Publishable executive summary

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Abstract

The main goal of the project was the integration of the two component ceramic injection moulding (2C-CIM) as a low cost and large series production technique for development of complex shaped ceramic components for automotive and railway applications offering a high degree of structural and functional integrity. The 2C-CIM technology which is known from two-coloured plastic parts should be qualified for the production of advanced ceramic components combining two ceramic materials with different or opposite properties without additional joining steps. The 2C-CIM technology should be developed as a high pressure and a low pressure variant for ensuring highest flexibility of production. Moreover, appropriate characterizing methods were to be developed which should enable the producer to detect defects in co-injected components even in the green state which is a key factor for a high efficient production technique. New methods of characterization could help to reduce scrap-rate remarkably and to prevent loops in the production cycle. The project tasks had to be solved by implementing simulation tools covering the complete production process, tailoring of ceramic feedstock properties considering simultaneous shrinking behaviour during co-firing and implementing of multifunctional ceramic parts into complete systems ready-to-use for railway or automotive applications.

Objectives

2C-CIM will allow producing advanced ceramic products at a large scale with increased functionality, high degree of complexity, but at a lower cost level in comparison to other shaping techniques. The reason is that ceramic materials offer the possibility to combine properties like electrical conductivity with electrical isolation, high toughness with extreme hardness and wear resistance, etc. Moreover, all these property combinations can be achieved in only one shaping step without additional joining processes by 2C-CIM. This project should launch 2C-CIM as a high-throughput production process for complex shaped ceramic components in Europe. Besides for automotive and railway applications this new technology is of enhanced interest for all branches requiring ceramic materials or property combinations as mentioned above, because novel products could be produces by using 2C-CIM which

cannot be achieved today for technical or economical reasons. In this way 2C-CIM will reinforce the competitiveness of the European PIM industry and of many industrial branches which will be able to provide new or improved products.
The project resulted in four 2C-CIM prototype parts. Functional testing and verification such as techno-economical assessment of the complete processing chain had been carried out for the four parallel case studies: (1) ceramic glow plug, (2) ceramic gear wheel; (3) ceramic valve seat, and (4) ceramic braking pads for high speed trains. For developing the 2C-CIM technology for both, low pressure and high pressure injection moulding, one case study resulted in a prototype produced by low pressure injection moulding (glow plug) and three case studies are attributed to high pressure injection moulding (gear wheel, valve seat, and brake disc).

For achieving the main project goal following tasks had to be fulfilled:

- specifying four functional components as case studies including material choice and freezing of the final design
- adaptation of powder surface properties to the requirements of feedstock production;
- development and supply of new feedstocks suitable for low/high pressure 2C-CIM and environmental-friendly debinding processes;
- linking of tools for simulation techniques of 2C-CIM parts enclosing the complete processing chain, i.e. tool design, injection moulding, debinding and co-sintering processes;
- design and construction of a 2C-CIM tool for testing parts
- establishing of a test rig for non-destructive testing (NDT) by thermography for injection moulded ceramic green parts;
- to develop debinding concepts for co-debinding of the feedstock couples
- to predict final distortion of testing and prototype parts after sintering by FEM analysis
- to develop testing samples by high and low pressure 2C-CIM and to characterise these parts
- to produce powders and feedstock couples with refined properties according to the results achieved the testing parts
- to design prototype parts and to simulate the mould filling process for these parts
- to desing and to construct tools for the four prototype components
- to develop prototype parts by high and low pressure 2C-CIM
- to fix the final composition of the feedstock systems for large-scale production of the prototype parts
- to investigate the prototype parts in the green and in the sintered state by non-destructive testing methods
- to establish technical guidelines for high-volume production of the case study components
- to test the prototypes under practically relevant conditions
- to estimate the production costs for the specified components
- to validate and to verify the results of the simulations results of the different processing steps
- to perform a comparison between traditionally used materials and the ceramic materials chosen for the ceramic components with respect to the main environmental impact and
- to organize two technical workshops.

Work performed

Different types of prototypes for four case studies and appropriate material combinations have been defined by the project partners. Technical specifications of the prototypes were provided. The definition of the prototypes and the material choice was accompanied by a patent survey and a market survey in the field of the specified components and materials.
Following combinations of ceramics have been selected:

- glow plug ➔ property combination electrical conductive/isolating: Si_3N_4/$MoSi_2$ with higher and lower content of $MoSi_2$ which serves as the conductive phase in the material
- gear wheel ➔ property combination high hardness/high toughness: Zirconia Toughened Alumina (ZTA)/Alumina Toughened Zirconia (ATZ)
- valve seat ➔ property combination high toughness/high wear resistance: Si_3N_4/SiAlON
- braking pads ➔ property combination high friction and mechanical strength/high strength with sufficient heat conductivity: Zirconia Toughened Alumina/Al_2O_3

A testing sample geometry has been agreed by the consortium which is quite similar to the geometry of the case study "Braking pad".
The initial powders specified in the technical specifications were made suitable for the production of the components chosen. Special attention have been paid for the implementation of nanosized ceramic powders in the feedstock development, especially for reducing the well-known difficulties connected with the high surface-to-volume ratio of nano powders. Surface treatment of existing powders for achieving suitable surface properties of feedstock preparation was carried out.
Simply shaped compounds of chosen initial powders and powder blend were prepared by conventional dry pressing for investigating the material and compound properties. By these investigations it had been estimated, how the bulk density and the particle size distributions of the powders or powder blends influence the shrinkage of the components during sintering such as the microstructure and properties of the sintered materials. The dry pressed components have been used for testing several mechanical, thermal, and electrical properties of the sintered single materials.
Material compound compacts of the desired material combination were fabricated using the same dry pressing technique considering an adjusted shrinking behaviour of both components. These two-component powder compacts have been used for co-sintering experiments. The development of co-sintering cycles were carried out by using dilatometry data.
For case study 1 (glow plug) one couple of low pressure injection moulding feedstocks have been developed (Figure 1 a). Two couples of feedstock containing ATZ and ZTA powders have been developed for case study 2 (gear wheel) basing on different initial powders, some of them with nanosized grains. The characteristic data of the feedstocks and their injection behaviour have been investigated. For the case study 4 (braking pads) it was necessary to develop one couple of feedstocks consisted of Al_2O_3 and ZTA.
Two-component testing parts had been developed by means of the round robin testing tool which has been constructed. Further testing tools had been developed for the low pressure injection moulding component (glow plug) and successfully tested.

To exchange data for the simulation between project partners it was necessary to develop an interface for linking the different simulation tools of the project partners. This allowed designing mould shapes and taking into account possible distortion of the multi-material parts after sintering.

The test rig for non-destructive testing of injection moulded green parts by thermography had been established. The test rig had been used for measurements including diffusivity, inclusions, cracks, inhomogeneities, and for characterizing the quality of the interface between two components.

The feedstocks adapted in shrinkage for subsequent co-debinding and co-sintering have been characterised and the thermal, rheological and mechanical values were used as input data for mould flow simulation and FEM analysis. Basing on these date the mould filling process such as the stress distribution in the injection moulded components in the green state and after sintering have been calculated.

Different debinding techniques, e.g. thermal debinding, extraction debinding with super critical CO_2, and combined aqueous extraction and thermal debinding, have been studied for developing a co-debinding process for the two-component injection moulded testing parts and prototypes. These investigations were supported by thermal analysis and kinetic models.

Testing tools were used for producing two-component testing parts by high and by low pressure injection moulding. The developed debinding and sintering routes were applied to these testing parts, and they were closely examined in the green, in the debindered and in the sintered state. Non-destructive testing methods like X-ray computed tomography and thermography have been proved to be very helpful tools for improving process parameters and at least the quality of the injection moulded parts. The material properties have been investigated at single component parts. Furthermore, the interfaces between the materials combined by two-component injection moulding were examined by microscopic investigations and non-destructive testing. Basing on the results and the experience gathered with the testing parts the properties of the initial powders and the feedstocks were adjusted and refined. The final shrinkage of the materials during sintering was measured and the design for the prototype parts was frozen. Final feedstock systems which should be used for the manufacturing of the specified case study components had been developed and prepared in sufficient quantities. In the case of the low pressure injection moulding component, the glow plug, three generations of prototype tools had been designed and constructed considering improved contacting conditions for the glow plug. All four case study components, glow plug, gear wheel, valve seat, and braking pad, have been produced with the prototype tools by two-component injection moulding. Due to problems with the rheological properties of the feedstock couple developed for the valve seat component the specified material combination silicon nitride / SiAlON had been substituted by the feedstock combination developed for the gear wheel. Thus the principle functionality of the tool and the component could be demonstrated. All components were characterised in the green, in the debindered as well as in the sintered state by non-destructive testing methods, i. e. X-ray computed tomography, thermography, and 3D coordinate measurement technique. Except the valve seat where the specified material combination had been exchanged all case study components were tested under practically relevant conditions. For all components technical guidelines and cost estimations for high-volume production have been established. The simulation results achieved for mould filling, debindering and sintering have been verified by non-destructive testing methods.

Results achieved in the CarCIM project had been disseminated to an international audience in three technical workshops organized by project participants:

1st workshop on non-destructive testing – technologies & application, April 9, 2008 in Vienna;
2nd workshop on simulation, Nov. 21, 2008, Besancon;
3rd workshop on 2-component powder injection moulding, June 24, 2009, Dresden

The 2nd workshop on simulation in powder injection moulding had been organized as a common event of the CarCIM consortium and the consortium of the CRAFT project MATLAW (Project no.: 033006). Both consortia dealt with simulation either in ceramic or in metal injection moulding. Moreover, both project dissemination plans are addressed to the same community. The venue chosen for this workshop (Besancon) is situated near the border triangle between France, Switzerland and Germany.

Results of the project

For all four case studies suitable material combinations have been chosen and the shrinking behaviour during the co-debinding and co-sintering process have been successfully adjusted. In case study "gear wheel" the shrinkage could be positively affected by using a certain amount of nano structured powders. These results have been applied for a patent. Novel cognitions have been gained in the field of extraction debinding with super critical CO_2, and debinding routes are now available for the removal of the thermoplastic binders from the two-component injection moulded parts. In this connection the time necessary for thermal debinding of low pressure injection moulded parts could be shortened to one third of the original time by using a kinetic analysis of the debinding process.
All feedstocks developed in the first project period have been characterised in detail by thermoanalytical, rheological and mechanical measurements for providing the input data for mould flow simulation. The injectability of the feedstock systems has been tested by producing single component parts. These parts were used afterwards for measuring the mechanical and electrical properties of the single materials.
Even though the total shrinkage of both components in case study "Gear wheel" had been adjusted, the investigations of the testing parts showed a different sintering kinetic of the materials ZTA and ATZ which shall be combined in this part. This mismatch caused cracks and delaminations during sintering. In spite of variations in the heating rate during sintering this problem could not be solved. Alternatively the materials choice for the gear wheel has been altered. The novel material combination for this case study is alumina and zirconia toughened alumina. Both materials show a quite similar sintering kinetic.
Since defects in injection moulded components often become obvious only at the end of the processing chain after time and cost consuming debinding and sintering steps non-destructive testing methods are getting more and more important. In the case of two-component injection moulded parts thermographic and computer tomographic investigations in the green state have been proven to be indispensable methods for characterising the accuracy of the interface between the different materials.
After designing and constructing the tools for three case studies, first prototype parts had been developed and tested (Figures 1-4).

Fig. 1 Glow plugs in the green state

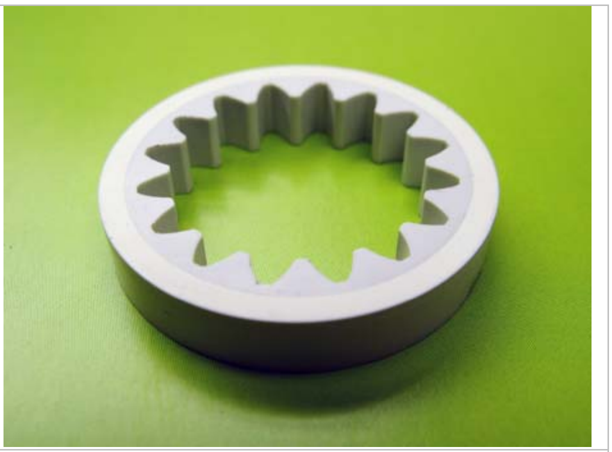


Fig. 2 Gear wheel made of alumina and ZTA in the green state

Fig. 3 Cross cut through a braking pad in the sintered state

Fig. 4 First component and completed valve seat in the green state

As verified by non-destructive testing methods the green parts were defect-free. However, the injection moulding process of the valve seat component required an enormous injection pressure due to the insufficient flowability of both feedstocks. As a consequence the performance of these parts was expectably bad, and cracks appeared during the debinding step. For showing the feasibility of this two-component ceramic part, the decision had been taken to substitute the feedstock combination by those of another case study component. By using the feedstock combination of the gear wheel perfect components of the valve seat could be produced.

The results of mould flow simulation, debindering and sintering have been verified by non-destructive methods too. For that purpose a special marker feedstock has been developed containing two different powders with different X-ray attenuation coefficients. In the reconstruction images of the X-ray computer tomographic investigations flow lines, weld lines and so-called dead water zones could be detected and compared to the mould flow simulation data. Fig. 5 and 6 show the comparison between real and simulated mould filling studies of the second component of the glow plug and the valve seat.

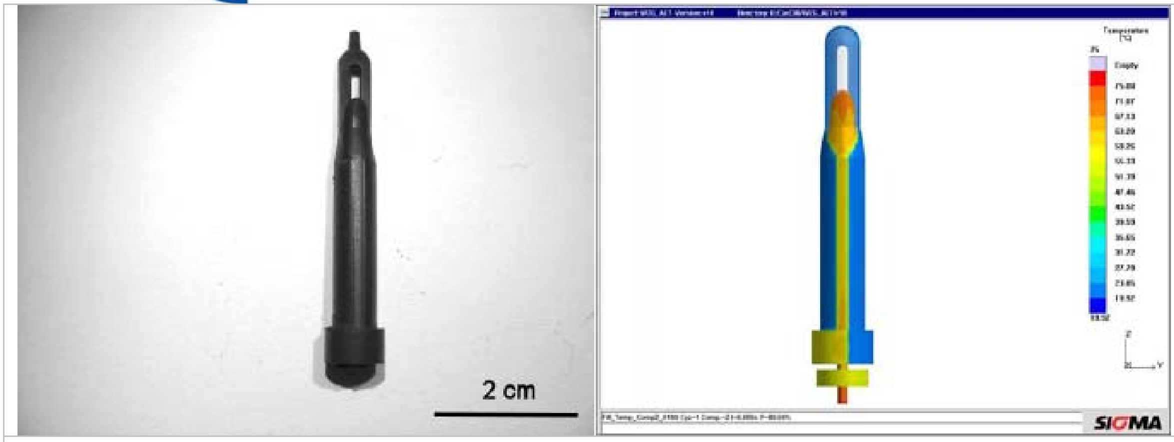


Fig. 5 Comparison between real part and simulation of a mould filling study for the electrical insulating component of the glow plug

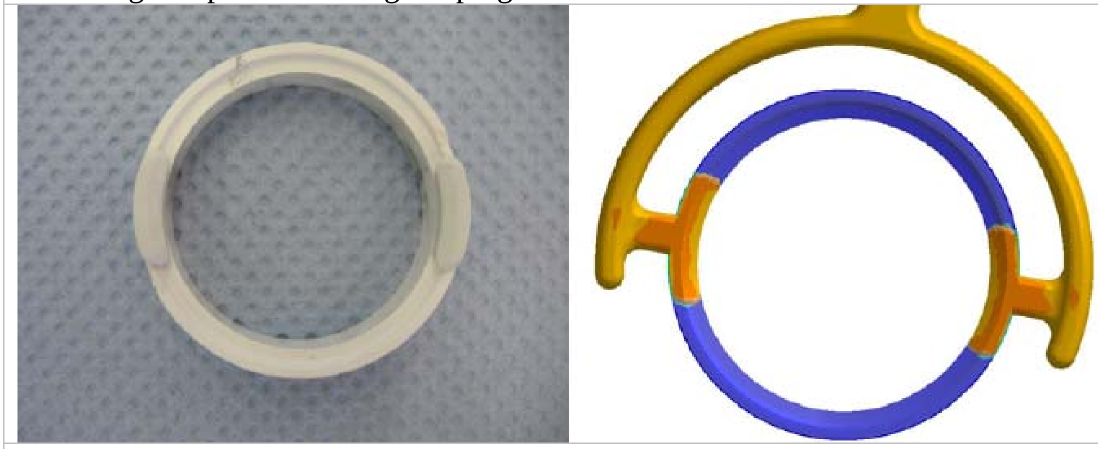


Fig. 6 Comparison between real part and simulation of a mould filling study for the second component of the valve seat

The functionality of the glow plug has been tested by ignition tests. With an applied voltage of 13 V a temperature at the tip of 1250 °C could be achieved within 3 sec. Testing of the components under practical conditions will be continued beyond the project (Fig. 7).

The torque measuring test of the sintered gear wheel showed that the components resist a torque of > 1 Nm (Fig.8) which had been demanded in the specification. Furthermore, the components of the gear wheel have been tested concerning wear resistance, fracture toughness and hardness. A slight deviation in roundness was detected by 3D coordinate measurements. The reason can be seen in the position of the two gates in the injection moulding tool and should be corrected in a series production tool.

The valve seat tool works very well (Fig. 5). Feasibility studies with another feedstock system confirmed that this component can be produced by means of two-component ceramic injection moulding. However, for the desired material combination a new feedstock concept basing on another binder system would have to be developed.

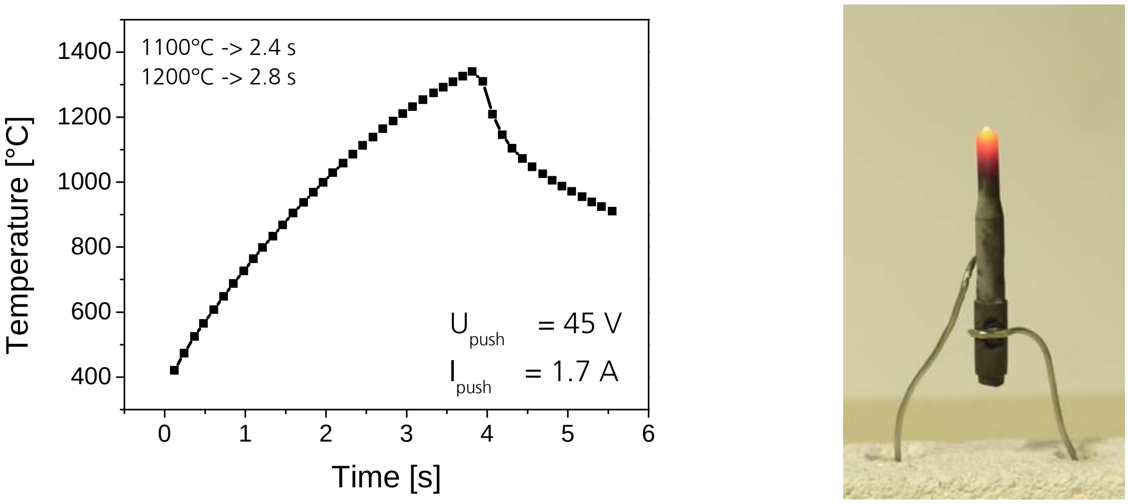


Figure 7 Ignition diagram and glowing glow plug during functional testing

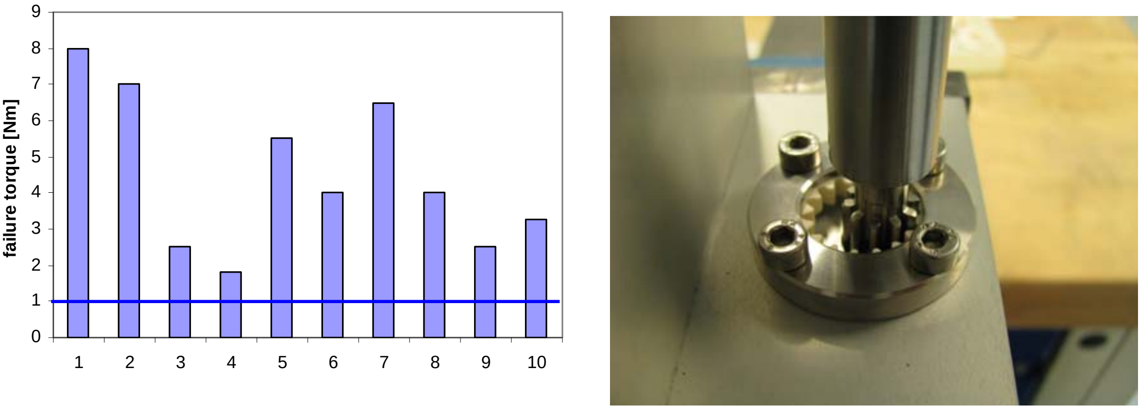
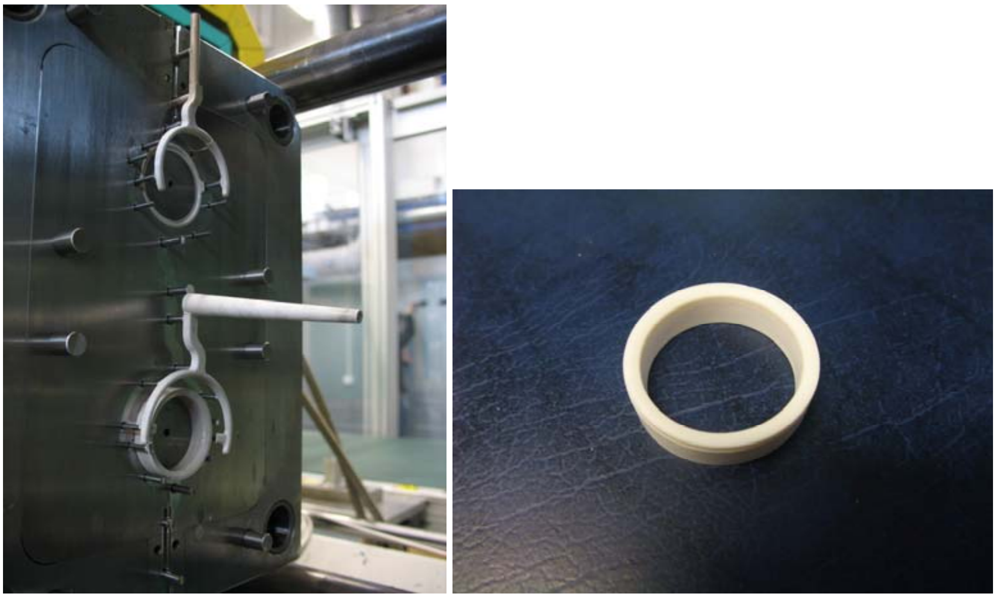


Figure 8 Results of torque measuring tests with ten gear wheels and measuring device

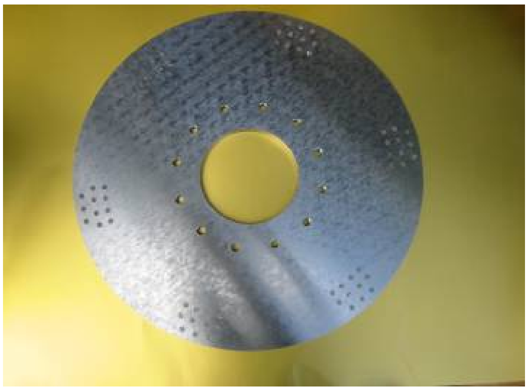
Figure 9

2C-CIM mould for the case study valve seat

Injection moulded sintered component (right)



The braking pads had been inserted into a steel braking disk in a casting process (Fig. 10). A bonding of the components in the steel disk could be obtained, and a braking test emphasized that the braking time from 180 km/h down to hold-up could be reduced by 17 %.



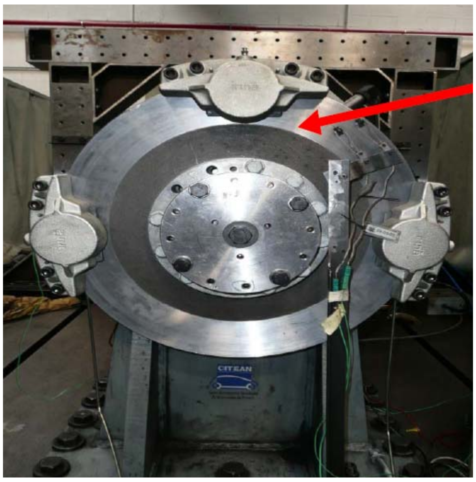


Figure 10 braking pads embedded in the steel braking disc and test bed for brakes

For all components technical processing guidelines and cost estimations are established.

Figure 11

All four CarCIM case study components in the sintered state

Conclusion

Two-component ceramic injection moulding is a very promising shaping technique for large-scale production of ceramic parts with novel functionalities and complex geometries. The advantage of this technology can be seen in the fact that two different ceramics can be combined without any additional time-consuming and expensive assembling or joining steps. However, two-component ceramic injection moulding is a very challenging shaping method. For each processing step of the technological chain both ceramic materials have to be adjusted to each other. Following principle requirements should be taken into account for the development of two-component ceramic parts:

1. The powders chosen for two-component injection moulding must be sinterable to full density at comparable temperatures and under the same gaseous atmosphere.
2. For avoiding critical stresses during sintering the powders shall have a similar sintering behaviour, i.e. the onset of shrinkage shall fall in a narrow temperature range for both powders and the shrinking rate shall be comparable.
3. For ensuring a precise adjustment in total shrinkage the volume content of solid in the feedstocks must be the same.
4. Since differences in the thermal expansion coefficients of the feedstocks may cause cracks, distortion or delaminations of the compounds already in the green state the same binder system or binder systems with comparable thermal expansion behaviour must be used for feedstock preparation.
5. The thermal expansion coefficients of the sintered ceramic materials play also a very important role as shown in this article, because differences in this property can result in stresses in the two-component part during cooling after sintering or during application of the part under cyclic heating and cooling conditions.

If stresses between both components cannot be excluded totally, they should be taken into consideration already in the design of the injection moulded parts.

The CarCIM project showed the feasibility of ceramic components with novel functionalities by two-component injection moulding. It emphasized the importance of non-destructive testing methods for ensuring the quality of the products in each processing step. Simulation of the processing chain can also be a helpful tool for avoiding problems in mould filling or with distortion and delamination of components. However, for improving the reliability of the simulation results further development work will be necessary. Tooling is a very challenging task for two-component ceramic injection moulding. For future works in this field more re-working cycles of the tool should be taken into consideration. The increase in difficulties for tooling in the case of two-component ceramic parts had been underestimated in this project.

In sum the CarCIM project can be evaluated as a very successful European project which has strengthened the cooperation between the former project partners sustainably. A lot of tasks have been solved and a lot future challenges in this field have been pointed up.