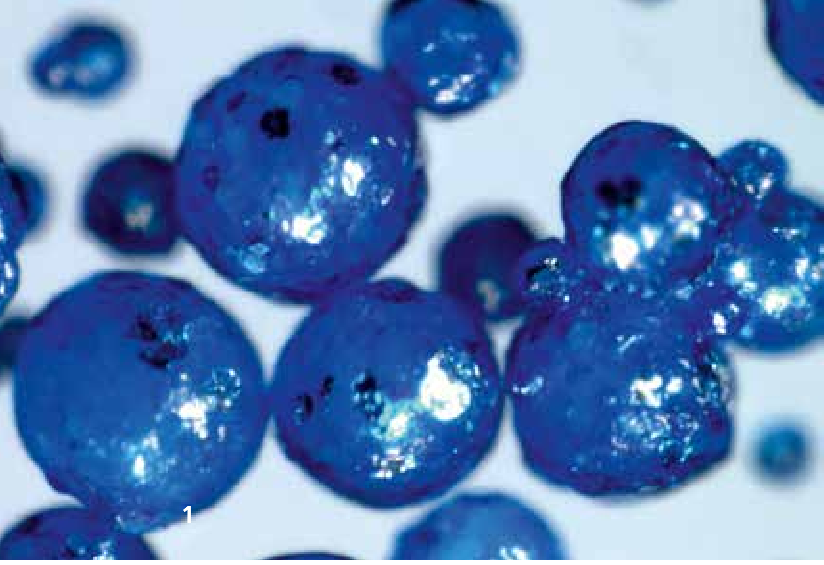
Fraunhofer ICT

FRAUNHOFER INSTITUTE FOR CHEMICAL TECHNOLOGY ICT

ENERGETIC MATERIALS – PARTICLE TECHNOLOGY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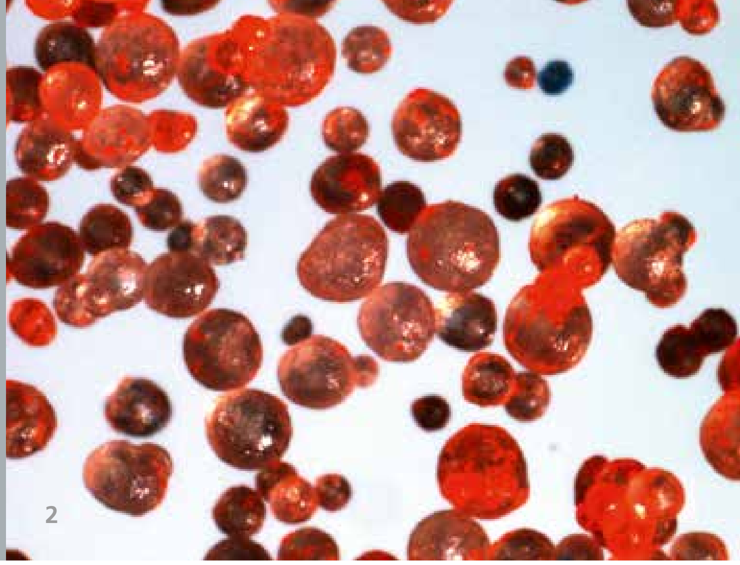
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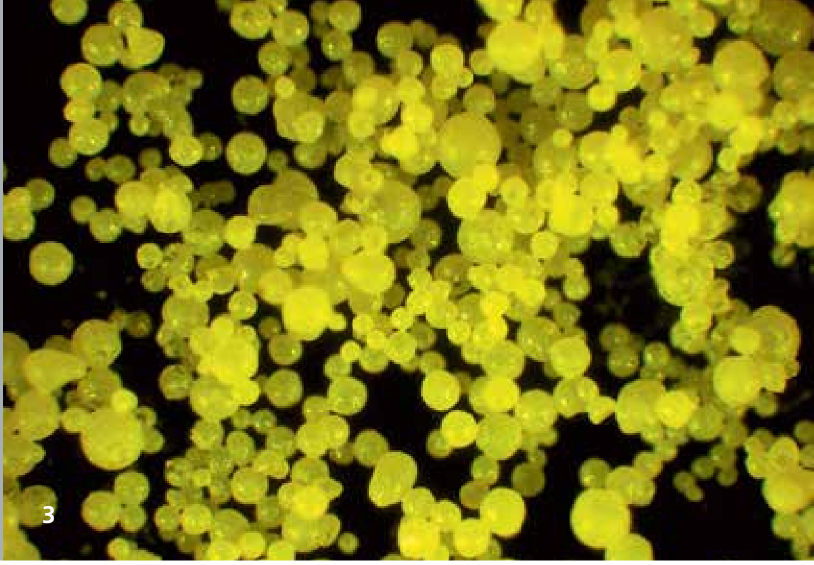
Due to the wide variety of industrial products available in particle form, particle technology has a high technological and economic significance. For many years the Fraunhofer ICT has used particle technology to shape the product properties of energetic materials. The experience gained with particulate systems paves the way for new fields of application and enables the innovative solution of problems.

The research group for particle technology works on a range of topics from production and characterization through to the processing and application of particulate products. In the area of defense research, energetic components are purified and tailored to meet the specific requirements placed on propellants or explosives. Besides drying, crystallization and milling processes, spray and emulsion methods, micro and nanocomposite processes and coating technologies are also applied. Particle and crystal structure analyses also form part of the group's portfolio.

PARTICLES IN A SAFE ENVIRONMENT

At the Fraunhofer ICT, particle technology has long been used as an effective way to design products with specific properties, especially energetic materials. A special infrastructure is available for the preparation of explosion-sensitive, high-cost and often hygroscopic substances, allowing rigorous requirements for safety and purity to be met. To meet safety requirements, the units are installed in an explosion-proof safety work room. They are remote-controlled and are operated with conditioned processing air. Various different processes are available for the manufacture and modification of particles, depending on the material system and desired properties of the particle collective.



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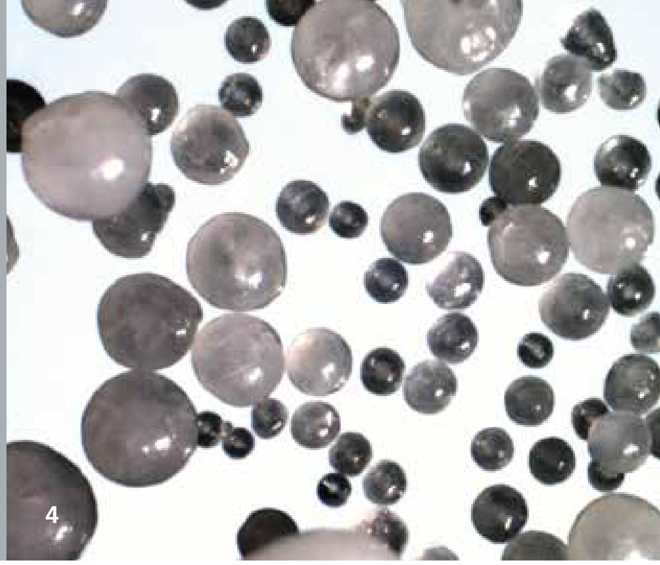

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1+2 *Coated ADN prills.*

3 *ADN/FOX-7 composites.*

4 *SCAN prills.*

COVER PHOTO

SCAN/Laropal® prills.

CRYSTALLIZATION

The Fraunhofer ICT's know-how in the field of cooling crystallization ranges from the measurement of fundamental crystallization data (solvent screening and measurements for solubility curves) to new developments and the optimization of crystallization processes. Solids with a suitable melting behavior and sufficient stability in the melted phase can be reshaped into prefered spherical morphologies using, for example, emulsion crystallization or prilling processes. The aim of the crystallization process is the production of so-called "smart materials" – particles with specific collective properties such as consistent particle size and morphology, heightened purity and increased mechanical and chemical stability. The production of insensitive energetic materials is also becoming increasingly important.

SMART MATERIALS

ADN prills

The environmentally-friendly oxidizing agent ammonium dinitramide (ADN), which is currently in high demand, is processed into prills (spherical particles) with the help of emulsion crystallization. ADN is an oxidizing agent for fuels with a melting point of 93 °C. Through emulsion crystallization the component is processed to form so-called ADN prills with an average size of, for example, 120 µm. Chemical stabilization of the raw material is necessary to ensure non-porous prills with good processability. The material is used, for example, for the development of cutting-edge propulsion systems for space vehicles. Researchers are currently working on a way to replace the conventional batch production process by a (quasi) continuous process. This would lead to lower manufacturing costs and would allow for production on a technical scale.

PSAN

An additional Fraunhofer ICT product is phase-stabilized ammonium nitrate (PSAN) which, like ammonium perchlorate, is used as an oxidant in solid propellants for rocket engines and gas generators. It is produced in the pilot plant by spraying a melt containing added stabilizers at temperatures of approx. 180–190 °C. The standard particle sizes are 30 µm, 50 µm or 160 µm. Using an atomization unit available on-site quantities of 50 kg can be processed. By incorporating foreign ions into the crystal lattice of the ammonium nitrate, its solid-phase conversions can be prevented or modified so that the product becomes volume-stable.


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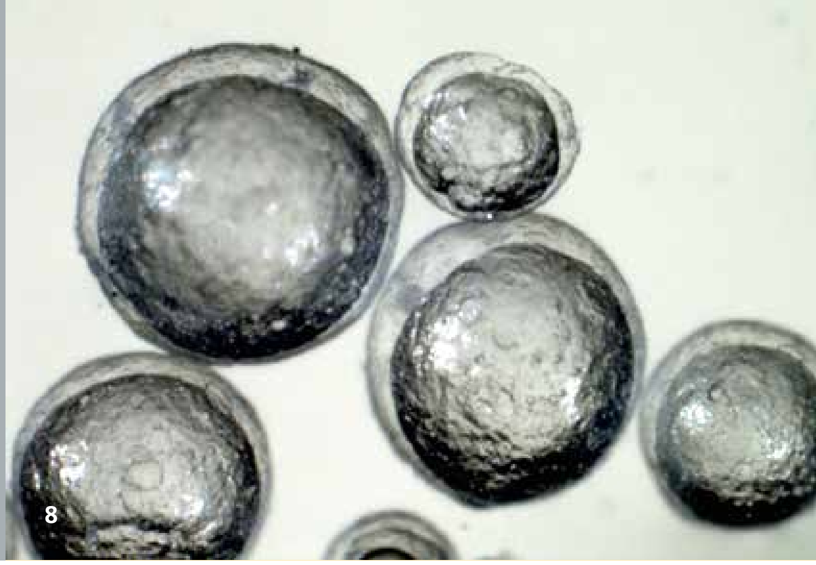
MILLING PROCESS

Wet milling

For the wet milling process, toothed-ring dispersing devices, an annular gap ball mill (Hosokawa Alpine 50 RSK) and a basket mill / dissolver combination (VMA-Getzmann DISPERMAT AE06-C1-Ex) are available. The toothed-ring dispersing device covers a particle size range of 10 to 30 µm. The DISPERMAT is suitable for micromilling or dispersion of highly viscous suspensions. An advantage is that, besides water, a variety of organic solvents and many other fluids can be used as suspension media for all appliances. Special adjustments for explosion protection enable the production of energetic materials such as RDX, HMX, FOX-7, and FOX-12 with particle sizes from 1 to 5 µm. On the other hand, nanoscale materials such as "carbon nanotubes" (CNTs) can be dispersed in almost any medium, or processed to form gels. This makes it possible, for example, to comminute high-risk material in the matrix to be processed, and it eliminates the need for an additional step to dry the particles and transfer them to another medium, which might have a negative effect on the dispersity. By this means the risk of contamination is significantly reduced.

Dry milling

An oscillating mill, a pin mill and a jet mill are available for dry milling. Using the pin mill, ammonium perchlorate, ammonium nitrate or potassium nitrate can be milled to obtain particle sizes in the region of 5 to 10 µm. The special infrastructure at the Fraunhofer ICT enables substances such as the highly hygroscopic oxidant ammonium perchlorate to be milled using the jet mill. Here particle sizes of 2 to 3 µm with a very narrow particle size distribution and a throughput of up to 10 kg per hour can be achieved.



PARTICLE COATING

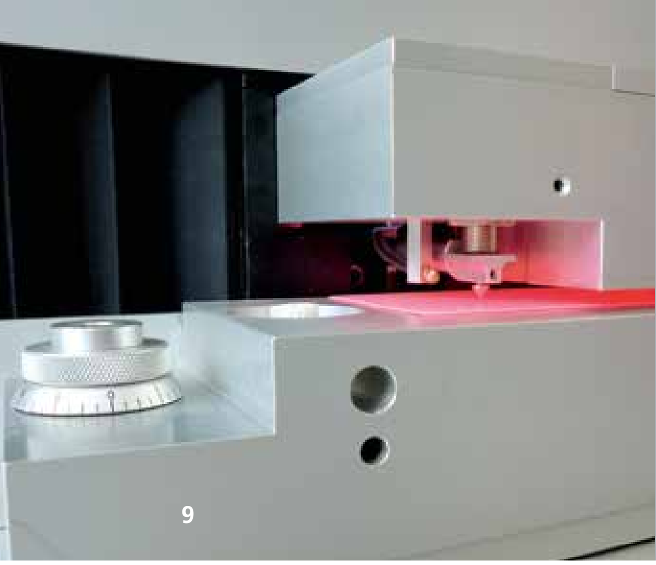
Manufacture of core-shell particles

A further process in particle refinement is their coating using fluidized-bed technology. Thin coating layers for tailored surface properties are applied to individual particles while avoiding agglomeration. The modified fluidized-bed units at the Fraunhofer ICT allow the processing of explosion-sensitive and extremely hygroscopic core materials such as ammonium dinitramide (ADN) and ammonium nitrate (AN). Possible coating materials include dissolved polymers or dissolved crystalline substances. The dissolved coating material is sprayed onto the fluidized particles in the bed using a nozzle system. The appropriate solvents for the coating materials can be either aqueous systems or a variety of different organic solvents. Pre-dried air or nitrogen can be used as a process gas. The devices used have a capacity for batch sizes from 200 g up to 5 kg. The aim is to improve the particle properties of the core material through encapsulation with small quantities of coating materials.

This allows the following improvements:
- Increased compatibility compared to reactive substances such as isocyanates
- Protection of hygroscopic materials from humidity and moisture
- Decrease in the sensitivity of energetic materials (desensibilization)
- Functionalization of particle coatings or composite particles by incorporating (nanoscale) active substances such as stabilizers, combustion modulators, bonding agents, conductive substances, CNTs etc.
- Increase in mechanical strength

Besides particle coating, fluidized-bed technology can also be used for spray drying, granulation and for the production of composite materials in which the generated matrix can contain embedded micro- or nanostructures. As an example, composite particles with a reduced sensitivity can be produced with a combination of 90 % octogen and 10 % FOX-7 composite particles.

5 *DISPERMAT® AE06-C1-Ex with mounted immersion mill TML-1.*
6 *Preparation for the milling process.*
7 *Fluidized bed coater during the coating process.*
8 *Core-shell particle under the macroscope.*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PARTICLE CHARACTERIZATION

The following methods are used to characterize the product properties:

- Measurement of the average particle size and the particle size distribution using laser diffraction spectroscopy (Malvern MASTERSIZER 2000) for particle sizes from 0.1 to 2000 µm
- Zeta potential measurements and particle size analysis using photon correlation spectroscopy (Malvern ZETASIZER 3000) for particle sizes of less than 1 µm
- Measurement of crushing strength of individual particles with an automatic granulate strength testing system (etewe GFP)
- Measurement of the specific surface of particle collectives using the BET method (Quantachrom NOVA 2000e)
- Density measurement of particle collectives using gas pycnometry (Quantachrom ULTRAPYCNOMETER 1000)
- Water determination using Karl Fischer titration (Metrohm 795 KFT Titrino and 703 Ti Stand)
- Optical particle analysis with a macroscope (Leica Z16 APO) using image analysis and a focal depth module.

Additional analytic techniques available are:

- Electron microscopy
- X-ray analysis
- Spectroscopy
- Chromatography
- Thermal analysis
- Rheology
- Mechanical testing
- Microcalorimetry, impact and friction sensitivity

11

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OUR SERVICE OFFER

- Stabilized ammonium dinitramide prills produced by emulsion crystallization with an average particle size of 120 µm
- Phase-stabilized and spray-crystallized ammonium nitrate (PSAN or SCAN) with spherical morphology, produced in standard particle sizes of 30, 50 or 160 µm in batch operations at 50 kg per batch
- Particle refinement through comminution, crystallization, fluidized-bed and coating methods
- Comprehensive particle characterization

9 *Testing the crushing strength of a granulate sample.*
10 *Laser light diffractor.*
11 *BET device.*
12 *Gas pycnometer.*
13 *Karl-Fischer titrator.*

OUR AREAS OF EXPERTISE

Processing
- Crystallization
- Agglomeration
- Atomization
- Micro-encapsulation
- Comminution
- Drying

Characterization
- Density
- Particle size analysis
- Granulate strength test
- Specific surface
- Determination of water content
- Microscopy

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PARTICLE TECHNOLOGY

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