3D Printed Al_2O_3 Components for Continuous Processes in the Chemical Industry

To extend the possible working conditions of milli- and microprocess technology, LAPP Insulators Alumina GmbH and TU Dortmund University (Biochemical and Chemical Engineering, Equipment Design) develop new concepts for milli- and microprocess technology plants with integrated key-components made of ceramics. In this case the development of a small scale extraction column [1] with ceramic components made of Al_2O_3 that are produced by 3D printing [2] shall be presented. LAPP Insulators Alumina key products are vacuum-tight ceramic-metal assemblies for a number of different vacuum applications.

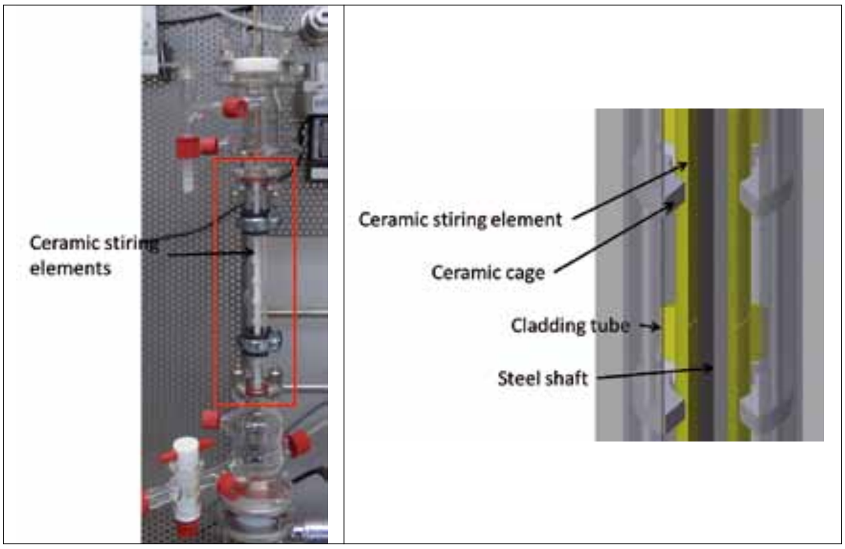

Fig. 1
Experimental setup and schematic illustration of the extraction column

Introduction

Continuous processes have significant advantages in the production of fine chemical and pharmaceutical substances in comparison to conventional batch processes [4]. Traditionally continuous processes are implemented especially in fields of applications in which huge amounts are produced. In recent time however continuous processes are more and more applied in the production of special chemicals and pharmaceutics in which the amounts of produced goods are rather small. Advantages of these processes are a good controllability of the reaction, small amounts of substances that are actually in the process, more easy and stable operating conditions and partly higher selectivities. Because of the high surface to volume ratios that are characteristic for milli- and microprocess technologies specific processes can be intensified. This leads to overall lower production costs by higher yield of the final product.

Currently components made of chemical resistant steels and glasses are used for these processes. For most fields of application the properties of these materials are sufficient concerning the thermal- and chemical resistance. To extend the possible working conditions of milli- and micropro-

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Ceramic components for a small scale extraction column

Extraction columns are used to remove a valuable component from a feed-flow [5]. This is realized by passing the feed-flow with the valuable components and an extraction flow (solvent) through the extraction column countercurrent. While passing through the column the valuable component will be transferred from the feed to the extraction-solvent. The extraction medium containing the valuable component will be collected at the end of the extraction column. The valuable component can be further and more easily separated in a second step. Extraction columns are used mainly in processes in which thermal sensitive substances have to be separated since the driving force of the extraction column is not thermal but physical/chemical.

When especially pure substances have to be produced (e.g. pharmaceuticals and fine chemicals) conventional used materials are not sufficient anymore since often very small quantities of these component materials can be transferred to the product by abrasion or chemical reaction. This is where the use of ceramics with its very high chemical- and wear resistance can be beneficial for the process. In Fig. 1 (left) the experimental setup of a single stage extraction column with ceramic components is shown. On the right side the schematic structure is illustrated.

The column in the first expansion stage consists of a steel shaft with attached ceramic stirring elements. These elements are rotating in a ceramic cage with intermediate plates. Everything is enclosed by a glass-tube (in the laboratory setup, later a ceramic tube can be used). By rotation of the ceramic stirring elements additional energy is induced into the system to improve the selectivity of the column. The intermediate plates are inserted to prevent recirculation of the fluid. This also improves the overall effectiveness of the unit. The volume of one extraction stage is approximately 35 ml [1]. In a production process several stages of the small scale extraction column can be connected in series.

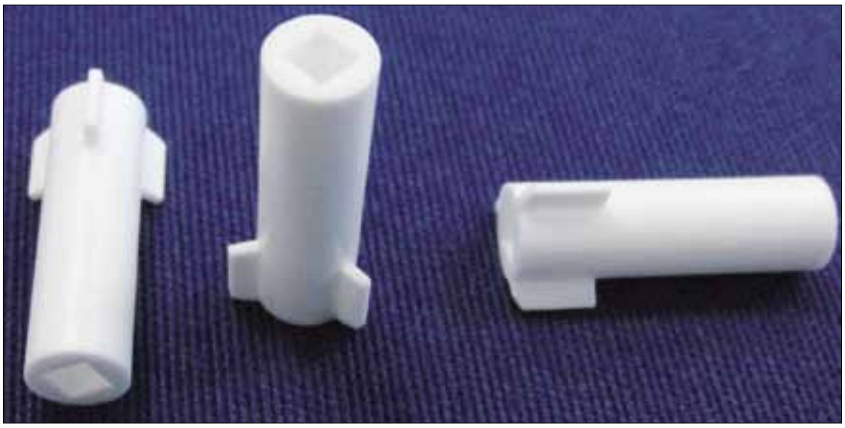

Fig. 2
Ceramic stirring elements (form-fit, Al_2O_3)

Fig. 3
Ceramic stirring elements with screw threats (Al_2O_3)

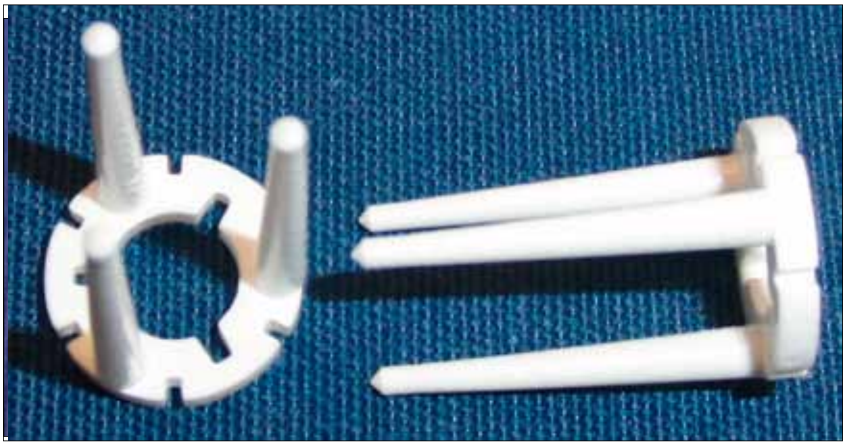

Fig. 4
Ceramic cage with intermediate plates (Al_2O_3)

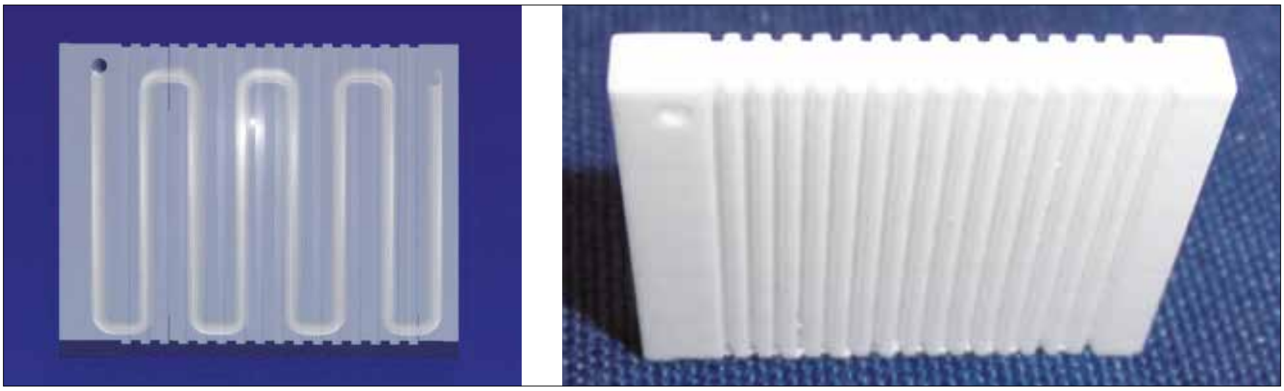

Fig. 5
Ceramic heat exchanger (Al_2O_3), CAD-model, and produced component

In Fig. 2 the 3D printed ceramic stirring elements made of Al_2O_3 are shown which are built in the current laboratory setup of the extraction column. These are form-fit elements which are attached to the steel shaft used in the initial function tests.
In the next development step the steel shaft is to be replaced by full ceramic elements. This prevents the contact of steel with the media that flow through the column. Screw threads were added to the ceramic stirring elements so the whole stirrer can be assembled by screwing single stirring elements into one another (Fig. 3). Additionally the length of the stirrer can be adapted quite easily by adding different stirrer- and intermediate elements. The challenge is to consider the different sintering shrinkage in horizontal and vertical direction of 3D printed parts.
Fig. 4 shows the ceramic cage with an intermediate plate. The advantages of 3D printing as a manufacturing process became apparent during the development process. Design changes of single components could fast and easily implemented.
For the technical implementation 3D printing brings these advantages to bear so the single components can be adapted easily to specific process requirements without the requirement to build or change manufacturing tools.

Integrative design with ceramics and additive manufacturing

Additive manufacturing can be easily incorporated into the process of integrative construction with ceramics [3]. The production method is the logical extension to the

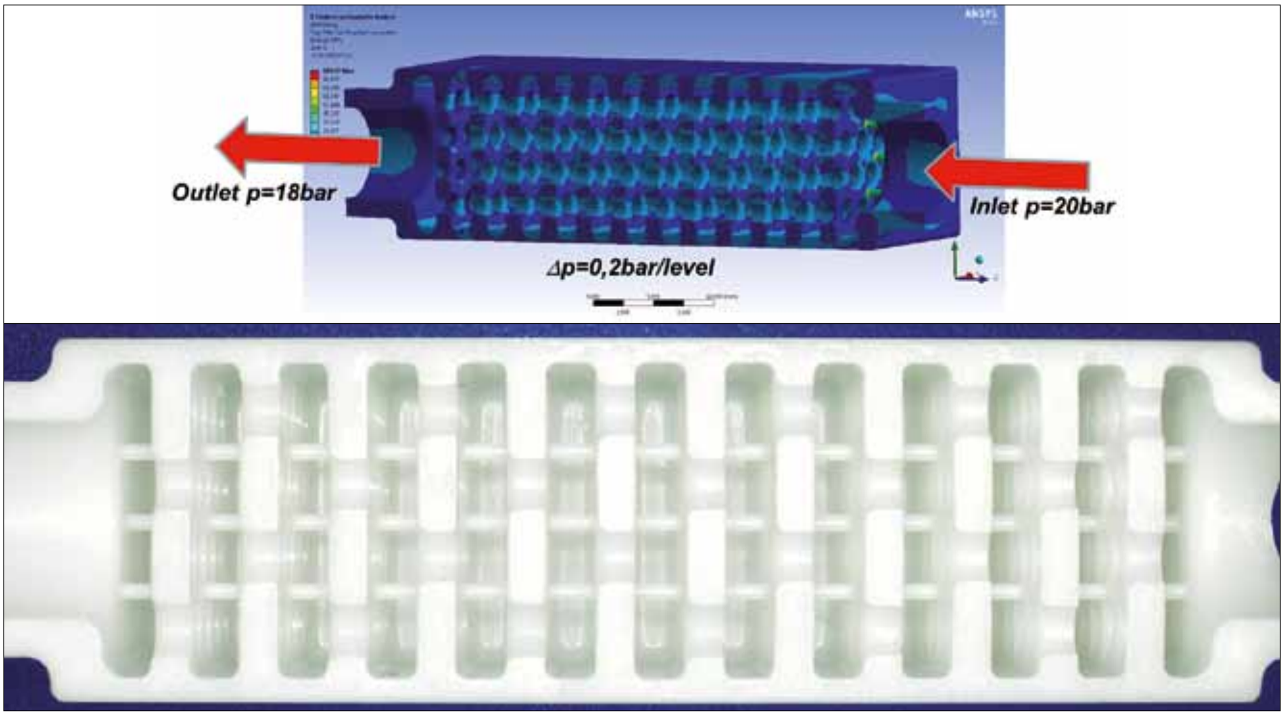

Fig. 6
Ceramic heat-finger (Al_2O_3), FE-model, and produced component (cut open)

component designs derived from the FEA. With the additive manufacturing technique optimized designs from FEA calculations can be produced in time without additional tooling costs. Furthermore components with internal structures for e.g. heat exchangers can be realized. Conventionally such components can only be manufactured by single components that have to be joined in a second step. As an example a heat exchanger in Fig. 5 is shown which was produced by 3D printing. The component has a channel structure enclosed with in- and outlets on the front and back. With the existing know-how LAPP Insulators Alumina GmbH is able to join metallic flanges vacuum-tight directly to the ceramic component. With this joint it is possible to qualify the ceramic-metal-assembly for inner pressures and high temperatures up to 450 °C.

The consequent implementation of the FEA-component-design is shown in Fig. 6 using the example of a cryo-heat-finger. By using the FEA the heat-finger could be designed that the mechanical load would not lead to a critical risk of rupture F of the component during operation. In this case the pressure at the inlet was 20 bar with a pressure drop of 0,2 bar/level.

For the design optimization the new degrees of freedom which 3D printing offers to the construction engineer could be used. After the design freeze the component was manufactured.

The small scale extraction column with ceramic elements was developed and tested in cooperation with TU Dortmund University within the framework of a master thesis. The extraction column will be exhibited at the ACHEMA 2015 fair (15.–19.06.2015) Hall 9.2, booth A66.

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