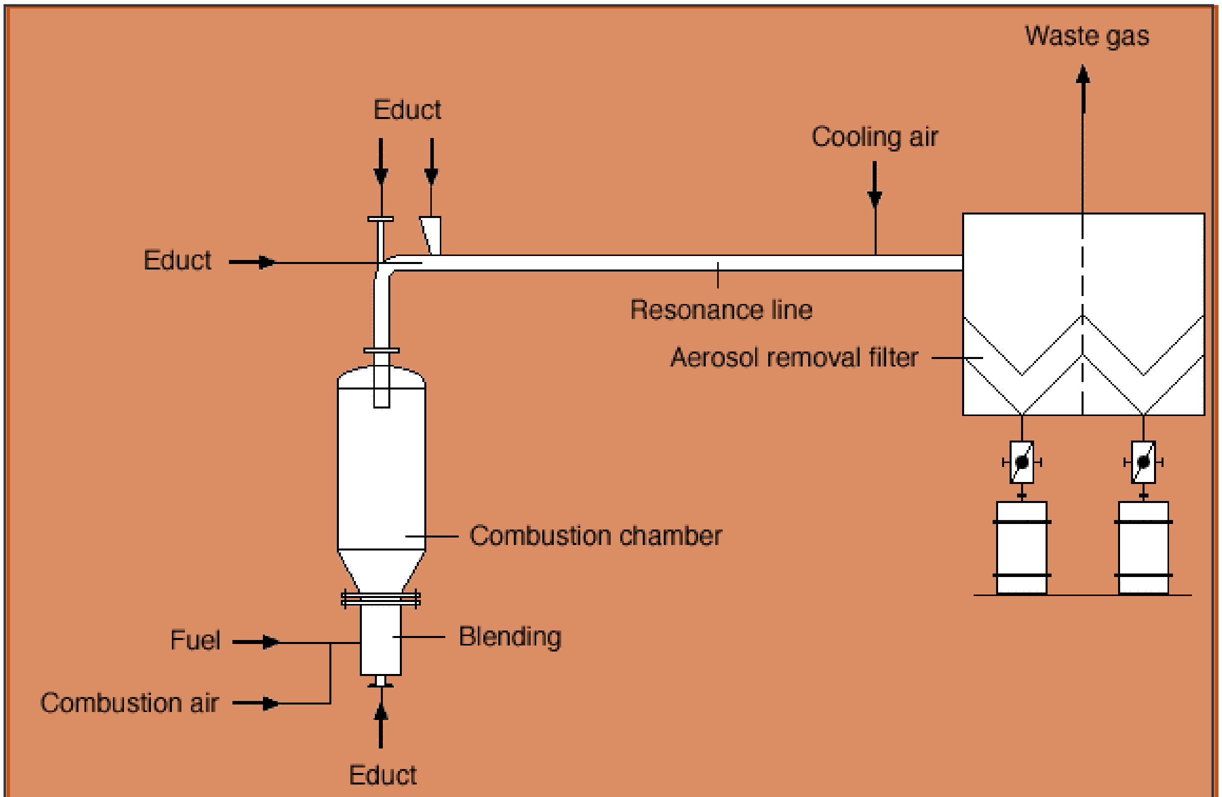


Fig. 6: The pulsation reactor, an extremely flexible production system.

There is a number of different possibilities to influence the reaction. Besides the type and speed of the gas flow (O_2, H_2, N_2, CO, CO_2,CH_4) the length of the resonance tube can be varied. The temperature of the resonance tube can be cooled or heated enabling a temperature gradient between combustion chamber and resonance tube. It is very interesting to inject the educts at different positions. Of course it is possible to inject two or more educts either at the same place or apart from each other to produce a mixed oxide or a core shell material. At the end of the resonance tube the material undergoes shock cooling to ensure a definite reaction time and reproducibility.

The flexibility of this system enables us to produce tailor-made powders concerning particle size, shape, stoichiometry and phase composition (figure 7). Furthermore composite materials and / or core shell materials can be produced combining advantages of different materials e.g. UV-protection and scratch resistance.

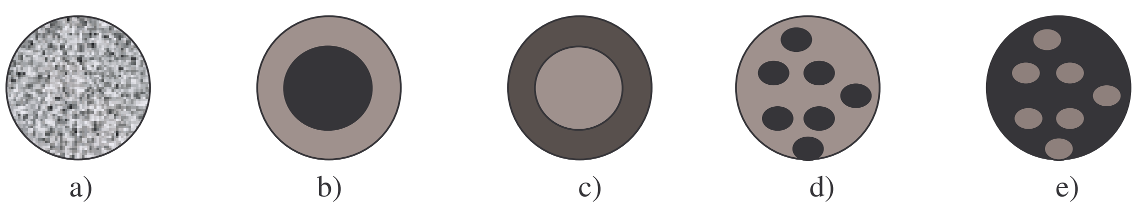
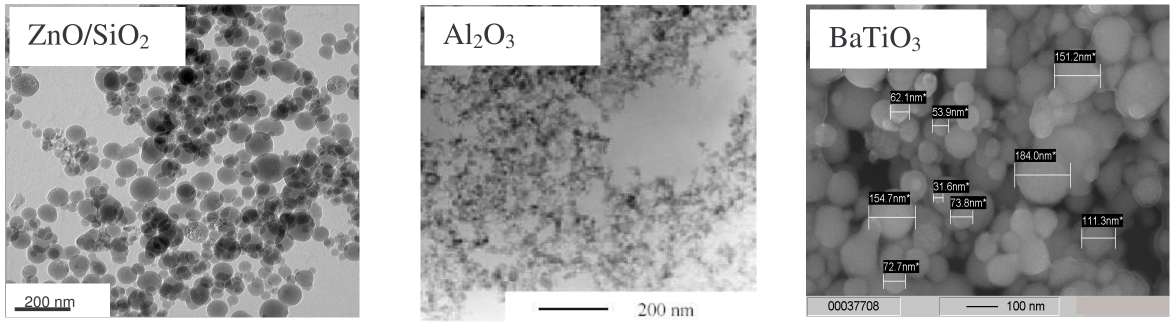


Fig. 7: a) mixed oxide; b) and c) core shell; d) and e) multiple core shell



ZnO/SiO_2 composite particles
UVB light adsorption
scratch resistance
also possible:
ZnO/Al_2O_3
$TiO_2/ZnO/Al_2O_3$

Al_2O_3
alpha-phase
gamma-phase
scratch resistance
anti abrasiva

$BaTiO_3$
catalysis
tetragonal phase
spherical
custom-made

Fig. 8: Different metal oxides synthesized via pulsation reactor and possible applications.

Applications

Metal oxides can be custom-tailored. Depending on the particle characteristics a broad field of applications can be found. The combination of zinc oxide and silica (figure 8) results in an interesting additive for automotive coatings. The interesting chemistry of zinc oxide offers additional possibilities in cosmetics.

The extreme hardness of corundum is an advantage in abrasives but used as an additive make it an anti abrasive or can give paints a high scratch resistance.

It is always the customer who tells us which properties our powder shall have. The table below is therefore just an exemplary overview of some powders produced with a pulsation reactor.

Table 1: Examples of materials produced at IBU-tec GbmH & Co. KG.

	Surface area (m²/g)	Phase composition	Particle size (nm)	Color
Al_2O_3	15	α- Al_2O_3	100-300	white
Al_2O_3	150	mixed	20-40	white
TiO_2	106	Anatas	5-50	white
TiO_2	37	Anatas/Rutil	40-60	cream
ZrO_2	14	mixed	10-50	white
Mullit	12-16	>90% Mullit	50-150	white
ZnO	25	Zincite	50-70	white
ZnO/SiO_2	34	SiO_2 /ZnO core shell, amorph	10-80	white
SiO_2	120	amorphe	10-30	white
$BaTiO_3$	12	tetragonal	30-60	white