

# Use of additive manufacturing processes for the production of glass-lined process equipment

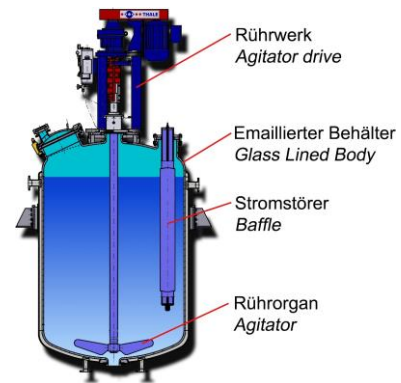
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## Introduction

Additive manufacturing processes are gaining increasing significance in the production of complex components. In contrast to the usual subtractive manufacturing methods to date such as turning, milling and drilling, in additive processes a work piece is built up in layers. That is to say, material is added instead of removing it by machining. The parts are manufactured directly from a digital data model of the geometry of the component made of formless (e.g. liquids or powder) or form-neutral (e.g. strips, rods, wires) materials using chemical and/or physical processes. Additive manufacturing processes are to be categorised under primary forming processes (such as casting, for example). Compared with primary forming processes to date, in order to produce a tangible product no tools are necessary in which the geometry of the work piece is "stored". Glass-lined equipment and systems have a solid place in processes in the chemical and pharmaceutical industry. A high degree of chemical resistance of the glass-lined surfaces in contact with the product, together with easy cleaning are the optimum requirements for highly corrosive chemical processes in multi-purpose systems.



*Fig. 1: Schematic drawing of a glass-lined reactor*

A glass-lined reactor (Fig. 1) comprises a **tank** with varying volumes. Today reactors can be manufactured with volumes up to 100 m<sup>3</sup>. On average the size of reactors today are between 8 m<sup>3</sup> and 16 m<sup>3</sup>. In the case of a 16 m<sup>3</sup> agitated tank the diameter of the reactor is 2,600 mm, the height of the tank about 3,750 mm and the glass-lined surface which is non-porous and flawless in accordance with DIN EN ISO 28721-1 is over 40 m<sup>2</sup>. In the case of a 100 m<sup>3</sup> tank the non-porous glass-lined inner surface is over 150 m<sup>2</sup>! The tank is fitted with a series of nozzles on the top and bottom of the tank, which the operator uses to introduce media or for emptying. The **agitator** is built above the so-called agitator nozzles in the middle of the top of an agitated tank. An electric motor via a gear box drives the agitator shaft, protruding into the tank, on which the agitating elements are fitted. Using a shaft seal the agitator shaft is sealed to be pressure-tight with the agitated tank. So-called **baffles** are installed in one or more nozzles on the top of the tank. Baffles are essentially tubular fittings which, together with the agitator, ensure first of all that the tank contents are thoroughly mixed. Besides these fittings other elements can also be used, such as, for example, dip pipes, feed pipes, sensor carriers to measure process parameters (temperature or pH-value), probes to monitor the glass lining etc. Glass-lined surfaces are highly resistant to strong acids, free from heavy metal ions, smooth, anti-adhesive and thus easy to clean. Furthermore, glass-lined equipment is, in many cases, a particularly economic solution compared to equipment made of high alloy stainless steels or materials such as nickel-based alloys and tantalum. Technical glass-lined reactors therefore have an established place in a multitude of process-engineering processes and often form the "heart" of the processes.

## Objective

Within the remit of a development project the potential applications and limits of additive manufactured components were comprehensively investigated at THALETEC. The development project pursued the following aims:

- Evaluating the benefit of the "new" technologies for process engineering taking into consideration the THALETEC core competency of "technical glass".
- Recognising the possibilities of the technology and its implementation in new kinds of technical solutions
- Developing guidelines for the design and development of additive manufactured glass-lined components
- Developing suitable systems of materials for the production of components which can be enamelled
- Developing new technical solutions and products on the basis of additive manufactured glass-lined components

## Important fundamental principles of additive manufacturing

Today there is a number of processes for additive manufacturing, which are differentiated mainly by the systems of materials used. Processes in which components can be manufactured from plastic are, for example, Fused Deposition Modelling (FDM) or stereolithography. Selective Laser Melting (SLM) however, is a process by which metallic components can be built up in layers. It is common to all processes that the data to guide the tool (jet or laser beam) is created from a 3D-CAD object using software. In the first calculation step the component is divided into individual layers. In the second calculation step the paths (vectors) are created for each layer, which the tool marks out. Processes relevant for THALETEC are FDM as well as SLM, which will be explored further below.

### Fused Deposition Modelling (FDM)

Fused Deposition Modelling describes a manufacturing process from the field of Rapid Prototyping, in which a work piece is built up in layers from a meltable plastic. Machinery for FDM belongs to the machinery class of so called 3D-printers. This process is based on liquefaction of a wire-shaped or rod-shaped plastic material by heating. The material solidifies when it then cools down. The material is applied by extrusion using a heated nozzle which is movable in the manufacturing area. The layer thicknesses vary between 0.025 mm and 1.25 mm depending on the specific application, with wall thickness of at least 0.2 mm. In the manufacture of the model in layers, the individual layers combine to form a complex piece. Projecting components can be created in this process with support structures which can be removed after manufacture.

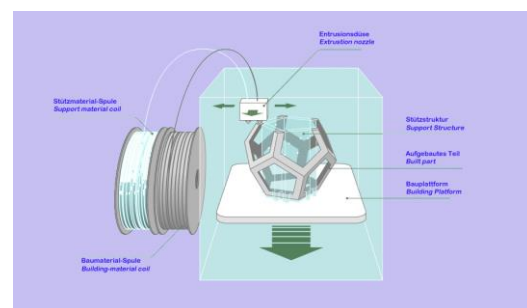
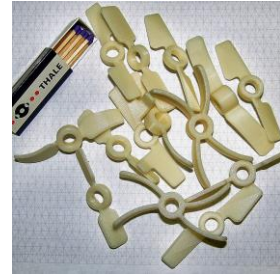


Fig. 2: Fused Deposition Modelling; basic diagram (ADDITIVELY-01, 2014).

For several years THALETEC has been using a 3D-printer, which manufactures models made of ABS-plastic (ABS=Acrylnitrile-Butadiene-Styrene), in order to develop, inter alia, prototypes for new kinds of agitating elements and to test and optimise these in its own technical agitating centre. In so doing, not just all standard agitating elements were/are tested and further developed, but also specialised agitating systems for our customers "printed" and tested as a scale

model. Depending on the FDM system used, components up to the original size (up to 2 m diameter) are possible, whereby limits are set for the stability of the components on the basis of the material.

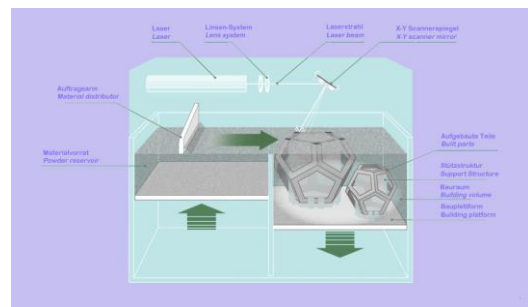
*Fig. 3: Models of agitating elements, manufactured using FDM process, for carrying out mixing tests on a laboratory scale and without chemical/thermal demands.*



Although many relevant process engineering parameters of agitating systems can be shown in model systems, there are some limits to the ABS material which do not permit the use of such components in real-life systems subject to high thermal and chemical demands which also occur in the case of glass-lined equipment.

## Selective Laser Melting (SLM)

In Selective Laser Melting the material to be processed is applied as a powder in thin layers on a base plate. Using a laser beam the powder layer is completely remelted in situ and after hardening forms a solid layer of material. After that the base plate is lowered by the thickness of a layer and powder is applied again. This cycle is repeated until the work piece is completely built. The completed component is cleaned of the excess powder, reworked as required or used immediately. Typical layer thickness for materials used is between 20 and 100  $\mu\text{m}$ .



*Fig. 4: Selective Laser Melting, basic diagram (ADDITIVELY-02, 2014).*

In order to avoid contaminating the material with oxygen, the process takes place in an inert gas atmosphere on the basis of Argon or nitrogen. Components manufactured by Selective Laser Melting are characterised by component thicknesses  $> 99\%$ . Together with the adjustment parameters and the operating parameters of the SLM system it can be guaranteed that the mechanical properties of additive manufactured components correspond to a great extent to those of a conventionally molten base material. In contrast to conventional primary forming processes (casting process) laser melting is characterised by the fact that tools or forms are redundant (formless manufacture) and thus time to market can be reduced. A further advantage is the great freedom of geometry, making component shapes possible which cannot be produced with fixed form processes or only at great expense. For SLM systems the following materials systems are currently available (information from Concept-Laser GmbH):

- High alloy steels
- Hot-working steels
- Stainless hot-working steels
- Aluminium alloys
- Nickel-based alloys
- Titanium alloys
- Titanium
- Cobalt-chromium alloys
- Bronze alloys
- Stainless steel alloys

## **Application possibilities and limits of additive manufacturing processes**

By far the greatest advantage of additive processes to manufacture complex geometries is the speed with which the implementation of a developed geometry can be realised in an actual component. In additive manufacturing the component is created directly from geometry generated on a CAD-system. The individual stages necessary in conventional manufacture from purchasing semi-finished products through the design and preparation of drawings, the preparatory work with the development of CAM-programs for machining, programming machines up to complex assembly processes can, in the main, be dispensed with. Thus additive manufacturing supports the objective of notably reducing the "time to market" of new products and solutions. As a result of the shortened process chain within the scope of product development in the sense of "Rapid Prototyping", prototypes are available quickly at reasonable cost which can already undergo extensive tests and optimisation at the prototype stage. For tests and investigations on technical agitating components it is also an advantage that prototypes can be manufactured to the exact scale desired using the additive process, which can be reproduced very easily when scaled up, that is to say, rendered in the original scale. Despite the restrictions which currently still apply to the maximum component size which can be manufactured on one piece, it is however possible to also create larger components by joining (adhesive bonding for FDM-manufactured components or welding for SLM-manufactured components). However, specialised FDM-manufactured components are also restricted in their application: the material, usually ABS, is not very strong so that highly resilient components cannot be manufactured using this. In addition, the surface quality of the plastic components is not always the best and requires finishing by hand. Therefore FDM-manufactured components can only be used successfully currently in process engineering applications for prototypes in test media and under (non-critical) test conditions in terms of temperature and corrosiveness.

## **Investigations on the suitability of enamelling additive manufactured components**

Therefore it was necessary to subject the SLM process as well as the systems of materials currently available for this process to a more precise examination in respect of the potential application in process engineering equipment and in particular to examine the suitability of enamelling additive manufactured components. In all considerations it was important that in respect of the enamelling systems available at THALETEC and certified by numerous customers and the application characteristics of the technical enamelling there should be no changes of any kind or even smears in respect of the enamelling of non-additive manufactured components; thus ruling out systems of materials which were not compatible with the THALETEC enamelling systems, RAS blue, RAS white, TPE (THALETEC PharmaGlass) and ABRISIST (highly wear-resistant technical glass). The following systems of materials were examined in respect of the suitability of enamelling with the existing technical glass systems, enamel adhesion, thermal shock behaviour, radii stability, "enclosed enamelling", surface properties of the SLM-manufactured component, microstructures and porosity, machining properties as well as weldability:

- (Modified) Nickel-based alloys: T25 and T26
- High alloy stainless steel: T13, T16
- Hot-working steel: T19

## **Suitability of enamelling and enamel adhesion**

Whether a component can be enamelled depends on a number of constraints. An important role is played here by the enamel system as well as matching the enamel system to the properties of the base material. In addition, the geometry of the component to be enamelled also has an impact on its suitability. Whilst the material properties are determined by the composition of the materials and the method of manufacture, the geometric properties of the components are mainly influenced by their function. Only the harmonisation of the material and geometric properties

will lead to a glass-lined product with substantial customer benefits. The fundamental suitability of enamelling each base material examined is the main prerequisite and basis for all further considerations so that these tests formed the beginning of the investigation. In order to test these, samples of SLM-manufactured components were enamelled using THALETEC's usual enamel systems. The tests were conducted on "simple", as well as complex samples in order to evaluate, if possible, all constraints occurring in subsequent practice during the scope of the tests. A test specimen was designed in accordance with Fig. 5 for the test enamelling. This SLM-manufactured component essentially has all more or less critical geometries: radii in one, two and three dimensions of varying measurements, concave and convex radii as well as flat surfaces. In addition, the component also has different wall thicknesses in order to be able to examine the sensitivity to tensions as a result of varying cooling down curves.

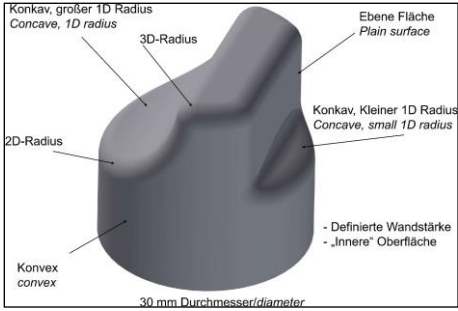


Fig. 5: Geometry of the test specimen.

Fig. 6 depicts the results of many enamelling tests on the test specimen shown. The enamelling was conducted without a problem with all of THALETEC's usual chemical enamel. The test specimen had an effective enamel layer thickness of about 0.8 mm, which corresponds to a minimum layer thickness of 1.1 mm in the usual enamel in process engineering equipment and components and thus complies with the relevant quality standards for glass-lined equipment (DIN EN ISO 28721-1).

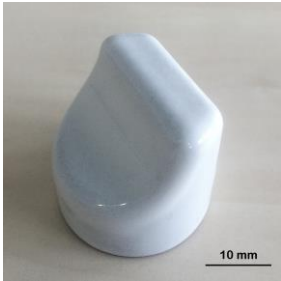


Fig. 6: Glass-lined test specimen to examine the enamelling suitability and the radii stability.

The reaction to impact stress was further examined using an impact test with a Wegener gun (stage 5). A close inspection of the impact pattern showed a good to very good adhesion of the glass lining on the substrate. The remains of the impact is sharply restricted and there is only a radial crack structure without web-shaped chipping on the edge, (see Fig. 7).

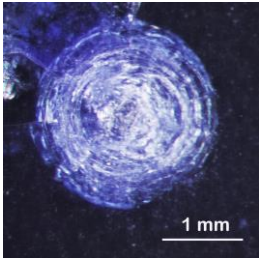


Fig. 7: Impression on the surface after firing pin impact test

## Microstructure and porosity

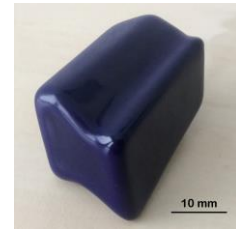
The SLM-manufactured components have a surprisingly homogenous enamel structure and a microstructure to a great extent free of inclusions and cavities. Although the manufacturers and suppliers of SLM systems define a residual porosity in the range of 1 % of the component volume, in practice it has been shown that a residual porosity significantly below 0.5 % is achievable by the correct and optimum adjustment of the process parameters. In images of polished specimens of SLM-manufactured components there are finely distributed air pockets in the order of magnitude of approx. 1 µm at the most. These pockets have no effect on the behaviour of the enamel of the SLM-manufactured component.

| Porosität [%] porosity |  | 0,42 |
|------------------------|--|------|
|                        |  |      |
|                        |  |      |

*Fig. 7: Polished section through SLM-manufactured components. The images show polished sections close to the build platform (top left, from the centre of the component (top right) and in the area of the last layers applied (bottom left).*

## Enclosed enamelling

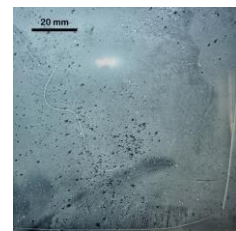
Because of the minimal presence of very small cavities in the base material there was firstly a fear that this could cause outgassing during the burning process. Outgassing is particularly undesirable in components for which the enamelling is meant to be carried out "enclosed". Because of the enamel layer covering the entire surface, gases which leak from the work piece cannot escape. Below the enamel layer there then develops a zone of defective enamel adhesion and because of the gas pressure tensions occur in the enamel layer which lead to chipping. To be able to assess this risk, enamelling tests were conducted on completely enclosed glass-lined components which also had a very complex geometry with very tight radii (Fig. 9). Exactly like the test specimen to test the suitability for enamelling and the radii stability, this test specimen was coated with all of the chemical enamel available at THALETEC and after enamelling aged at temperature for a few days. It showed that even with enclosed enamelling there was no outgassing. Furthermore the phenomenon of chipping caused by hydrogen release (fish scales) was not observed. The main reason for this is the choice of base material, the alloy components of which work as hydrogen traps. The geometry of the test specimen is also suitable for testing the radii stability of the enamelling on the base material. Up to a glass lining thickness of about 1 mm it is possible to enamel radii of 2 mm with a high degree of process reliability. Usually one aims for radii of at least 6 mm in process engineering glass-lined components, so that the easily achievable 2 mm radii represent a significant improvement in the design feature. Thus the design engineers have new design options which they have not been able to realise for glass-lined components to date.



*Fig. 8: Completely enclosed glass-lined test specimen.*

## Thermal shock and weldability

The thermal shock behaviour of the material was tested using a SLM-manufactured sample sheet made from the materials system T26 which is particularly suitable for enamelling. First of all, the test was conducted on a sample sheet 150x150x10 mm, which was halved for the second test and using an appropriate welding method was applied. By using this method the thermal shock properties of the base material, the thermal shock properties of a welded joint as well as the fundamental weldability of SLM-manufactured components made from the materials system T26 could be tested. For the welding tests the sample sheet was halved and a welded seam prepared in a V shape with a welding gap of 3 mm. Then the welded seam was welded with a compatible filler material in accordance with the welding parameters appropriate to the materials system in the WIG process and smoothed out. After enamelling in accordance with DIN EN ISO 13807 (crack formation temperature in the quenching test) the thermal shock test was then conducted. For the homogenous test sheet as well as the welded test sheet a crack formation temperature in accordance with DIN EN ISO 13807 of 190°C was determined. This lies within the permitted tolerance.



*Fig. 9: Thermal shock test on welded test sheet. Cracks made visible with the Statiflux test.*

## Machinability

The materials system T25 and T26 relates to high alloyed special steels modified for enamelling which are very tough and highly ductile. These properties must be taken into account on machining the components. In principle these materials can be machined without a problem, but only at low feed rates tailored to the materials system and correspondingly low machining forc-

es. In addition, tools must be used which are suitable for the materials system. These are, for example, milling cutters, turning tools and drills made of carbide.



*Fig. 10: Example of machining tests. On a SLM-manufactured component widths across flats were reworked and thread tapped.*

## **Application example: Temperable agitating element for glass-lined agitated tank**

In a development project a specialised agitating element was manufactured for the LabTec-Programme of THALETEC (THALETEC-01, 2014). LabTec includes process engineering components and equipment specifically for applications in the laboratory and the technical centre. The agitating element is a component additive manufactured using the SLM process which was then enamelled. The agitating element has some special features: thus the agitating blades are hollow inside. The cavity in each of the blades is connected with two ring channels. The surface inside the agitating element also has tapered projections. The cavities together with the ring channels in the hub of the agitator help to temper these. In so doing a liquid medium is fed via a ring channel, then flows through the agitating blades and exits via the second ring channel from the agitating blades. The structured surface inside the agitating blades supports the formation of a turbulent flow and thus leads to an improvement in the heat transfer. The minimum wall thickness of the agitating blades was determined as 3 mm using the Finite Element Method on the basis of the stress data of the agitator (torque, drive power).

### **Procedure in developing the agitating element**

Meeting the process engineering objectives set is, of course, at the forefront of the development of agitating systems. In the case of agitating systems these are properties such as mixing time, the power used by the agitators in stirring liquids, the capacity to stir up solids etc. Many of these properties can already be determined by using tests to scale. In an initial development stage therefore plastic models of agitating elements were made using additive manufacturing from ABS plastic (3D printed) using a FDM system to scale on the basis of a 3D-CAD model. These models were then measured in a glass vessel and the technical agitating characteristics determined. As the additive manufacture is carried out solely on the basis of CAD-data, at this development stage variations in the geometry can be effected quickly, simply and inexpensively. Because of the very precise geometry of the scaled down FDM-manufactured components the measuring results also lie within the parameters of the accuracy required. After this iterative process of developing the external shape of the agitating element, the concept and design of the inner structure of the component took place. It was shown that the inner geometry of the component is influenced by the requirements of meeting the functional demands, as well as by the requirements arising from the generative manufacturing process. For example, it must be ensured when designing the component that residual powder material can be completely removed from the cavity inside the agitator blade. Furthermore, overhanging structures require supports from a specific flank angle. These support structures are also manufactured from the powder and after the component is complete they must be removed by hand or by sand blasting. Support structures within a component, however, are not accessible and have to remain in the component where they have a negative impact on the function. Therefore when designing the agitator it must be ensured that no support structures are required inside the agitator. This was achieved by the fact that the flank angles of all surfaces which are in the direction of the build platform do not have an angle less than  $45^\circ$ . From angles of  $45^\circ$  and above SLM systems do not require support structures. Using further iteration steps the geometry was then examined and optimised in respect of the stability regarding the mechanical stress occurring during stirring (mainly bending moments on the crossovers between the agitator blades and the agitator hub), regarding pressure loading as a result of the pressure of the heat transfer medium as well as against oscillation. In so doing, it was shown that Additive Manufacture here has a significant

advantage over conventionally built agitators: material can be targeted at those places where there is higher mechanical stress and omitted at those places where stresses are less or where walls must be as thin as possible. It is therefore very easy to design according to stress and in a way therefore which saves materials.

## Results

After the agitator was designed in accordance with the above-mentioned functional requirements as well as parameters arising from the manufacturing process, the additive manufacture took place using a SLM system of the company, Concept-Laser. The result is shown in Fig. 12. The agitator has a diameter of approx. 250 mm and weighs approx. 800 g. It has a build volume of 106 cm<sup>3</sup> and was additive manufactured in about 8 hours. A dimensional check prior to subsequent processing demonstrated that the variance between the target geometry from the CAD-system and the actual geometry on the component was only a small 1/10 mm. As the variance can have positive as well as negative impact it is sensible that surfaces which require a high degree of precision are manufactured with an excess and then finished mechanically.



*Fig. 11: Additive manufactured agitator using SLM process.*



*Fig. 12: Support structures on the component are removed mechanically after generative manufacturing.*

In Fig. 12 the support structures can be clearly seen which are required to generate the component layers which are aligned parallel to the build platform (also see Fig. 13). The remains of these support structures were removed mechanically by grinding and then by sand blasting. Then the component was lined with the highly chemical-resistant THALETEC chemical enamel RAS blue using the usual application method. The burning processes were, however, adjusted to the requirements of the base material as well as the geometric properties of the component (wall thickness, wall thickness transitions), so that the enamel was homogenous and without tension lines and defects. The thickness of the glass lining is at least 1.2 mm overall so that the standard requirements of the enamel are met in full.



*Fig. 13: Agitating element for the THALETEC LabTec-System, manufactured using SLM process and a highly acid-resistant enamel applied. The diameter of the agitating element is 250 mm. Heating and cooling media can flow through the inner cavity.*

The final agitating element is depicted in Fig. 14. The agitating element is as good as a conventionally manufactured component in terms of strength, stability and especially chemical resistance. In comparison with a conventionally manufactured agitating element a SLM-manufactured component can, however, weigh almost half as much, whereby the important critical rotational speed in the fast running agitators can be increased by more than 40 %. In addition, the agitating element can be tempered while a heated or cooled thermal transfer fluid is pumped through the agitator shaft and through the agitator blades. The immensely important thermal transfer in many process engineering processes can see a double digit percentage improvement by the use of such an agitating element.

# Conclusions

Assuming a materials system suitable for enamelling, the manufacture of glass-lined components using Selective Laser Melting (SLM) for applications in process engineering is an extremely suitable method. It makes it possible to have components with a high degree of function integration as well as specialised solutions which would not be possible with conventional manufacturing processes or would be uneconomic to manufacture. Using systematic investigations of all relevant requirements it could be proved that the performance characteristics of additive manufactured glass-lined components do not differ from conventionally manufactured components made of the usual materials to date (e.g. fine grained steels). Two aspects should be noted: Additive manufacture, like other "conventional" manufacturing processes, whilst having many possibilities is also subject to limits and restrictions which must be taken into account in the development of products and solutions. The greatest restriction within the scope of the design configuration of the components manufactured by THALETEC to date has proven to be the necessity of support structures which must be subsequently removed. This significantly restricts the freedom of the design, especially if components are to be manufactured with inner structures (channels, cavities, etc.). Secondly, it has been shown that it is not always sensible to use additive manufacture. Whenever the manufacture of a component is possible using conventional processes (reshaping, welding, turning, milling, etc.), it is sensible to carry out a cost-effectiveness review of "conventional versus additive" manufacture. Additive manufacture does not always come out as the most cost-effective solution! Only when components are to be manufactured which cannot be produced conventionally, can additive manufacturing display its advantages to the full. Certainly the design engineer needs to change his thinking: whilst to date the possibilities and limits of the conventional manufacturing process implicitly determined the configuration of the designed component, these limits are stretched by the possibilities of additive manufacture. Only when the developer and design engineer have taken on board these possibilities, will he be in a position to deal in the right way with the tool box now available to him in the additive manufacturing process and find really new solutions and not just adapt existing technical solutions. For THALETEC the possibilities of additive manufacture open up a brand new spectrum of innovative technical solutions which can now be implemented step by step in new products with many customer benefits.

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