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Advanced Materials

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1. Introduction

Material science has always affected the public and societies. Numerous innovations arise from findings within this field. Materials played an important role in history. Here are some examples.

- During Tang dynasty, it was discovered that firing clay to high temperatures resulted in a tough, strong, translucent material- in porcelain.
- In 1860, Etienne Lenoir invented the spark plug, which he used in a gas engine of a “Hippomobile”. The property of porcelain to be an electric insulator gave rise to the invention.
- In 1911 superconducting materials were presented for the first time. Since then, superconducting materials, particularly high temperature ceramic superconductors, have been vigorously investigated as they promise groundbreaking future applications in energy transportation and storage.
- In 1981 the Columbia space-shuttle had her successful maiden flight. The re-entry of the space shuttle through the atmosphere causes temperatures up to 1650 °C. Tiles made of highly porous ceramic composites were used to protect the shuttle despite the fact that one can break them by hand.

These historical events point out the versatile properties and constraints of ceramics and show that ceramics can be promising future materials even if they have been known for millennia. Nowadays, advanced materials are used in the electro-, medical-, space- and car industry e.g. are used by all of us every day. This paper highlights how BUCHI products and solutions are used in the following fields:

- Ceramic powder – The influence of ceramic powder quality on the resulting ceramic body
- Battery research – Advanced materials as promising anodes and cathodes in Li-Ion batteries
- Transparent materials research – Fascinating ceramics with versatile properties and applications
- Bioceramics research – The use of advanced materials as bone and dental implants

2. Groups within the vast world of advanced materials

The word ceramic comes from the Greek word for “pottery”. Potteries include earthenware, stoneware and porcelain and are derived from clay, granite, feldspar and glass.

Nowadays, ceramic materials include much more than just pottery. In contrast to traditional ceramics, advanced ceramics are mainly based on oxides and non-oxides. In the light of the superior properties of these types of ceramics, they are referred to as advanced materials, advanced ceramics, high-performance ceramics or technical ceramics.

Table 1 shows some of the raw materials and the properties attributed to advanced materials made from them.

Name	Aluminium oxide	Zirconium oxide	Silicon carbide	Silicon nitrides
Chemical Formula	Al_2O_3	ZrO_2	SiC	Si_3N_4
Category	Oxides	Oxides	Non-oxides	Non-oxides
High firmness	●			●
High toughness	●		●	●
Temperature stability	●	●		
Wear resistance	●		●	●
Corrosion resistance	●		●	
Flexural strength		●		
Fracture toughness		●		
Wear resistance		●		
Low thermal conductivity		●		
Tensile strength		●	●	
Thermal shock resistance			●	●
Low thermal expansion			●	●
High thermal conductivity			●	
Semiconductor properties			●	●
High chemical resistance				●

Table 1 – Selection of raw materials and the properties attributed to the advanced materials made out of them. The list is not complete.

The entirely different properties of these advanced materials determine the use in various applications.

- Aluminium oxide is used for watch displays, capacitors, vehicle armor etc.
- Zirconium oxide is used for refractory ceramics, dental implants, femoral heads etc.
- Silicon carbide is used for diodes, sensors, space telescope mirrors etc.
- Silicon nitride is used for bearings, cutting tools, passivating agents in solar cells etc.

3. The ceramic manufacturing process

The manufacturing process for ceramic is basically similar to most types of advanced materials. It starts by the production of raw material and subsequent powder conditioning. This is where spray drying plays a significant role. These processes are followed by pressing the powder into a “green body” (an object whose main constituent is weakly bound ceramic material) and shaping it. There is a variety of powder compaction techniques such as uniaxial pressing, isostatic pressing, injection molding, extrusion, slip casting, gel casting and tape casting which are not discussed at this point.

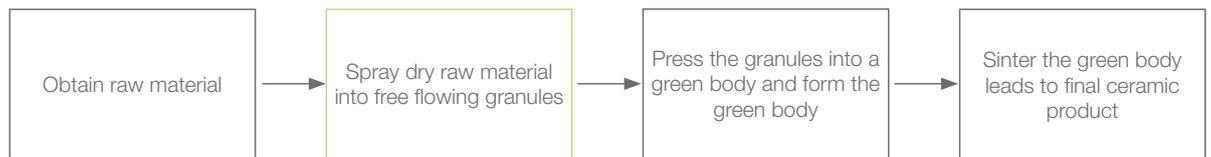


Figure 1 – Manufacturing Process

As a final step, the shaped green body is sintered and finished. This results in the desired ceramic product. As there are many different powder compaction techniques, there is also a myriad of different sintering techniques such as liquid phase sintering, spark plasma sintering, pressureless sintering and more.

4. The importance of spray drying in advanced materials manufacturing

Primary material for advanced materials can be produced in two fundamentally different approaches known as the “bottom-up” and the “top-down” approaches.

A bottom-up approach starts by wet chemical synthesis of particles which subsequently can be transformed into granules by spray drying. This results in powders of high purity with well-defined properties.



Figure 2 – Bottom-up approach to gain raw material

In a top-down approach, feedstock material is ground to fine particles; typically by ball milling, vibratory milling, jet milling or other comparable milling techniques. Depending on the grinding technique, this can either result in dry fine powders or in suspensions of fine particles which subsequently are transformed into granules by spray drying.



Figure 3 – Top-down approach to gain raw material

4.1. Granulation by spray drying

Granulation and controlled agglomeration refer to the process of forming granules out of fine particles. Granules of several μm are achieved by mixing the raw material with binders and dispersants and subsequent spray drying. Granulation by spray drying is by far the most important granulation method although several others exist. [1]

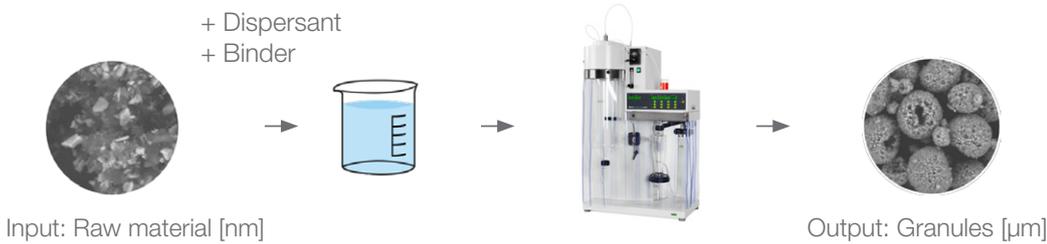


Figure 4 – Granulation by spray drying

Since fine dry particles show poor flow ability, it is necessary to agglomerate these particles into larger granules. Larger granules clearly show a better flow ability and are much easier to handle. This makes the material applicable to industrial processes. Granules are furthermore less hazardous to human health compared to fine dust since they cannot be inhaled and therefore do not collect within the lungs. [1]

4.2. Composite forming by spray drying

Composite forming by spray drying is a process where a material is included into a matrix. The matrix material envelops the guest material to form a composite. As an example, Vail et al. made a polymer-encapsulated ceramic powder, where silicon carbide powder was encapsulated in a poly(methylmethacrylate) latex polymer. [14] Another example is given by Costa et al. who introduced a hematite pigment within a transparent ceramic matrix of silica or zirconia. [15] Both researchers spray dried a slurry made up of the two components which resulted in granules of composite material.

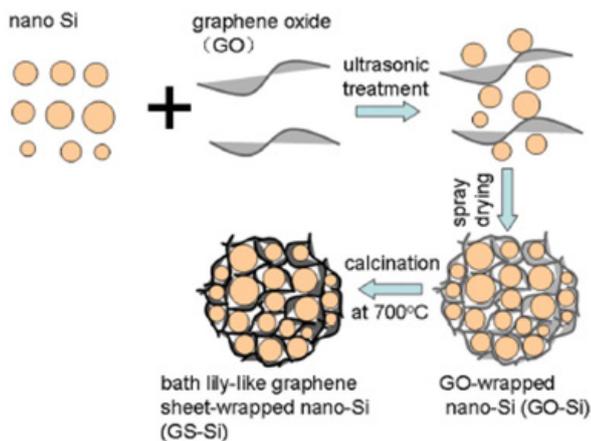


Figure 5 – Example showing the principle of composite forming through spray drying . [4]

The composite forming process can either be used to introduce new properties to a material or to have undesired properties of a material reduced. For example, Lai et al. introduced better electric conductivity to graphite anodes by manufacturing a Silicon/flake graphite/carbon-composite material. [2] The reduction of undesired properties is well presented by de Buysser et al. They had the positive thermal expansion coefficient of ZrO_2 compensated by adding different amounts of ZrW_2O_8 , which has a negative thermal expansion coefficient. This allowed designing materials with tailored thermal expansion coefficient. [16]

5. Spray drying advanced materials

In granulation by spray drying, a suspension of primary particles, binder and surfactant is atomised into small droplets. Then they are injected into a stream of hot gas which causes the fluid to evaporate from the droplet surface. The droplet shrinks continuously to a nearly spherical granule made up of fine particles. [1]

In the best case, the formed granules are uniform solid spheres. Hollow granules are not desired since they lead to defects known as craters or “blow holes” within sintered materials. The morphology of the granules depend on the spray drying parameters, on the solids loading, the rheology of the sprayed slurries and on the degree of flocculation within the slurry. The introduction of binders and plasticisers help to form solid particles, but can also cause problems during spray drying. [1] Interesting references to investigations on the prerequisites to gain well-formed granules and their influence on the sintered ceramic product are given in Chapter 6.1.

With the robust Mini Spray Dryer B-290 and the Nano Spray Dryer B-90, BUCHI offers tailored solutions to produce particle sizes ranging from 300 nm up to 60 µm. A wide selection of nozzles and nozzle tips allow spray drying of almost any liquid and even two immiscible liquids at once. A broad range of glassware allows separating even very small particles from the stream of hot air as well as to process light sensitive substances.



Figure 6 – B-290 advanced in combination with the inert loop B-295



Figure 7 – Nano spray dryer B-90 in combination with the inert loop B-295

The use of the Inert Loop B-295 enables the user to dry solutions or suspensions of organic solvents in a fully closed system. This maximizes user safety and minimizes solvent wastage. In combination with the Dehumidifier B-296 the results can be perfectly reproduced thanks to accurately conditioned drying air.

Due to its ergonomic design, the BUCHI Spray Drying products and solutions are very easy to assemble and to clean. The compact design allows placing them under a standard laminar flow bench, furthermore the remote control and safety curtain, the user is shielded. The automated nozzle cleaning avoids clogging and makes the use of the spray dryer very straight-forward.

The Mini Spray Dryer B-290 as well as the Nano Spray Dryer B-90 allow processing of small amounts of material. This leads to high yields which can reduce costs in research and development thanks to effective formulation optimization. There have been more than 700 publications and 400 patents in which BUCHI spray dryers were used. Customers all over the world appreciate the professional local application support offered by BUCHI and profit considerably from the scalable spray drying process.

6. Research carried out using BUCHI Spray Drying instruments

The following chapters give an insight into some of the research carried out using BUCHI Spray Drying Solutions. The focus is on powder-, transparent materials-, battery- and bioceramics research.

6.1. Ceramic powder research

Many efforts have been done by several researchers to determine factors leading to dense, spherical granules which are viewed as ideal particles. Depending on various production factors, the shape, size and size distribution of granules can differ substantially. Using a B-290/B-295 solution, Choudhary et al. found out that there is a prominent relation between slurry concentration, feed rate and the shape and size distribution of the dried ceramic powders. [7] Ramavath et al. worked out that free flowing alumina powders are achieved with the B-290/B-295 System. Both research groups used spray drying to form granules. They pointed out that good flow-ability of powder is given if the particles are dense, of spherical shape and the size distribution is narrow. This finding is especially essential because the industrial ceramic production process involves die-filling and pressing where a high flowability of the powder is crucial. [6]

In contrast to other evaporation technologies such as rotary evaporators, ovens and freeze dryers; spray dryers produce particles of defined size and shape in a one step process. The possibility of producing powders with predefined specifications such as granule size, morphology, density and moisture content makes spray drying a valuable production method for the powder producing industry. [6]

Table 2 shows a selection of references to research carried out between 2004 and 2015 on ceramic powders.

Spray dried slurry	Reference	Institution
Aqueous dispersion of Alumina powder (30 wt.%) with Darvan 821A and PVA (2 wt.%)	Ramavath et al. (2014) "Effect of primary particle size on spray formation, morphology and internal structure of alumina granules and elucidation of flowability and compaction behaviour"	International Advanced Research Centre for Powder Metallurgy and New Materials, Hyderabad, India
Acidic Alumina-Zirconia suspension	Naglieri et al. (2010) "Follow-up of zirconia crystallization on a surface modified alumina powder"	Politecnico di Torino, Torino, Italy Université de Lyon, Villeurbanne, France
Aqueous ZrO ₂ & WO ₃ Slurry with Polyethylene Glycol	De Buysser et al. (2004) "ZrO ₂ -ZrW ₂ O ₈ Composites with Tailor-Made Thermal Expansion"	Ghent University, Ghent, Belgium
Alumina Nanopowder with Darvan 821A and PVA (2 wt.%)	Choudhary et al. (2013) "Experimental Investigation on Flowability and Compaction Behaviour of Spray Granulated Sub-micron Alumina Granules"	International Advanced Research Centre for Powder Metallurgy and New Materials, Hyderabad, India

Table 2 – Literature on ceramic powder research (2010-2015) All researchers used BUCHI Spray Drying systems.

6.2. Battery research

In 1991 when the commercial Li-Ion battery came to the market, advanced materials found their way into commercial batteries. The anode material of older batteries was graphite, which remained until now a widely used material in negative electrodes. The positive electrode at this time was made of LiCoO_2 . Nowadays, the trend in cathode materials goes to mixed oxides, spinel and olivine-structures. [3]

The production of LiCoO_2 cathodes is related to several environmental, safety, and cost issues. To solve these problems, much research is carried out on new cathode materials. The purpose of spray drying within the publications stated in Table 3 was frequently to produce micro spherical granules. [5]

The traditional graphite used in the first Li-Ion-batteries is insufficient to meet the actual demands on anode materials, since theoretical specific capacity is quite low. In 2004 it was discovered that graphene is an excellent substrate to accommodate active materials. This is due to the fact that graphene has a high electronic conductivity, a high surface area and superior mechanical properties. Since then, many composites containing graphene were investigated as they showed a better cycling performance and rate capability. Within the publications stated below, BUCHI Spray Drying solutions are used to form different composites - often within graphene sheets. [4]

Table 3 shows some references to research carried out between 2010 and 2015.

Spray dried slurry	Reference	Institution
Aqueous dispersion of Nano-Si & graphene sheets	He et al. (2011) "A novel lily-like graphene sheet-wrapped nano-Si composite as a high performance anode material for Li-ion batteries"	Shanghai Jiao Tong University, Shanghai, China Univeristy of Wollongong, Wollongong, Australia
$\text{TiOSO}_4 \cdot \text{H}_2\text{SO}_4 \cdot \text{H}_2\text{O}$ and (NH_4) $\text{NbO}(\text{C}_2\text{O}_4) \cdot \text{H}_2\text{O}$ in Nitric acid solution	Ventosa et al. (2013) "TiO ₂ /Anatase Composites Synthesized by Spray Drying as High Performance Negative Electrode Material in Li-Ion-Batteries"	Ruhr-Universität, Bochum, Germany
Fe_2O_3 & Graphite in aqueous solution	Zhou et al. (2012) "Facile spray drying route for the three-dimensional graphene-encapsulated Fe ₂ O ₃ nanoparticles for lithium ion battery anodes"	Jiao Tong University, Shanghai, China Monash University, Melbourne Australia
Graphene oxide in aqueous solution	Qiu et al. (2014) "Evaporative Assembly of Graphene Oxide for Electric Double-Layer Capacitor Electrode Application"	Stevens Institute of Technology, Hoboken, USA
	Zhang et al.(2014) "Polyimide Encapsulated Lithium-Rich Cathode Material for High Voltage Lithium-Ion Battery"	Jiao Tong University, Shanghai, China
	Liu et al.(2012) "LiFePO ₄ /C Microspheres with Nano-micro Structure, prepared by spray drying method assisted with PVA as Template"	South China University of Technology, Guangzhou, China

Spray dried slurry	Reference	Institution
	Li et al.(2015) "Facile synthesis of spherical $x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{Li}(\text{Mn}_{0.33}\text{Co}_{0.33}\text{Ni}_{0.33})\text{O}_2$ as cathode materials for lithium-ion batteries with improved electrochemical performance"	Xiamen University, Xiamen, China
	Li et al.(2015) "Facile synthesis of spherical $x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{Li}(\text{Mn}_{0.33}\text{Co}_{0.33}\text{Ni}_{0.33})\text{O}_2$ as cathode materials for lithium-ion batteries with improved electrochemical performance"	Xiamen University, Xiamen, China
	Feng et al. (2014) "Nano/micro-structured Si/CNT/C composite from nano-SiO ₂ for high power lithium ion batteries"	Jiao Tong University, Shanghai, China
	Wang et al. (2014) "Hierarchical Sulfur-Based Cathode Materials with Long Cycle Life for Rechargeable Lithium Batteries"	Jiao Tong University, Shanghai, China
	Sakao et al.(2012) "Synthesis and Electrochemical Properties of Porous Titania Prepared by Spray-drying of Titania Nanosheets"	Yokohama National University, Yokohama, Japan
	Yuan et al. (2014) "One-Pot Spray-Dried Graphene Sheets-Encapsulated Nano-Li ₄ Ti ₅ O ₁₂ Microspheres for a Hybrid BatCap System"	Jiao Tong University, Shanghai, China Sinopoly Battery Research Center, Shanghai, China

Table 3 – Literature on battery research (2010-2015) All researchers used BUCHI Spray Drying systems.

6.3. Transparent advanced material research

In the early days, transparent laser host materials were synthetically grown crystals. These crystals, in general, were small, had optical defects and particle inclusions due to the crystal growth process. Hence, they showed limitations in the use as laser host materials, especially since lasers became more powerful. [13]

During the last decade, remarkable progress has been made in the production of transparent ceramics serving as laser host materials. This was due to new powder synthesis and sintering methods. The manufacturing of highly pure powders in a bottom-up approach, as described in chapter 4, gave rise to transparent ceramics with minimal optical defects. [13]

Due to their superior strength, transparency and refractory characteristics, transparent ceramics are not restricted to laser host materials but are also interesting for transparent armor, camera lenses and radiography screens.

Within the field of transparent ceramics, BUCHI products and solutions are often used to spray dry suspensions of mixed raw materials into spherical granules and to bring in sintering agents as well as dispersants homogeneously into the ceramic powder. [10,11]

Table 4 shows some references to recent research carried out between 2010 and 2015 on transparent ceramics.



Spray Drying
Advanced materials

Spray dried slurry	Reference	Institution
Y ₂ O ₃ & Al ₂ O ₃ & Er ₂ O ₃ & Yb ₂ O ₃ and Tetraethylortho-silicate (0.5wt.%) and Polyethylene glycol (1 wt.%)	Cavalli et al. (2013) "Synthesis and optical spectroscopy of transparent YAG ceramics activated with Er ³⁺ "	Università di Parma, Parma, Italy ICT Prague, Prague, Czech Republic Università di Verona, Verona, Italy
Aqueous solution of Lu _{1.9} Eu _{0.1} O ₃ and Polyethylene Glycol & Ammonium Polymethacrylate	Seeley et al. (2011) "Transparent Lu ₂ O ₃ :Eu Ceramics"	Lawrence Livermore National Laboratory, Livermore, USA
Aqueous solution of Y ₂ O ₃ & Al ₂ O ₃ & Pr ₆ O ₁₁ and tetraethyl orthosilicate (0.5%) and Polyethylene Glycol (1wt.%)	Cavalli et al. (2014) "YAG:Pr ³⁺ transparent ceramics for applications in photonics: synthesis and characterization"	Università di Parma, Parma, Italy ISTEC CNR, Faenza, Italy Università di Verona, Verona, Italy ANK Service, Novouralsk, Russia Ural Federal University, Ekaterinburg, Russia University of Canterbury, New Zealand Utrecht University, Utrecht, The Netherlands
	Serantoni et al. (2012) "Improvements in the production of Yb:YAG transparent ceramic materials: Spray drying optimisation"	National Research Council of Italy, Faenza, Italy
	Esposito et al. (2011) "Experimental features affecting the transparency of YAG ceramics"	ISTEC CNR, Faenza, Italy IFAC CNR, Sesto Fiorentino, Italy
	Esposito et al. (2013) "Microwave sintering of Yb:YAG transparent laser ceramics"	ISTEC CNR, Faenza, Italy Institute of Applied Physics RAN, Nizhny Novgorod, Russia
	Ramavath et al.(2014) "Optical and mechanical properties of compaction and slip cast processed transparent polycrystalline spinell ceramics"	International Advanced Research Centre for Powder Metallurgy and New Materials, Hyderabad, India Naval Materials Research Laboratory, Ambarnath, India
	Alderighi et al. (2010) "Characterization of Yb:YAG ceramics as laser media"	IFAC-CNR, Sesto Fiorentino, Italy ISTEC-CNR, Faenza, Italy

Table 4 – Literature on laser research (2010-2015) All researchers used BUCHI Spray Drying systems.

6.4. Bioceramics research

Medicine has made great progress within the past decades. Also because of great progress in materials science. Current research is mostly carried out on bone graft material, bone substitutes, dental implants and dental cements.

Hydroxyapatite ceramics are used as bone graft materials thanks to their biocompatibility and osteoconductivity as well as to their adaptable porosity. Production of bone graft material starts by spray dry hydroxyapatite slurry into fine spheroid powder. Subsequently, the powder is homogenized in water with binders and surfactants. Then, microbubbles of air are introduced to the slurry before it is gelled, dried, shaped and sintered. This results in bone graft material with a defined porosity. [11]

In manufacturing bone substitutes (femoral heads for example) biocompatibility, strength, toughness and wear resistance are significant. Indispensable prerequisites to receive a defect-free ceramic with these properties are an even size- and phase distribution, and the absence of hollow granules. This can be achieved by control over the slurry rheology and spray drying parameters as described by Naglieri et al. [12]

Saadaldin et al. used miserite containing glass ceramic as a substitute for metallic dental implants. Granulation by spray drying led to a more homogeneous glass with a lower melting point compared to conventional preparation methods. [9] Besides dental implants, dental ionomer cements are in the focus of research. The benefit of spray drying is to dry a ball milled glass frit suspension and to agglomerate the particles into spherical granules. [10]

Donadel et al. spray dried a suspension of nano-coated iron oxide particles using BUCHI products. Since iron oxide particles are magnetic but not biocompatible, they must be coated with biocompatible material- in this case with hydroxyapatite. Such particles could be used in the treatment of cancer. [8]

Table 5 shows some references to recent research carried out between 2009 and 2015 within the bioceramic field.



Spray dried slurry	Reference	Institution
Basic suspension of iron oxide & Hydroxyapatite	Donadel et al.(2009) "Preparation and characterization of hydroxyapatite-coated iron oxide particles by spray-drying technique"	Universidade Federal de Santa Catarina, Trindade, Brasil
Si(C ₂ H ₅ O) ₄ & Al(NO ₃) ₃ ·9H ₂ O & Ca(NO ₃) ₂ ·4H ₂ O & CaF ₂ & KNO ₃ & H ₃ BO ₃ and La(NO ₃) ₃ ·6H ₂ O in aqueous solution	Saadaldin et al. (2013) "Synthesis of bioactive and machinable miserite glass-ceramics for dental implant applications"	The University of Western Ontario, Ontario, Canada
	Lopez-Noriega et al.(2014) "Incorporation of polymeric microparticles into collagen-hydroxyapatite scaffolds for the delivery of a pro-osteogenic peptide for bone tissue engineering"	Royal College of Surgeons in Ireland, Dublin, Ireland Trinity College Dublin, Dublin, Ireland RCSI & TCD, Dublin, Ireland
	Watanabe et al.(2014) „Controlled release of a protein using a ceramic carrier and zinc ions as a novel approach to the treatment of osteoporosis“	Kawakita general hospital, Tokyo, Japan Tokyo Institute of Technology, Tokyo, Japan
Aqueous suspension of glas frit powder	Monmaturapoj et al. (2012) „Preparation and characterisation of glass ionomer cements incorporating barium and magnesium ions“	National Metal and Materials Technology Center, Pathumthani, Thailand Kasetsart University, Bangkok, Thailand

Table 5 – Literature on bioceramic research (2010-2015) All researchers used BUCHI Spray Drying systems.

7. Conclusion

BUCHI Spray Drying solutions have shown to be popular, reliable and appreciated systems used in many fields of materials research. The robust products combined with the myriad of accessories and the professional application support allow finding a tailored solution to almost every need of almost every customer. No matter in which field of material research they are working.

The applications arising from the materials field undoubtedly affected, affect and will affect our daily life. BUCHI is proud to be a supporting wheel in the clockwork of innovation within this fascinating field of research and development. BUCHI employees are working ceaselessly to come out with products and solutions further facilitating customer's work and opening up new ways of robust, reliable material processing.

8. References

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