GERMANY

# IWM – IAPK Colloquium 2017: Sintering as a Future Technology – Opportunities and Challenges

**On 16.03.2017, at the Technology Centre on Europaplatz, in Aachen/DE, the IWM–IAPK Colloquium was held under the heading "Sintering as a Future Technology – Opportunities and Challenges". In his welcome address, speaking to the over 100 attendees, the host, Prof. Dr-Ing. Christoph Broeckmann (Fig. 1), proclaimed that sintering is a technology with a long past, but has great future potential, too.**

*Fig. 1*
*Prof. Dr-Ing. Christoph Broeckmann*

The event gave an insight into relevant issues from research and industry concerning sintering of both metallic and ceramic materials. As well as spotlighting technological developments of modern sintering processes, the event focussed on numerical methods for simulation of the sintering process. To follow on from the presentation of the papers, a tour of the Institute for Materials Applications in Mechanical Engineering had been arranged.

## Papers

*"Sintering and Interaction with the Atmosphere – Current Knowledge and Challenges for the Future"* was the heading of the introductory talk presented by Christian Gierl-Mayer (Vienna University of Technology/AT). Conventional sintering models for metallic materials are based essentially on the diffusion of metal atoms, which is driven by the minimization of surface energies of dispersed systems. These models neglect the chemical boundary surfaces generated as a result of the thermodynamic conditions on the surfaces of the starting powders.

Practically all relevant metal powders are therefore covered by layer of oxide, which must be removed to enable successful formation of a stable contact so that the mechanisms described in the models can proceed. The classical alloying systems in iron moulding (Ni, Mo, Cu) can be easily reduced on account of their low oxygen affinity and in corresponding powder mixes with iron. Iron is the element that is most difficult to reduce. In moulding, for a long time this meant it was relatively easy, with associated low expense, to guarantee this reduction and accordingly to sinter these systems successfully in a stable process.

These classical alloying systems are subject to high surges in price and in fine powder form they are a health concern. Therefore, for some time, attempts have been made to replace these with classical alloying elements from hot-melt metallurgy (Cr, Mn, Si, V). This means, however, huge challenges for sintering technology. The conditions necessary for this are expressed in a high quality standard for the sintering atmospheres. Much more precise knowledge of the processes is necessary so that such systems can be sintered successfully. For example, typical endogas furnaces can no longer be used for many of these alloying types. High-temperature sintering is the only possibility to remove the oxides practically completely from these systems and therefore utilize the full potential of these alloys in respect of their mechanical properties.

These temperatures, however, lie much higher than those usual today which can be achieved in an economically viable process using belt furnaces. What remain and, in some cases, are intensified are the problems of the debinding process and decarburization based on diverse mechanisms. Methane formation and consumption of the added carbon for reduction of the oxides leads to uniform decarburization, humidity in the atmosphere to undesirable decarburization processes at the surface.

Markus Schneider (GKN Sinter Metals/DE) spoke on *"Material – Process – Product: Trends in PM Manufacturing".* A persisting trend is the desired shortening of the product development time. A consequence of this is that different materials and different geometry proposals are analysed/validated virtually, before prototypes or first component tests are realized.

A key role here is played by the availability of appropriately prepared data sets, which can be applied uniformly and easily by all designers or calculation engineers (with, for instance, "drag and drop"). Another aspect of the hoped-for shortening of the product development time is the use of additive-manufactured components. The applications for this are wide ranging. Samples for installation planning of complex component assemblies, to teach coordinate measurement machines, for demonstration to customers (marketing) or for functional tests in which the surface and the mechanical properties play a (still) subordinate role. Basically, however, a potentially new market is to be opened up with additive-manufactured components. As, presumably, it will always be geometrically complex-shaped (in some cases, topology-optimised) components, profound material understanding is essential.

With the removal of seemingly superfluous material, the effective certainty is reduced dramatically. Accordingly, the assumptions made regarding the material must be certain. Precise knowledge of the internal stresses and process sensitivities is essential. It should not be forgotten that additive-manufactured components consist of many layers of powder, for this reason a reproducibly high powder bed quality is necessary.

To guarantee this high powder bed quality, comprehensive characterization of the powders (including, amongst other things, the powder flowability) is necessary. If electrification progresses in automotive engineering and in industry, soft-magnetic components (SMC) will gain great importance. These components are no longer exposed purely to mechanical stresses, but increasingly to thermal, electrical, magnetic and corrosive stresses. Therefore, it is necessary to establish methods that can characterize the behaviour of a material when it is exposed to these complex stresses.

*Fig. 2*
*In the auditorium: Dr Anke Kaletsch (IAPK-RWTH), and Dr Alexander Bezold (IWM-RWTH)*

Sebastian Riehm Deng (IAPK – Institute of Ceramics Process and Application Engineering/DE) presented work on *"Numerical Simulation of Sintering in the HIP Process".* Hot isostatic pressing (HIP) is known as a manufacturing process with which materials can be compacted to full density without internal porosity.

For this purpose, the components are exposed in a HIP system to temperatures up to 2000 °C and pressures up to 200 MPa simultaneously for several hours of holding time. Pre-compacted components that exhibit closed porosity from the outside can be compacted without further pre-processing in the HIP system. Metal powders, on the other hand, must be filled into a gas-tight metal capsule and sealed under vacuum (powder HIP). To save resources, especially for high-performance materials, a near-net shape is desired: the components or capsules are dimensioned prior to HIP so that finishing after HIP can be reduced or even eliminated completely.

Deformation and shrinkage caused by the HIP process must be taken into account. To predict the compaction and the shrinkage caused by HIP, at IWM/IAPK a numerical simulation model has been developed. With this model, it is possible, based on knowledge of temperature- and density-dependent material parameters, to calculate the compaction during the process and the change in shape. As a result, on the one hand, suitable parameters for a HIP cycle can be designed. On the other hand, the component shape can be optimised before HIP so that after HIP the component has the desired shape.

Markus Zwick (FGK/DE) reported on the *"Challenge of Sintering Transparent Ceramics".* With the development of the sodium vapour lamp, the development of transparent ceramic also began. In the 1960s, translucent alumina was discovered for the first time and advanced with specific further development to transparency. The special feature is that alumina does not exhibit any optically isotropic crystal structure and therefore transparency can only be achieved when the microstructural components (grains and grain boundaries) are below the wavelength of visible light (<300 nm), so scattering effects recede into the background. In optically isotropic (cubic) crystal systems, there is no necessity for fine microstructural components.

For both variants, however, foreign phases in the form of pores, impurities, secondary phases and other defect structures must be avoided. This is the key challenge in the fabrication and sintering of transparent ceramics. The objective of preparation, shaping and sintering is therefore the production of defect-free microstructures with maximum homogeneity and, in the case of optically anisotropic crystal structures, a particle size <300 nm. Important for successful sintering is the availability of green bodies with homogeneous and high packing density. Precondition for this is that during preparation the agglomerates that inevitably exist in the highly sinter-active powders are broken up and a shaping technology is used that produces green bodies without any density gradients.

In this context, it should be noted that in the individual steps of preparation contamination, for example by attrition particles during grinding, is prevented. With the homogeneously dense packing of the particles, a uniform sintering curve and therefore a homogeneous structure are ensured. Pores with cluster structures, which can be formed if preparation and shaping are not sufficiently effective, are not usually eliminated in traditional sintering or may even lead to the formation of intracrystalline pores, which can only be removed in pressure-assisted sintering processes. In many cases, pore-free densification can only be realized in a two-stage sintering process. In the first sintering process, the body to be densified is usually sintered to the last stage of sintering and therefore to complete elimination of pores and then redensified in a pressure-assisted sintering process, hot isostatic pressing.

Besides elimination of pores, with this two-stage process a minimum particle size is achieved, which is essential in the case of optically anisotropic crystal structures. In this two-stage process, grain growth is selectively suppressed and pores eliminated with the assistance of pressure. Another important focus is the decomposition of organic additives. Irrespective of the shaping method, organic additives are used for selective adjustment of the processing properties in the different processes.

These must be removed from the component without leaving any residue before the start of sintering and the associated densification process. To this end, the respective debinding steps must be selectively adjusted as even the tiniest residues of carbon compounds can lead to a substantial deterioration of the optical properties.

It should therefore be noted that for successful sintering of ceramic components, with the objective of optical transparency, besides selection of the suitable sintering process, preparation, shaping and debinding are the key to success and if sufficient attention is not paid to the above-mentioned process steps, no transparency can be obtained.

Stanley van Kempen (IAPK/DE) reported on the *"Simulation of Co-Sintering of Ceramic Multilayer Composites".* In the project "Design Development and Modelling for Innovative Firing Chamber Lining Concepts" funded by Germany's Federal Ministry for Economic Affairs and Energy, within the framework of the cooperative project "Development of Combustion Techniques in the Clean Energy Center for Climate-Friendly Energy Generation", the IAPK is working together with the project partners Siemens AG and Friedrich-Alexander-University of Erlangen-Nuremberg to develop a design methodology for the fabrication and application of optimum multilayer composites. The variation of the layer materials and their sequence in a laminate enables the production of different functional profiles for versatile application. The laminates are fabricated in the co-sintering process. Here, green ceramic tapes or paper ceramics are laminated and sintered.

Different sintering shrinkage and thermal expansion between the layers lead to internal stresses, deformation and potentially premature failure. For simulation of the fabrication process, a phenomenological sintering model was further developed and implemented in the FEM tool Abaqus. With the help of this model, internal stresses and deformation during the co-sintering process can be predicted. This enables, amongst other things, conclusions in respect of layer compatibility and identification of suitable layer sequences and thicknesses. More than 70 different ceramic tapes and preceramic papers fabricated by project partners are available to fabricate the laminates. The developed model enables the prediction of the free sintering shrinkage and densification of a monolithic material with an error <3 % and of the sintering and deformation behaviour of co-sintered multilayer composites with an error <12 %. The model permits a qualitatively good prediction of any internal stresses. The model was integrated in an optimisation process that enables application-optimised design of multilayer composites.

*"FE Simulation of Field-Assisted Sintering"* was addressed by Sree K. Sistla (IWM/DE). Field-Assisted Sintering Technology (FAST) is based on a modified hot moulding process in which electric current flows through the graphite mould. The material is heated up directly by the current, not conventionally with an external heater. FAST is a pressure-assisted short-time sintering process with low voltage and high current density. Numerical investigations based on finite element simulations of FAST are very complex: electrical, thermal and structural effects have to be combined in one step. For higher prediction quality, the material behaviour (density, thermal conductivity, electric conductivity, specific heat, etc.) as a function of the temperature must be taken into consideration. In the presented investigation, graphite was used as a material for the mould and tungsten carbide (binderless hard metal) as a powder material.

Thermophysical analysis was conducted to determine the above-mentioned material properties of the graphite. The numerical simulation of the FAST with a multiphysics simulation based on a "coupled thermal-electrical-structural" approach was shown. The study ended with the comparison of the simulation results of temperature distribution with pyrometer measures and the relative density, which was determined after Archimedes.

*"Electron Beam Melting – from Powder to Component"* was discussed by Thomas Weissgärber (Fraunhofer Institute for Manufacturing and Advanced Materials (IFAM)/DE). Electron beam melting is a powder bed process with high efficiency. High energy densities and very fast scanning enable high build rates. In contrast to laser beam melting (fine powders 10–45 µm), it is possible work with relatively coarse powders 45–150 µm, which also necessitates certain compromises in respect of surface quality.

Current development directions at the IFAM are: extension of the material spectrum (Ni basic superalloys, steel, refractory metals, carbides), processing of non-standard powders, enlarging the useful build space (currently 20 cm × 20 cm × 38 cm, and diameter 35 cm at 38 cm build height), improvement of the surface qualities and the setting up of a user centre.

*"Multiphysics Simulation – A Promising Tool for the Analysis of the Laser Beam Melting Process"* was presented by Karl-Heinz Leitz (Plansee SE/AT). Laser beam melting of molybdenum was described as an example to show that the process enables unique design possibilities at short process times for small unit numbers with minimal material input. The process is highly dependent on the material. Powder properties and powder application have a crucial influence on the process and machining result. Under development are multilayer components and internal stress and deformation analyses on process and component level.

*"Field-Assisted Sintering Technology/Spark Plasma Sintering: Current Situation and New Developments"* was the subject addressed by Olivier Guillon (Jülich Research Centre/DE). Around 1800 FAST/SPS systems are now installed, roughly 2/3 of which in industry. The trend is currently towards larger components and more complex geometries, which is why near-net shaping is coming into focus.

Accordingly, work is being done on new mould concepts, which also have to be redesigned in respect of the materials. Various variants of the sintering techniques are available: Spark Plasma Sintering (SPS), Field-Assisted Sintering (FAST), Electric Current Activated Sintering (ECAS), Pulsed Electric Current Sintering (PECS), Field Activated Pressure Assisted Sintering (FAPAS), Plasma Activated Sintering (PAS) and Current-Activated, Pressure-Assisted Densification (CAPAD). Attempts are underway to develop hybrid technologies from these.

Stronger electric fields could favour the solid-state sintering of oxide-based materials. "Cold" sintering describes an effect in which densification is promoted by crystal water bonded in the material. It is important that in the analysis of the sintering mechanisms, "clean" test conditions are ensured to avoid misinterpretation.

*"Defect – Activated Sintering: Is it Really Necessary to Explain High Shrinkage Rates?"* asked Torsten Staab (University of Würzburg/DE). Defects on metallurgical powders (pure Cu, Ni, Wo) were analysed with different microscopic methods. It could be shown with all the methods that no displacements are detected before shrinkage starts. These only occur when the relaxation temperature (40 % of the melting temperature in Kelvin) is reached. It was proven that a much finer powder fraction (around 10× smaller than assumed) is responsible as the driving force for densification and explains the observed high shrinkage rates.

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