

# New Challenges in Thermal Processing

Christian Klaus<sup>1</sup>, Dr Karl Wegner<sup>1</sup>, Thomas Rammelt<sup>1</sup>, Dr Matthias Ommer<sup>1</sup>

Ibu-tec specialises in the thermal treatment of materials in rotary kilns and pulsation reactors. For more than 40 years, the company, which is based in the German city of Weimar, has provided services that include developing processes in the lab, scaling up to production level and contract and toll manufacturing in industrial quantities. It also executes complex material and waste recycling processes on behalf of its customers.

The development of new materials and the improvement of existing products both make an important contribution to our economic growth. Certain applications require precisely defined material properties that are often difficult to achieve. One of the major challenges in this context is to scale up laboratory syntheses to a production volume while retaining the necessary material characteristics, because this is often time-consuming and costly.

One process in particular that this applies to is the flame spray pyrolysis. The considerable investment involved in developing and maintaining production on a scale larger than that of a laboratory often makes the prospect of scaling up and continuing with projects unattractive, despite the fact that this is a promising process which is capable of retaining the existing properties of the material. The few existing plants used for industrial purposes are generally not available for small production runs and development projects.

One option for scaling up the process is the use of pulsation reactors. They can close the gap between flame spray pyrolysis in the laboratory and production on a scale of millions of tonnes.

## Flame spray pyrolysis

In the flame spray pyrolysis (FSP) process, an organic raw material solution (the precursor) is sprayed to produce an aerosol and ignited using a supporting flame. Nanoparticles are formed in a self-sustaining flame and are separated by a filter [1] [2]. Typical process parameters are the fuel/oxygen ratio, the precursor concentration, the dispersion pressure drop and the material throughput.

Flame spray pyrolysis processes with new materials are generally carried out in university institutions, which means that there are rarely any opportunities for increasing the throughput. As a result, the existing development potential cannot be adequately exploited.

## Pulsation reactors

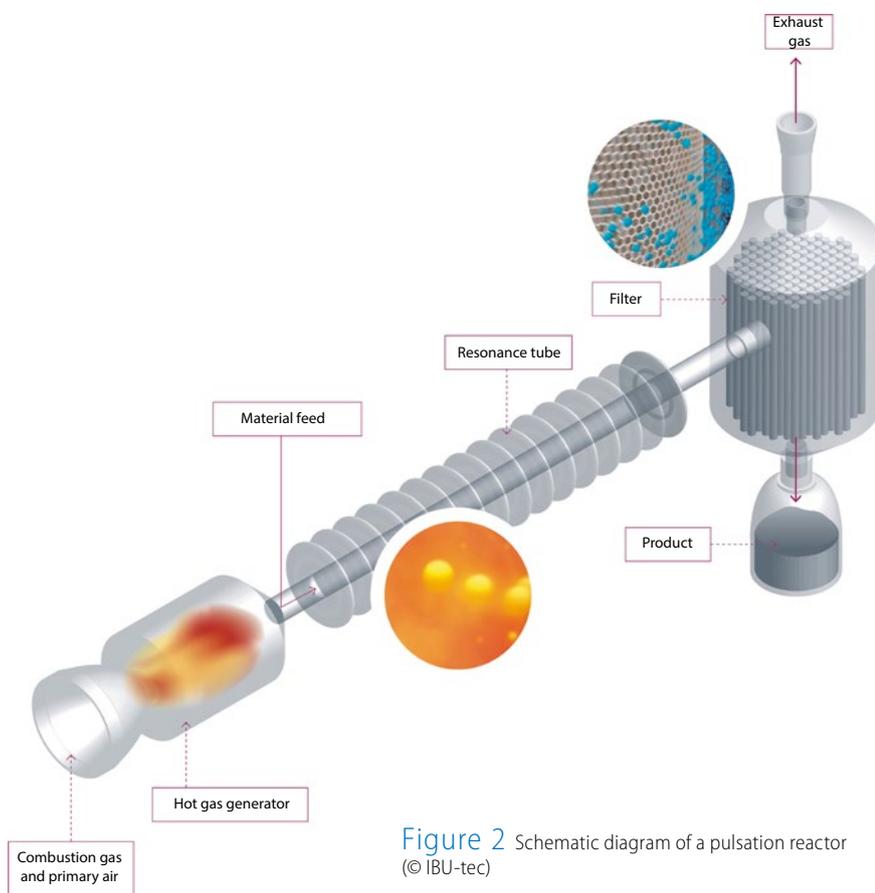
Pulsation reactors have a pulsating flame and are based on the same principle as an air flow reactor. The material is treated in a flow of pulsating hot gas for a residence time of just

a few seconds and at temperatures between 250 °C and around 1300 °C. The material can be supplied in the form of a suspension, a solution or a powder. The key process parameters are the temperature, the pressure amplitude and frequency of the pulsation, the feeding rate and the residence time. Ibu-tec operates pulsation reactors of different sizes ranging from flexible, modular pilot systems up to production plants (Figure 2).

A gas mixture is ignited in the combustion chamber and the resulting overpressure is released into the resonance tube. The inertia of the outgoing gas flow causes a vacuum



Figure 1 Aerial photograph of the Ibu-tec site (© IBU-tec)



**Figure 2** Schematic diagram of a pulsation reactor  
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to form briefly in the combustion chamber which draws in new fuel. This ignites and the subprocess starts all over again. Depending on the design of the plant and the parameters, this subprocess is the starting point for the underlying frequency of the pulsation reactor which ranges from 1 to 500 Hz. The raw material is fed into the resonance tube where the thermal treatment occurs while being transported in the flow of the pulsating gas. After the hot gas leaves the reaction chamber, a cooling gas is introduced to reduce the temperature of the gas flow. Afterwards the product can be separated by an exhaust filter or cyclone. [3]

One advantage of the pulsation process is the homogeneous flow profile, which results in consistent process conditions for all the particles. Combined with the accurate control of the temperature during the treatment and the short residence time, this makes it possible to achieve products with more homogeneous properties than would be possible in conventional air flow reactors.

The treatment temperatures of the flame spray pyrolysis process are higher and the residence times much shorter compared to the pulsation reactor, but the pulsation reactor allows for much more flexibility in the choice of raw material. In addition, up to 150 kg of material can be processed per hour in a pulsation reactor. Even larger quantities are technically feasible, which means that commercially viable volumes can be produced more cost-effectively.

### Transfer of flame spray pyrolysis and scale-up

The challenge was to scale up a process that had been developed on the basis of flame spray pyrolysis to a production volume in the pulsation reactor. This was demonstrated using the example of zirconium oxide. An organic zirconium (IV) solution was used as raw material. The project started with the development of the process on a laboratory scale using a bench-

top flame spray pyrolysis unit (ParteQ GmbH). After the required product properties had been achieved, the process was increased to production scale in the pulsation reactor.

By increasing the concentration of the raw material solution and increasing the input rate by a factor of 13, it was possible to achieve a 20-fold increase in the hourly production volume when compared with flame spray pyrolysis. The pilot plant produced a material with properties in the same range as the equivalent material from the flame spray pyrolysis process, but with a slightly higher specific surface area and a slightly lower residual carbon content. The crystalline phase composition was also comparable. The characterisation of the particles under an electron microscope showed a very similar morphology and size distribution (Figure 3).

In addition to the benefits offered by pulsation reactors for the production of nano-scale powders with very specific properties, this study shows that it is possible to transfer flame spray pyrolysis processes to a production scale using this technology. This opens up new prospects for the manufacture of sample quantities of high-quality, innovative materials or for the start-up of commercial production.

### Material and waste recycling in rotary kilns

Economies throughout the world are dependent on the availability of scarce raw materials, particularly those obtained from geological deposits. In addition, once the easily accessible deposits have been fully exploited, the extraction of these materials is proving to be increasingly difficult, either because of the growing technical challenges or because of legitimate social or environmental concerns. For this reason, the emphasis within industry and society as a whole is increasingly on recovering materials and recycling waste.

Changing economic and social conditions are resulting in companies paying growing attention to processes that were previously peripheral to their main business or were only the subject of research projects.

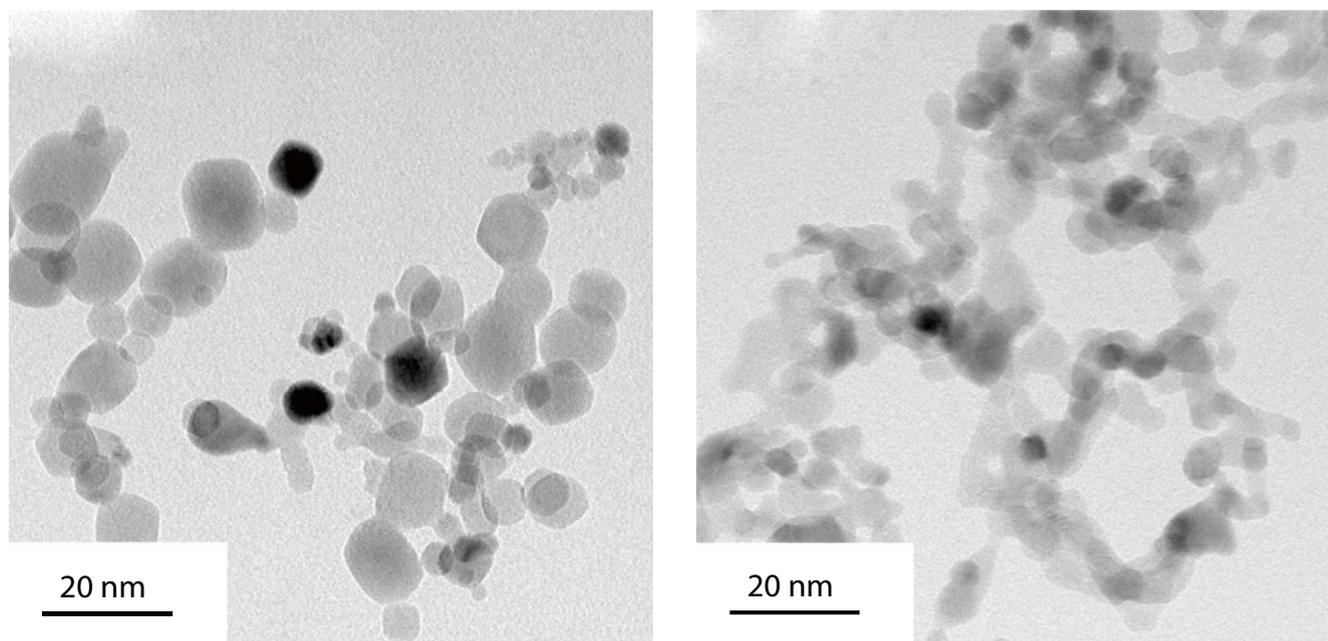


Figure 3 Transmission electron microscope images of  $ZrO_2$  from a flame spray pyrolysis unit (left) and a pulsation reactor (right) (© IBU-tec)

One key example of this is the recycling of construction materials, for example after buildings are demolished or roads are torn up. These materials can be utilized as a substitute for the disappearing resources that have been used in the past. One particular material of this kind is gypsum. The forthcoming phase-out of coal-fired power stations will remove the main source of FGD gypsum, which has previously been produced as a by-product of flue gas desulphurisation. This will result in a serious supply shortage which could otherwise only be compensated for by overexploiting the remaining natural deposits of the mineral. One solution to the problem is to reuse gypsum from demolished buildings.

The need to minimise the  $CO_2$  emissions from the production of concrete is another case of this kind. On a global scale, concrete manufacturing has the largest impact on the climate of any single industry. Increasing the proportion of recycled concrete used as an additive in new construction projects could result in huge reductions in emissions. Other examples of this kind include the regeneration of used catalysts from the chemical industry and the recycling of phosphorus from sewage sludge.

### Recycling in rotary kilns and its challenges

The thermal recovery of waste presents challenges for process engineering, because the materials involved have a wide variety of different properties. For example, sewage sludge is a homogeneous mass with a paste-like consistency, while rubble from roads or demolished buildings consists of fragments of different types of materials with a broad size distribution. In addition, there are significant variations in the chemical composition of the materials.

As a result, the heating value of the material that is being processed varies considerably and this factor needs to be taken into account in recycling projects involving thermal treatment. Therefore, the choice of treatment method plays a decisive role.

The main systems used for the thermal treatment of materials and waste are rotary kilns (Figure 4), fluidised bed reactors and grate-firing boilers. The differences between these systems indicate their suitability for different types of materials.

A rotary kiln is an ideal choice, largely because it can handle a wide variety of waste, with processes that include the treatment of fragmented waste from construction sites

and the combustion of sewage sludge. Neither fluidised bed reactors nor grate firing boilers can treat such a broad range of materials.

If the rotary kiln is designed and engineered for the purpose, it can accommodate a broad spectrum of recycling processes and manage fluctuating throughputs without problems. Grate-firing boilers offer similar advantages, but fluidised bed reactors are less flexible.

Because of the wide range of process parameters (such as atmosphere and temperature), it is possible to use rotary kilns for a variety of processes. This opens up a broader spectrum of applications when compared with other types of system, even if optimising the energy consumption of certain processes can prove to be time-consuming. The combustion of hazardous waste in rotary kilns is a well-established solution. At the other end of the spectrum, for example, is the pyrolysis of used tyres in the absence of oxygen to produce carbon black.

Sewage sludge with a high water content can also be dried and processed or undergo thermal conversion to an inert ash in a rotary kiln. By carefully selecting the process parameters, it is possible to destroy the organic pollutants and even to expel inorganic sub-



Figure 4 The directly fired rotary kiln (GDO) at IBU-tec (© IBU-tec)

stances, such as arsenic and mercury. The result is an ash rich in bio-available phosphorus which can be used as a mineral fertiliser.

## Conclusions

The examples shown demonstrate the relevance of an established technology such as

the rotary kiln in relation to the latest developments in the area of recycling. They also show that relatively unknown technologies such as the pulsation reactor can produce new materials and also allow processes to be scaled up. Both systems are ideal for increasingly challenging tasks in the field of thermal processing.

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*Contact:*  
 IBU-tec advanced materials AG,  
 99425 Weimar, Germany,  
[www.ibu-tec.com](http://www.ibu-tec.com)

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