

Additive manufacturing and vulcanization of natural and synthetic rubbers

Additive Fertigung und Vulkanisation von Natur- und Synthesekautschuken

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Additive manufacturing of thermoplastics and metals is a sustainable and established process in industry for the rapid production of individual technical components. For a long time, this technology was not accessible for the group of elastomers, or only to a limited extent in the form of thermoplastic elastomers or silicone rubbers. The development of the Additive Manufacturing of Elastomers (AME)-process has enabled the additive manufacturing of high viscosity rubbers. In future, additively manufactured rubber components may be used in technical logistics in particular. On the one hand, the supply of spare parts such as sealing and damping elements is possible, and on the other hand, the production of individual geometries for grippers in handling technology. For the additive manufacturing of rubber, an industrial 3D-printer was modified by a twin screw extruder, which can process rubber filament and deposit it on a printing plate in strand form, similar to the thermoplastic Fused Filament Fabrication (FFF)-process. The use of a screw extruder is necessary because the viscosity of the rubber does not decrease sufficiently with heating, making it impossible to guide the filament through conventional print heads for thermoplastic filaments. The AME-process is a two-step manufacturing process. First, the components are additively manufactured, followed by vulcanization in a high-pressure autoclave or heating oven. Single-part production is a particular challenge in this case, as the vulcanization time depends on the rubber compound and the component geometry. In order to avoid waste, it is therefore necessary to know the optimum vulcanization time before vulcanization. For this purpose, a simulation was developed and validated that outputs the degree of crosslinking in the component as a function of the vulcanization temperature and time.

[Keywords: additive manufacturing, rubber, heat transfer, vulcanization, tensile testing]

Die additive Fertigung von Thermoplasten und Metallen ist ein nachhaltiges und in der Industrie zur schnellen Herstellung von individuellen technischen Bauteilen bewährtes Verfahren. Lange Zeit war diese Technologie für die Werkstoffgruppe der Elastomere nicht oder nur eingeschränkt in Form von Thermoplastischen Elastomeren oder Silikonkautschuken zugänglich. Durch die Entwicklung des Additive Manufacturing of Elastomers (AME)-Verfahrens ist nun auch die additive Fertigung von hochviskosen Kautschuken möglich. Besonders in der technischen Logistik können zukünftig additiv gefertigte Kautschukbauteile Einsatz finden. Einerseits ist die Bereitstellung von Ersatzteilen wie Dichtungs- und Dämpferelementen möglich, aber auch die Fertigung individueller Geometrien für Greifer in der Handhabungstechnik. Zur additiven Fertigung von Kautschuk wurde ein industrieller 3D-Drucker um einen Zweisechneckenextruder erweitert, der Kautschukfilament verarbeiten und ähnlich zum thermoplastischen Fused Filament Fabrication (FFF)-Verfahren strangförmig auf eine Druckplatte auftragen kann. Der Einsatz eines Schneckenextruders ist notwendig, da die Viskosität des Kautschuks nicht ausreichend durch Erwärmung abnimmt und somit eine Führung des Filaments durch konventionelle Druckköpfe für thermoplastische Filamente nicht möglich ist. Das AME-Verfahren ist ein zweistufiges Fertigungsverfahren. Zuerst werden die Bauteile additiv gefertigt, anschließend folgt die Vulkanisation in einem Hochdruckautoklav oder Wärmeschrank. Hierbei ist besonders die Einzelteilfertigung eine Herausforderung, da die Vulkanisationszeit abhängig von der Kautschukmischung und der Bauteilgeometrie ist. Um keinen Ausschuss zu produzieren ist es daher notwendig die optimale Vulkanisationszeit vor der Vulkanisation zu kennen. Hierfür wurde eine Simulation, die den Vernetzungsgrad im Bauteil in Abhängigkeit von der Vulkanisationstemperatur und -zeit ausgibt, entwickelt und validiert.

[Schlüsselwörter: Additive Fertigung, Kautschuk, Wärmeübertragung, Vulkanisation, Zugversuche]

1 INTRODUCTION

Additive manufacturing of thermoplastics and metals is meanwhile a common manufacturing process, which is used especially in prototyping and spare parts production [1]. With the exception of silicone rubbers [2] and thermoplastic elastomers [3], additive manufacturing of elastomer components was not possible for a long time. With the development of the so-called Additive Manufacturing of Elastomers (AME)-process for high-viscosity natural and synthetic rubbers [4] and another process for liquid rubbers [5, 6], additive manufacturing of conventional rubber-based elastomers is also becoming possible.

It should be noted that additive manufacturing of these elastomers is always a two-step process. In the first step, the components are additively manufactured using a 3D-printer before they are vulcanized in the second process step, which gives them their rubber-elastic properties [7]. Since non-vulcanized rubber tends to flow and is consequently not dimensionally stable [8], a form-giving shell of thermoplastic is printed serially [4] (Figure 1).

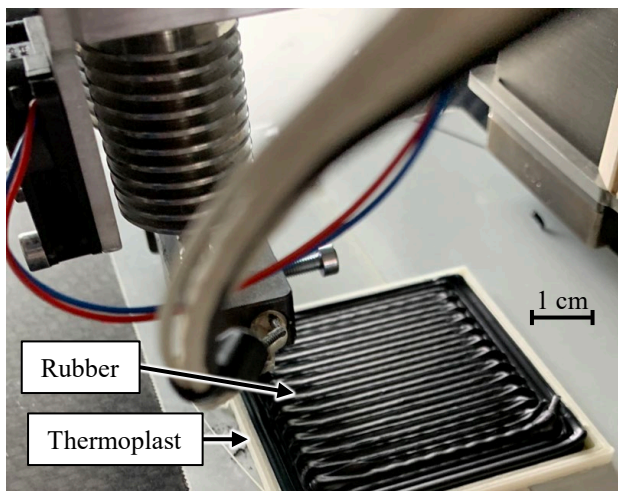


Figure 1. Printing of a rubber component and its shell

In this paper, the AME-process is considered for additive manufacturing of high viscosity natural and synthetic rubbers. Previous publications have focused on the development of the AME-3D-printer [7], the generation of a printable G-code, the additive manufacturing itself, and the qualification of the process using tensile testing, demonstrating that additively manufactured components can compete with conventionally manufactured components [9]. In comparison, the focus of this paper will be on vulcanization. Vulcanization is a time- and temperature-dependent process. If a component is not sufficiently vulcanized, it is said to be undercured and the rubber-elastic properties have not yet fully developed. If a component is vulcanized too

long or at too high temperature, it is said to be overvulcanized, resulting in reversion and associated embrittlement of the component, which is also disadvantageous for the rubber-elastic properties [8].

Up to now, the vulcanization time has been determined experimentally for each component by preliminary tests [9]. However, this is neither sustainable nor economical, especially for the production of individual components or single spare parts. Therefore, for the AME-process to be economically used, the vulcanization time for each component must be determined before the real vulcanization process. For this purpose, a method for the simulative determination of the vulcanization time in a heating oven is presented in the main part of this paper, and it is also validated on the basis of tensile tests. First, however, the developed AME-3D-printer and the process chain of the AME-process are presented. The paper closes with potentials of the AME-process for technical logistics which are presented on three example components and a summary of the central results.

2 AME-3D-PRINTER AND PROCESS CHAIN

The development of the AME-3D-printer has been described in detail in previous articles [7, 9]. Nevertheless, this section will briefly introduce the printer (Figure 2) and the process chain of the AME-process (Figure 3). The 3D-printer is originally based on a fused filament fabrication (FFF)-3D-printer, to which a twin-screw extruder was added to process rubber. The two existing print heads for thermoplastics were retained. In addition, a freely rotating wheel was integrated to provide rubber filament. In figure 2, the travel paths of the printer are also shown in red. The printer has a print area of 500 mm x 500 mm x 250 mm.

The AME-process consists of six steps. In the first step, the component to be printed is designed in a CAD program. In the next step, a thin-walled shell is created for the component. This shell is necessary because the non-vulcanized rubber is not dimensionally stable. However, because the rubbers used are so highly viscous, shells are used only for overhangs, bridges and for high components that begin to flow under their own weight. Both the part and the shell are saved in STL format, as this is necessary for generating the G-code using slicing software in the third step. The G-code is then further modified to the AME-process, this procedure has already been described in detail in a previous publication [9]. The fourth step is the 3D-printing of the component and its eventual shell. Here, the rubber component is printed using a rubber filament. The shell is printed using thermoplastic material. It is essential that in each layer the shell is printed before the component to avoid any flow, this is ensured when modifying the G-code. After printing, vulcanization follows either at atmospheric pressure in a forced air oven or in a high-pressure autoclave. Finally, in the last step, the shell is removed from the component and, if necessary, the component is post-processed.

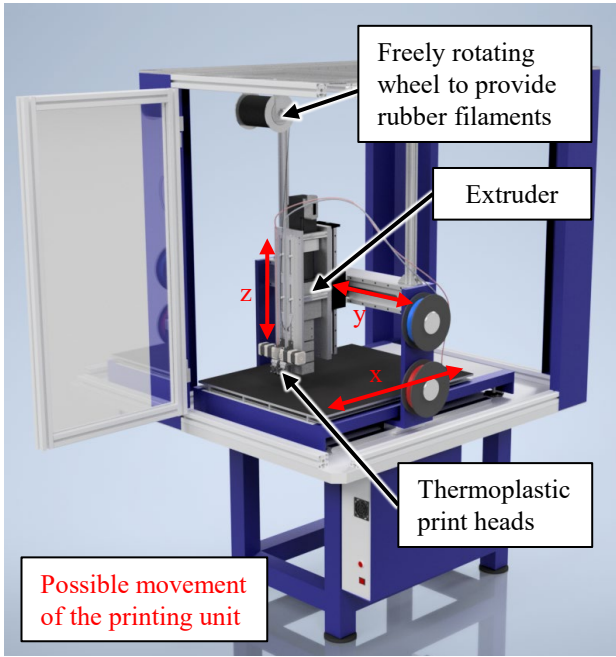


Figure 2. Setup of the AME-3D-printer

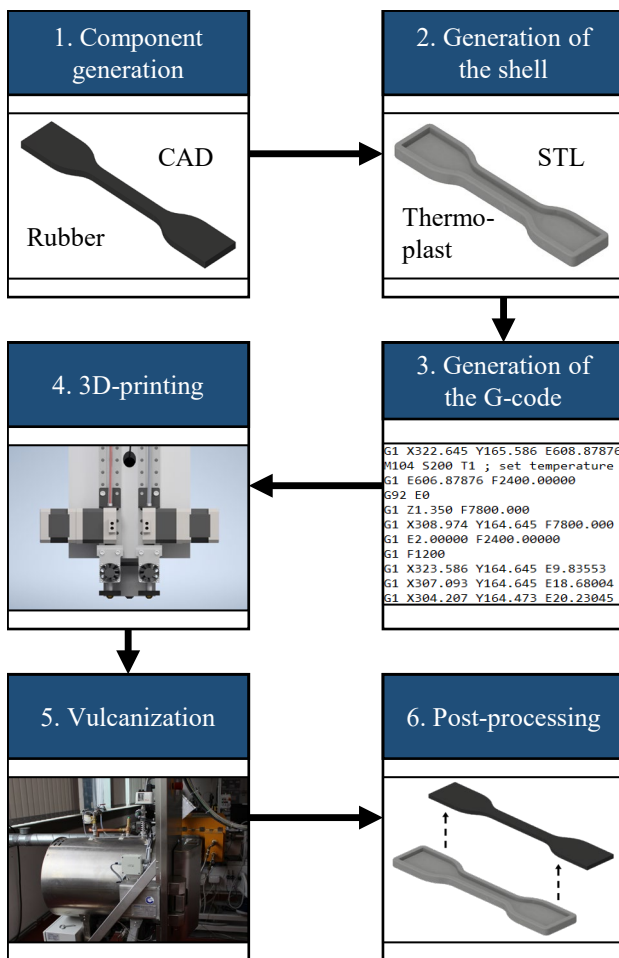


Figure 3. Process chain of the AME-process

3 METHOD FOR THE SIMULATIVE DETERMINATION OF THE VULCANIZATION TIME

Up to now, vulcanization has received limited attention. However, in order to be able to use the AME-process economically, it is necessary that vulcanization time is known before the real vulcanization process and does not have to be determined experimentally for each component. For this purpose, a simulation model was developed in ANSYS which simulates the heat transfer in the component and links the location- and time-specific temperature in the rubber component with the time- and rubber compound-specific crosslinking isotherms. The crosslinking isotherms, under consideration of temperature, determine the time-dependent degree of crosslinking [8]. These crosslinking isotherms must be recorded for each compound at different temperatures using a vulcameter. The recorded torque of the vulcameter correlates directly with the degree of crosslinking [8]. For hydrogenated nitrile-butadiene rubber (HNBR) and nitrile-butadiene rubber (NBR) compounds regarded in this paper, the temperatures of 120 °C, 130 °C, 140 °C, 150 °C and 160 °C were observed. At lower temperatures, vulcanization takes longer, at higher temperatures, reversion already occurs during vulcanization. Figure 4 shows a cuboid cut open in the middle in the simulation environment. Figure 5 shows exemplary the torque curves of the vulcametry of HNBR at different temperatures.

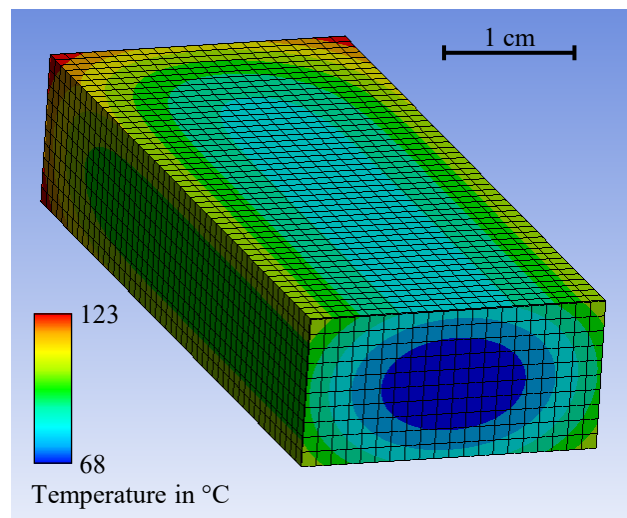


Figure 4. Simulation of heat transfer during vulcanization in a cuboid

At the beginning, the temperature of the component is at room temperature and the forced-air oven is preheated to the vulcanization temperature. Thermal-transient analysis in ANSYS was used to simulate the forced convection [10]. For this purpose, a heat transfer coefficient of 20 W/(m²K) was validated in preliminary tests for the oven [8]. The following standard values are also assumed for rubber compounds: A density of 1200 kg/m³, a specific heat capacity of 1860 J/(kg*K) and a thermal conductivity of

0.26 W/(m*K) [8]. When the component is placed in the preheated oven, the temperature of the component increases over time. The temperature drop in the oven due to opening the oven door is not considered in the heat transfer simulation.

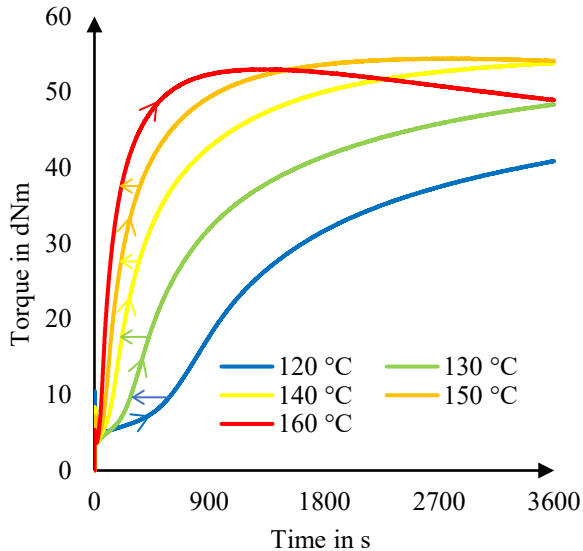


Figure 5. Exemplary crosslinking process along crosslinking isotherms of an HNBR-based rubber compound

It can be seen that the component heats up from the outside to the inside (Figure 4). If the component is in the temperature range from 115°C to 125°C, it is supposed that the vulcanization proceeds as shown on the first isotherm (blue curve, Figure 5). If the temperature in the component is above the first temperature corridor, the torque achieved so far is determined. The determined torque serves as starting point on the next higher crosslinking isotherm, which in this case represents the temperature range from 125 °C to 135 °C. Crosslinking now proceeds according to the curve of this crosslinking isotherm until this temperature corridor is also exceeded. This process continues until the last isotherm is reached. The vulcanization is carried out up to the vulcanization time t_{90} . This time corresponds to a degree of crosslinking of 90%, which correlates with 90% of the maximum torque absorbed by the crosslinking isotherms. According to the definition, complete vulcanization then takes place over the cooling time [8]. The degree of vulcanization can be defined in relation to the location in the component by the temperature distribution in the component. After all, the most critical point in the component should be vulcanized as well as possible.

4 VALIDATION OF THE SIMULATION BY TENSILE TESTS

To validate the simulation model, the calculated vulcanization time is compared with experimentally determined data. For the experimental data, rubber components

are vulcanized in an oven for different durations. In this case, the components are not printed components, instead they are pressed components. Pressed components can be used because the printed components are also full-volume components [9]. This means that the results obtained can be transferred directly to the printed components. Three samples are produced for each vulcanization time. The vulcanized specimens are then tested for their tensile strength. The tensile strength is used to draw conclusions about the crosslinking during vulcanization. The higher the tensile strength of the specimen, the higher the degree of crosslinking. The highest tensile strength achieved is synonymous with the highest degree of crosslinking and the optimum vulcanization time [11, 12].

From the tensile test results, the mean value and the standard deviation for the tensile strength are determined for each vulcanization duration. The validation takes place using two exemplary components. On the one hand, an S2 tensile bar made of HNBR rubber and, on the other, a cuboid made of NBR rubber with the dimensions 100 mm x 20 mm x 10 mm are investigated.

Figure 6 shows the simulated heat transfer of the S2 tensile specimen under investigation. The figure shows the temperature profile in the tensile specimen after 60 seconds of vulcanization. The color gradient from blue for lower temperatures and red for higher temperatures shows which areas in the component vulcanize faster and which more slowly. The areas that vulcanize fastest in the tensile specimen are the corners of the component. The two areas that vulcanize the slowest are located in the blue area inside the tensile specimen. From the simulation model, an optimum vulcanization time of 695.5 seconds was determined for the HNBR tensile specimen.

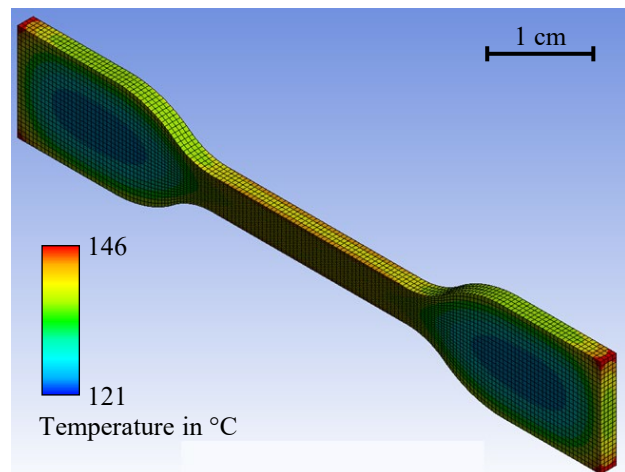


Figure 6. Simulation of heat transfer during vulcanization in a tensile specimen

The arithmetic mean of the tensile strengths of the tensile specimens is shown in Figure 7. The highest tensile strength is achieved by the tensile specimen with a curing time of ten minutes. Based on the diagram, a rapid increase

in tensile strength can be seen before reaching the maximum at ten minutes. Between the vulcanization times of five and ten minutes, the tensile strength increases by 8.5 MPa. Compared to the rapid increase before the maximum, the tensile strength decreases only slightly after the maximum. Five minutes after the maximum, the tensile strength decreases by 1.5 MPa and after 25 minutes the difference stabilizes at 1.3 MPa. Overall, the experimentally determined and the simulated optimum vulcanization time are close to each other. Since the test intervals are five minutes, it is possible that the optimum value is after the 600 seconds and thus was not experimentally recorded. Due to the high difference before the maximum and the lower difference after the maximum, it is less critical to cure the component slightly longer than too short.

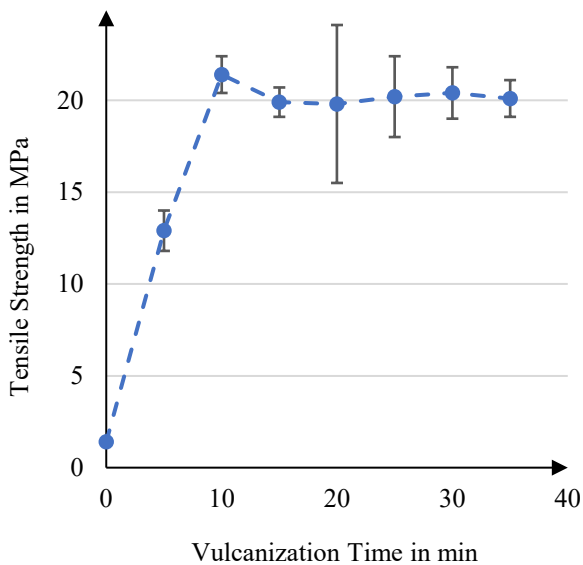


Figure 7. Tensile strength of tensile specimens made of HNBR with different curing times

Figure 4 already shows the heat transfer simulation of the cuboid with the dimensions 100 mm x 20 mm x 10 mm. From the color gradient, it can be seen that the corners of the cuboid are vulcanized through the fastest. The area that takes the longest is at the center of mass of the cuboid. From the simulation model, an optimum vulcanization time of 1076 seconds was determined for the center of mass of the cuboid made of NBR.

For the experimental determination of the optimum vulcanization time, five single plates with the dimensions 100 mm x 20 mm x 2 mm were tied together to form a cuboid with the dimensions 100 mm x 20 mm x 10 mm with a thin nylon thread and hung in an oven. After the specimens were cured for different lengths of time, the middle plate of each cuboid specimen was tested for tensile strength. The tensile specimens required for the tensile test were punched from the middle plate, since the center of mass of the cuboid is located in this plate, where the area

that is vulcanized the latest is located. Figure 8 graphically represents the tensile strength of the individual specimens. The experimentally determined optimum vulcanization time is 1200 seconds. With a vulcanization time of five minutes less, almost no crosslinking has yet taken place, and the tensile strength is only 4 MPa, while at the optimum vulcanization time it is nearly 16 MPa. If the curing time is five minutes longer, there is only a slight decrease in the tensile strength compared to the optimum. The calculated value of 1076 seconds is close to the experimental value. Due to the test intervals of five minutes, it is possible that the optimum value is before the 1200 seconds and thus was not recorded experimentally. Similarly, the opening of the oven door was not taken into account in the simulation, which means that the brief drop in temperature in the oven is not considered and therefore the simulated vulcanization time is shorter than the real one.

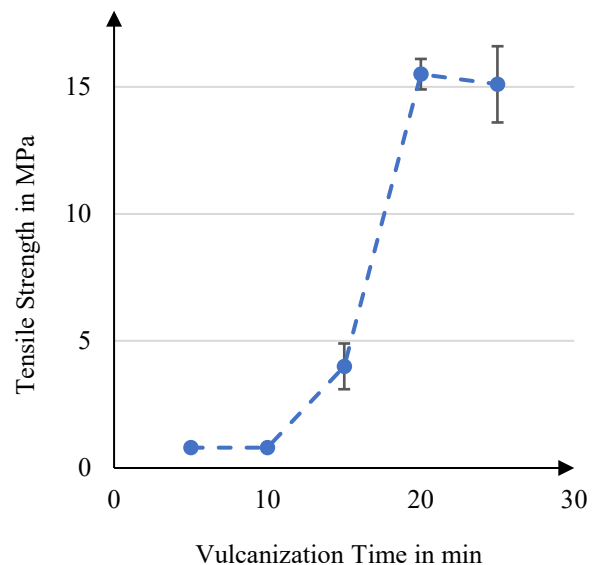


Figure 8. Tensile strength of punched-out tensile specimens from a cuboid made of NBR with different curing times

The vulcanization time simulated in this way should therefore generally serve as a minimum limit for the vulcanization time to be set in reality, which can be extended by a safety factor, since the rubber compounds considered exhibit plateau behavior [8]. Therefore, a slight overvulcanization is less critical than an undervulcanization. The result of the simulation can also be further improved by smaller temperature differences of the crosslinking isotherms.

5 POTENTIALS FOR TECHNICAL LOGISTICS

Additive manufacturing in general, but also additive manufacturing of rubber components in particular, offers great potential in this industry sector. It has already been demonstrated that tensile specimens taken from printed

components or sheets can compete with tensile specimens from conventionally manufactured components and achieve up to 90 percent of the tensile strength and elongation, depending on the printed pattern. On the one hand, even spare parts such as guide rolls (Figure 9), sealing and damping elements can be manufactured on-demand for the company's internal technical logistics processes, which can lead to lower downtimes [13, 14]. Likewise, individual geometries for grippers in handling technology can be manufactured (Figure 10).

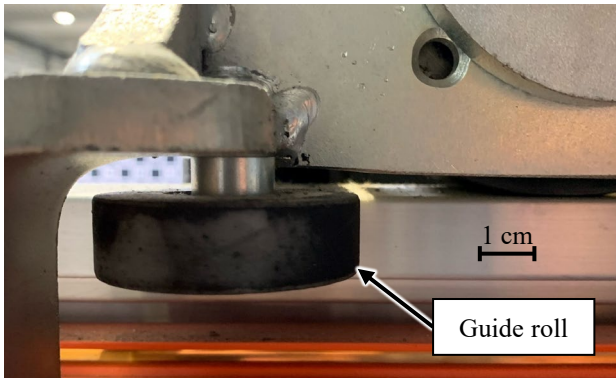


Figure 9. Guide roll of a guidance rail

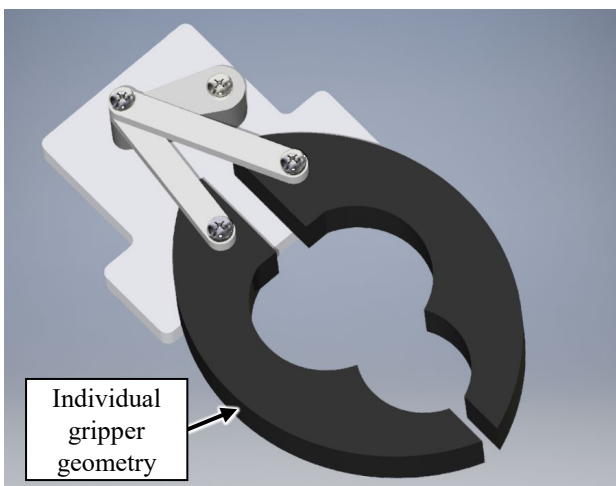


Figure 10. Individual gripper geometry for a handling system

Guide rollers are a very simple component to produce by additive manufacturing, as they have neither overhangs nor bridges. The same applies to the gripper. To give this component the necessary stiffness, layers of thermoplastic material can be printed during the printing process. As a result, the gripper has a high level of adhesion due to the rubber on the surface and, at the same time, high stiffness due to the reinforcement inside the component. Likewise, these components fit easily into the existing print area. More complex components such as a spring bellow (Figure 11) are also printable but the procedure is more complex due to the use of a necessary support structure. For this component in particular, a support structure made of water-soluble thermoplastic PVA should be used, as this is also

required inside the component and would otherwise be difficult to remove.

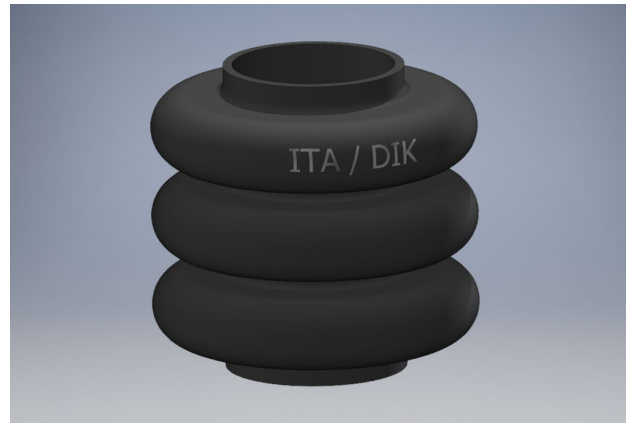


Figure 11. Complex component with overhangs exemplified by a spring bellow

In addition, additive manufacturing can help to shorten in-house production times, as components can be manufactured and shipped to the customer directly after generating a printable file. It is also imaginable that the customer prints its own spare part on its own 3D-printer and thus the company's internal supply chain only consists of providing the printable file [15]. Additive manufacturing can reduce stocks when manufacturing directly on-demand for customers [16]. This also reduces capital locked up in the form of stocks. Especially when rubber component manufacturing companies are contractually obligated to provide spare parts over the entire product life cycle, additive manufacturing of rubber components can help ensure that production is sustainable [17]. Rubber components age due to embrittlement in storage [18]. As a result, stored components may have to be destroyed before they could be shipped to the customer. Demand-oriented production can therefore lead to sustainable and economical spare parts production. The main advantage of the AME-process is that it can be used for single-part production without any problems. Conventional extruder or injection molding systems have to be filled with large quantities of rubber, which makes single-part production uneconomical [19]. Likewise, molds have to be stored for injection molding, which again requires storage space [20]. So for small quantities, additive manufacturing of thermoplastic components is more economical than injection molding. This statement can be applied to the AME-process due to its close relationship to the FFF-process. The cost of additively manufactured components is independent of the number of units. Whereas the cost per component decreases with the number of units in injection molding, for example. Therefore, there is a component-specific break-even point up to which additive manufacturing is more economical than injection molding or extrusion [19].

6 CONCLUSIONS

In previous publications, it has already been shown that additively manufactured rubber components can compete with conventionally manufactured components in terms of their tensile strengths. First, the developed 3D-printer and the process chain of the AME-process were presented, referring to previous publications. However, limited attention has been paid to the required vulcanization. So far, the vulcanization time has been determined experimentally. Since the experimental determination of the necessary vulcanization time for prototype, single and spare part production is both economically and ecologically opposed to an industrial use, it is necessary that the vulcanization time is determined before the actual vulcanization. A simulation model was therefore developed that predicts the vulcanization time depending on the component and rubber compound. This method was validated on the basis of tensile tests on two components, one made from HNBR and the other from NBR. There are only small deviations between the experimentally and simulatively determined optimum vulcanization time. For example, the current simulation model did not take into account the temperature drop in the oven caused by opening the oven door to insert the samples. Likewise, only crosslinking isotherms at intervals of 10 °C were recorded. If the temperature drop is taken into account and if more crosslinking isotherms are deposited with a smaller temperature distance between them, the quality of the simulation model should increase further. Finally, potentials for technical logistics were identified, which are particularly to be found in shortened supply chains, smaller stocks and lower downtimes due to rapid production of spare parts. The presented simulation model and the previous conclusions on the additive manufacturing of rubber components now provide all necessary results for an industrial sustainable use of the AME-process for production of prototypes, single and spare parts.

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