Combustion-driven soft machines
Design, manufacturing and application

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COMBUSTION-DRIVEN SOFT MACHINES:
DESIGN, MANUFACTURING AND APPLICATION

A thesis submitted to attain the degree of

DOCTOR OF SCIENCES of ETH Zurich

(Dr. sc. ETH Zurich)

presented by

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2016
The difference in winning and losing is most often … not quitting.

_Walt Disney_

Is this science what you do?

_Dr. Martin Zeltner_
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Zusammenfassung


dann die weiche Maschine befreit. Dass solche weiche Maschinen vielseitig einsetzbar sind, wird in den nachstehenden Kapiteln gezeigt.

Daher beschreibt **Kapitel 2** eine verbrennungsgetriebene Wasserpumpe, welche mit dieser adaptierten Drucktechnologie hergestellt wurde. Das Design dieser Pumpe besteht aus einer rotationssymmetrischen Brennkammer im Innern, welche die Verbrennungenergie über ihre Wände an die umschlossende Pumpkammer abgibt. Dabei wurde der Einfluss der Verbrennung auf die Fördermenge und die Energieeffizienz analysiert. Des Weiteren wurde der Effekt einer Aussenwandverstärkung mittels eines Aramidgewebes untersucht. Es wurde gezeigt, dass mit dieser weichen Maschine Pumphöhen von 13 Meter (entsprechend 1.3 bar) möglich sind, obwohl diese nur eine Effizienz von weniger als 0.1 % aufweist.


Summary

The development and the manufacturing of machines, which are designed to simplify our daily life, has always been a key driver for human kind. Starting from very primitive tools made of wood or stone, we now produce goods from metal and polymers using computer assisted precision machines. The resulting complexity increase of modern manufacturing is reflected by the number of necessary machine parts. Nevertheless, nature shows that machines can also be built in a different way.

In Chapter 1 this difference in manufacturing is discussed for traditional machines and machines designed by nature. This comparison then reveals what these two routes of manufacturing have in common. Thereby the principle of backlash is illustrated for both routes through the example of two spur gear wheels and a human muscle. As a result of this illustration, muscles can act on a much smaller scale than traditionally manufactured machines. Muscle cells not only interact with each other but they can even perform in a deformed state. This is a big difference to traditional machines because the built-in materials generally do not allow such a deformation (i.e. rigid metal parts). These machine parts lack the possibility of having more than one function (i.e. only structural integrity or energy transmission). Good examples for part materials that can fulfill more than one function are given by soft machines. In particular, the example of a soft machine imitating a human hand is illustrated. This machine consists of several silicone elastomer chambers, which together can perform the complex task of grasping many different objects. Thereby no extensive control is necessary because inflating soft chamber structures passively adapt themselves to the object of interest. Thus, the rubber material is not only responsible for the structural integrity of the machine but also has to perform the grasping task through its deformation. These soft machines therefore consist of fewer parts than traditionally manufactured machines. Synthetic rubbers offer a variety of very interesting properties. Material properties, especially from silicone based materials, are therefore presented. Machines produced from silicone elastomer can even be driven by the internal combustion of hydrocarbons. This requires a manufacturing technique which can produce soft machines without any bonding surfaces (especially in the regions where the material gets deformed). By using lost-wax casting principles, a mold can be produced using computer assisted design software and modern 3D printing. The mold is then filled with silicone prepolymer. After curing, the mold material is
dissolved in solvent in order to free the soft machine. The following chapters demonstrate the wide application range of combustion-driven soft machines.

This is why **Chapter 2** illustrates the case of a combustion-driven water pump which was produced from the novel injection technology. The pump design consisted of a rotationally symmetric combustion chamber which can transmit the combustion energy through wall deformation. Through the material deformation, the fluid in the outer pump chamber is squeezed out. Mainly combustion parameters were investigated in order to understand their effect on the overall pump rate and the corresponding energy efficiency. Further, the effect of outer wall reinforcement was investigated by aramid fabric incorporation. The resulting combustion-driven soft pump was able to pump up to an altitude of 13 meters (i.e. corresponding to a pressure of 1.3 bar), even though the resulting energy efficiency was in the range of 0.1 %.

**In Chapter 3** the same manufacturing technology was used in order to produce a combustion-driven soft robot. This robot was not connected to an external energy source and moved by combustion induced jumps. The explosion was triggered by igniting a mixture of laughing gas and lighter gas. Gases were therefore injected into the combustion chamber from two on board storage tanks. A spark gap transformer then ignited the mixture after manually pushing the button on a remote controller (RC). Also the necessary battery pack was part of the on-board robot equipment. After a jump, the roly-poly design automatically put the robot back into an upright position for the next jump. Investigations were performed on the jumping behavior of the robot and on the resulting material stress.

**Chapter 4** describes the manufacturing and analysis of a fluid soft pump. The pump design is inspired by mammalian veins. In other words, this soft pump not only consists of soft actuation segments but also of entirely soft and vein-like valves. These valves prevent the fluid from flowing backwards and therefore direct the flow. This pump consisted of four actuation segments and five valve segments. In order to produce this soft machine as a single soft part, 3D printed molds were stacked and glued together. Then the resulting mold was filled with pre-polymer. After crosslinking the silicone rubber, the mold was dissolved in solvent. The use of soft silicone elastomer also allowed pump examination using standard medical analysis techniques. Ultrasonography could be used in order to live track valve motion. Color Doppler imaging further allowed the illustration of predominant flow fields. In order to improve image contrast, small
amount of micro glass beads were incorporated. This increased the actual backscattering area and allowed a simpler differentiation between soft machine wall and pump fluid. For a precise recording, the soft pump was driven by pneumatic expansion even though combustion expansion mode was available as well. The pump was further tested for its parameters. The impact of the actuation sequence (i.e. pump segments were actuated in different sequences) was investigated on the pump rate. Also operation at a bending angle of 26° was possible without significant pump rate loss. Last, the pump was frozen while still containing the pump fluid. After thawing, the pump was able to operate without any visible damage.

**Chapter 5** summarizes the findings on how soft machines can help to reduce the actual part number. The advantage in terms of manufacturing tolerances only represents one part of the drawn conclusions in this work. Overall, the results presented in this thesis show that combustion-driven soft machine principles represent an alternative thinking compared to traditional machine design and manufacturing. In consequence, this emerging field can become a part in classical mechanics in the near future.
1. Towards manufacturing of combustion-driven soft machines
1.1 Machine manufacturing

Already before 4000 B.C., humans have processed materials such as gold, meteoric iron or copper. They shaped them through hammering with basic tools of stone, wood or bone. Since then, humans have improved their skills in processing earths and metal ores. The words production and manufacturing started to characterize this material transformation around the 15th century. Nowadays, manufacturing is defined as the task of transforming materials and information into goods, which satisfy the human needs. Economically, manufacturing creates many jobs and is therefore jointly responsible for the wealth-generation in almost any country in the world. In numbers, the manufacturing industries account for 26.3% of the gross domestic product (GDP) in Switzerland in the year 2014. Other countries, such as the United States (20.5% in the year 2013) or China (42.7% in the year 2014), also owe part of their wealth to the manufacturing industry.

One way to understand the origin of this wealth creation is through product design. The actual number of parts in a product increases with its complexity. A simple example to understand this increase can be achieved by comparing two systems, for example a lawn mower and an aircraft. A lawn mower’s task is to cut the grass to a desired height, whereas a plane’s task is to safely transport up to a few hundred of people from point A to point B. This is also reflected by the effective part number of a lawn mower and a plane. In fact, a vast majority of all manufactured products consist of more than one part, as Table 1.1 suggests.

Table 1.1. Approximate number of parts in products (adapted from Kalpakjian (2010)).

<table>
<thead>
<tr>
<th>Product</th>
<th>Parts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Common pencil</td>
<td>4</td>
</tr>
<tr>
<td>Rotary lawn mower</td>
<td>300</td>
</tr>
<tr>
<td>Grand piano</td>
<td>12’000</td>
</tr>
<tr>
<td>Automobile</td>
<td>15’000</td>
</tr>
<tr>
<td>Boeing 747-400</td>
<td>6’000’000</td>
</tr>
</tbody>
</table>

In other words, product design divides a complex system into subsystems (and even sub-subsystems) in order to handle system complexity. To each subsystem, a distinct boundary
and exchanging flows (i.e. energy, material information) are assigned. With this knowledge, they are then transformed into simple mechanical assemblies. These assemblies consist of components which help to fulfill the designated task. From this point, design becomes manufacturing, as Scheme 1.1 also illustrates.\(^8\)

\[\text{Scheme 1.1. Stages of a product life, adapted from D. G. Ullman.}^8\]

In this step of a product life, components are specified and manufactured. In other words, its material (i.e. gray cast iron for the lawn mowers cylinder head) and its manufacturing (i.e. sand casting) are chosen.\(^9\) This entire process requires a broad material and manufacturing knowledge in order to produce parts that actually can perform according to their design. Summarizing, design and manufacturing involve a lot of labor and therefore explain why their share in the GDP is considerable.

**1.1.1 Traditional manufacturing**

As stated above, the main objective in the design of a complex product is the reduction of its complexity into manufacturable parts. Good examples for this reduction process are machines. In the following Scheme 1.2, the reduction is shown for the complex task of transforming fuel into motion.
**Scheme 1.2.** The complex task of transforming fuel into torque is illustrated at the example of a combustion engine. The scheme highlights the reduction in complexity from the left to the right. At the most right, the force transmission is illustrated by the interaction of two gear wheels. The red arrow indicates the necessary backlash such that this force transmission between the teeth can occur without the risk of jamming.

Combustion engines such as four-stroke engines are a good example for a successful fulfilment of this task.\(^\text{10}\) The combustible mixture of fuel and air is injected into the cylinder. Then, the piston compresses the mixture. Upon an electrical ignition, the combustibles explode, expand and force the piston to move. The resulting movement is transmitted via the connecting rod to the crankshaft and thereby accelerates it. The rotating crank itself is connected to gear wheels, which transmit the rotation to other wheels or v-belts. Thus, these parts (i.e. piston, rod etc.) help to transform fuel into torque.

The fact that parts can transmit torque without a fixed connection is still astonishing. Theoretically, two gear wheels (also called a gear pair) can be designed perfectly without the risk of losing the transmission performance. However, reality shows that backlash is inevitable for such a gear pair. Therefore, this free space is tolerated between two parts to avoid the risk of jamming.\(^\text{11,12}\) Again, considering **Scheme 1.2,** this can be observed for the interaction of a gear pair. The teeth of the two wheels interlock and thereby transmit torque. The need of backlash results from the fact that gear wheels can only be produced within certain dimensional limits. If too much or too little backlash is allowed, the transmission would result in mechanical wear (i.e. material abrasion) or even mechanical deformation. Due to machine and assembling error, a gear pair cannot be produced without accounting for the difference in production. This difference between the maximum and the minimum limit, where a specific dimension is allowed to vary, is defined as tolerance.\(^\text{13}\) There are many institutes which define tolerances for standard parts (e.g. International Organization for Standardization (ISO), German Institute for Standardization (DIN)
or American National Standard Institute (ANSI). By considering again the example of the gear pair, there are also standards defining their center distance. Assuming that the gear pair consists of two spur gears, some of the following tolerances are given according to DIN 3964 (see Table 1.2).

Table 1.2. Part of defined tolerance values according to DIN 3964, listed in tolerance fields for spur gears. $A_{ae}$ and $A_{ai}$ represent upper (+) and lower (−) tolerance limits.

<table>
<thead>
<tr>
<th>Center distance in mm</th>
<th>$A_{ae}$ and $A_{ai}$ in $\mu$m</th>
<th>ISO tolerance field</th>
</tr>
</thead>
<tbody>
<tr>
<td>over 30 until 50</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>+5.5</td>
<td>+8</td>
</tr>
<tr>
<td>6</td>
<td>+8</td>
<td>-5.5</td>
</tr>
<tr>
<td>7</td>
<td>+12.5</td>
<td>-8</td>
</tr>
<tr>
<td>8</td>
<td>+19.5</td>
<td>-12.5</td>
</tr>
<tr>
<td>9</td>
<td>+31</td>
<td>-19.5</td>
</tr>
<tr>
<td>10</td>
<td>+50</td>
<td>-31</td>
</tr>
<tr>
<td>11</td>
<td>+80</td>
<td>-50</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Center distance in mm</th>
<th>$A_{ae}$ and $A_{ai}$ in $\mu$m</th>
<th>ISO tolerance field</th>
</tr>
</thead>
<tbody>
<tr>
<td>over 50 until 80</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>+6.5</td>
<td>+9.5</td>
</tr>
<tr>
<td>6</td>
<td>+9.5</td>
<td>-6.5</td>
</tr>
<tr>
<td>7</td>
<td>+15</td>
<td>-9.5</td>
</tr>
<tr>
<td>8</td>
<td>+23</td>
<td>-15</td>
</tr>
<tr>
<td>9</td>
<td>+37</td>
<td>-23</td>
</tr>
<tr>
<td>10</td>
<td>+60</td>
<td>-37</td>
</tr>
<tr>
<td>11</td>
<td>+95</td>
<td>-60</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Center distance in mm</th>
<th>$A_{ae}$ and $A_{ai}$ in $\mu$m</th>
<th>ISO tolerance field</th>
</tr>
</thead>
<tbody>
<tr>
<td>over 80 until 120</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>+7.5</td>
<td>+11</td>
</tr>
<tr>
<td>6</td>
<td>+11</td>
<td>-7.5</td>
</tr>
<tr>
<td>7</td>
<td>+17.5</td>
<td>-11</td>
</tr>
<tr>
<td>8</td>
<td>+27</td>
<td>-17.5</td>
</tr>
<tr>
<td>9</td>
<td>+43.5</td>
<td>-27</td>
</tr>
<tr>
<td>10</td>
<td>+70</td>
<td>-43.5</td>
</tr>
<tr>
<td>11</td>
<td>+110</td>
<td>-70</td>
</tr>
</tbody>
</table>

The values are listed according to their tolerance field. This tolerance field is determined by the machine design and can have a value of, for example, $js7$. Further assuming a center distance of 70 mm, the tolerable distance lies between 69,085 mm and 70,015 mm. The resulting backlash between a gear pair can be estimated to be in the range of a few tenth of a micron (i.e. tolerances for the manufacturing of a spur gear wheel, which are defined by DIN 3961 and DIN 3962, are in the range of a few micron and are therefore neglected)\textsuperscript{16,17}. Thus, a gear pair can fully operate with backlash but without the risk of jamming if the actual center distance lies within the tolerance.

1.1.2 Manufacturing challenges

Manufacturing parts according to standards has a big impact on machine development.\textsuperscript{18,19} Before standards were commonly accepted by the manufacturing industry, some rework of machine parts was necessary to assemble a machine. With the acceptance of common standards, parts are manufactured without the need of any rework during assembly. This of course reduced manufacturing time and therefore increased profitability of the manufacturing industry.\textsuperscript{20} Standards have therefore unified manufacturing and allow a more complex machine design.\textsuperscript{21} The trend of increasing technical complexity of today’s machines also has an influence
on the number of parts and the machine sophistication. As a result, they have to perform superior to the last machine generation while accounting for the latest environmental constraints (i.e. emission standards for combustion engines). This inevitably results in an increased number of necessary parts because the given constraints have to be respected. In modern engines the control of constraints is executed by a control system, which measures and adapts machine parameters (i.e. fuel to air ratio for an effective combustion). From a design perspective, the control system has to be as small as possible because a new engine still has to outperform older engine generations. Hence, one possible way to save weight and space is by reducing machine part sizes.

Part size reduction goes hand in hand with tolerance limits and device reliability. If a machine part is designed smaller, also these limits are set lower for its manufacturing. In consequence, the price per part may increase because manufacturing has to account for these limits by appropriate manufacturing machines (i.e. more precise). Together with the trend of increasing machine complexity, modern manufacturing needs to produce many small parts which would be produced at higher cost per part. Such a development can be maintained if new manufacturing machines themselves are produced at lower cost. Then, these machines can be acquired for producing more precise parts while maintaining the part’s quality and price. If one would not account for the consequences of reduced part sizes, a new device would just become too expensive. Also, device reliability depends on the number of built-in components. Each component has a certain risk to fail. The risks arise from defects in crude material, design errors, wear, manufacturing errors (i.e. hairline cracks) and so on. Because each part has a risk to fail, the probability of device failure scales with the actual part number. This is a big challenge nowadays, especially upon considering again the before mentioned necessity of more device parts. The complexity reduction in machine design and the increasing demand for high performance machines should therefore result not only in an increased number of necessary parts. Traditional design and manufacturing should be confronted and challenged with this development. For the example of combustion engines, traditional torque transmission principles have to be questioned in order to find new ways to transform energy into motion.
1.1.3 Nature’s manufacturing

Nature has found a way to efficiently transform a chemical energy source into motion. Animals and humans conquer the world through many forms of locomotion (i.e. running, swimming or flying). Their main actuators, which are responsible for the fulfillment of this complex task, are muscles. Like a combustion engine, they can transform their energy source into muscle contraction. Scheme 1.3 illustrates the complexity reduction of a human muscle into its essential parts.

Scheme 1.3. The complex task of transforming an energy source into contraction is illustrated with the example of a human muscle. The scheme highlights the reduction in complexity from the left to the right. At the most right, the force transmission is illustrated by the interaction of actin and myosin, the two responsible proteins for muscle contraction. The red arrow indicates the necessary tolerance such that the proteins can interact with each other.

Unlike with classical combustion engines, the transformation of energy into motion (in this case, the muscle contraction) is not executed in a few cylinders. The contraction itself happens within a myofibril, a rod-like unit of a muscle cell at the expense of adenosine triphosphate (ATP). Such a rod is divided into sarcomeres, consisting of the structural protein actin, and the motor protein myosin. Assuming an average number of 280'000 muscle fibers (i.e. for biceps brachii) and an average number of sarcomere per fiber of 68'000 (i.e. for an average fiber length of 170 mm), an approximate number of 19 billion sarcomeres can be estimated. Hence, nature applies its energy transforming units (i.e. the sarcomeres, consisting of many parallel actin and myosin filaments) all over its actuation unit. In other words, the complexity reduction is not performed by
a series of consecutive parts each fulfilling a certain task (see Scheme 1.2) but rather by many small and interconnected units (see again Scheme 1.3)

Even though a muscle has a different design than a combustion engine in order to transform energy into motion, the necessity of free space (i.e. backlash) is still present. Like two gear wheels always have a certain backlash, the two proteins myosin and actin also need backlash so that the myosin head can slide over the actin structure. This backlash can be estimated by considering the head of a myosin. The head represents the minimum space which is required for this sliding. A myosin head is considered to have a size of roughly 300 to 400 Å. Comparing this to the few tenth of a micron for the gear wheels, nature is capable to produce its parts with a backlash of several orders of magnitude smaller. In other words, the length scale between a complete actuator (i.e. a muscle) and a single part (i.e. structure protein actin) is bridged with many intermediate structures, each adapted to the needs at a given length scale (see again Figure 1.3).

1.2 Soft machine principles

As recognized above, nature builds very small energy transforming units having an even smaller backlash. Due to this precise manufacturing, the transition between two unit materials (i.e. proteins) and their function (i.e. contracting upon an electrical signal) becomes somewhat vague to the human eye. As a result, sarcomeres seem to work as numerous interconnected soft parts within skeletal muscle cells. The advantage of having a vast number of energy transforming units is that not all have to contract simultaneously and therefore can adapt to the actual work load. Furthermore, this selective calling for units to contract allows the muscle to operate also in deformed state. This stays in big contrast to deformations in traditional machines. If the crankshaft from a combustion engine is only slightly deformed, the entire machine can jam. Therefore, traditional machines clearly lack a certain flexibility in order to adapt themselves to a continuously changing environment.

Soft machines offer the possibility to avoid these numerous small interconnected machine parts, manufactured at very high precision. These machines are built from very few, mostly elastomeric parts. Soft parts not only contribute to the structural integrity but also help to transform energy into motion. Known energy sources are electrical charge, pneumatic
expansion,\textsuperscript{36-42} gas combustion,\textsuperscript{43,44} or magnetic fields.\textsuperscript{45-47} Depending on the source, energy transformation can occur through direct elastomer deformation of an incorporated shape memory alloy (SMA) by applying an electrical charge. Also, incorporated magnetic particles can, upon applying a magnetic field, cause the elastomer to actuate. Nevertheless, the most used concept of these machines is based on fluid expansion. Due to the expanding fluid, the pressure inside a certain part of the soft machine is elevated. The elastomeric nature of the surrounding walls results in their deformation. This is illustrated in the following Scheme 1.4:

\textbf{Scheme 1.4.} (a) Wall deformation inside a soft chamber is shown due to fluid expansion. The black connector represents the inlet for the expansion. (b) Depending on the wall rigidity, the deformation is more or less severe. (c) Selective fabric layer incorporation into a wall prevents its elongation. (d) The expansion is then used to push the machine from the ground or (e) to squeeze fluid in adjacent chambers.
A good example illustrating the benefits of elastomeric parts was given by Raphael Deimel and Oliver Brock.\textsuperscript{48} They created a soft machine inspired by a human hand. The machine was capable of dexterous grasping (i.e. high variability in grasping) and was able to fulfil 31 of 33 grasps according to the Feix taxonomy.\textsuperscript{49} The hand was an assembly of several soft actuators, representing each a distinct part of a human hand (i.e. fingers, thumb or palm). Each actuator was made from an elastomeric chamber with a partially incorporated fabric layer to selectively prohibit elongation. Radial fibers around each soft part further stabilized the actuator. Upon inflating the actuator chamber with air, the expanding chamber started bending. The radial fibers further increased the attainable curvature resulting in grasping. The advantage of soft machines becomes visible upon considering Figure 1.1. This figure captures the different grasps of the soft hand. The only control necessary in order to work with the soft actuator was the air pressure in each chamber. Depending on the pressure, the bending of a finger or the thumb could be adjusted. More importantly, the soft hand did not have to rely on any force sensors in the finger tips in order to adjust its grasping. This task was fulfilled by the elastomeric material in a passive manner. Hence, this soft machine not only has fewer parts than an ordinary robotic hand but also can avoid additional sensor technology due to a passive adaption of the elastomer to its surrounding.\textsuperscript{50}
Figure 1.1. Soft robotic hand produced by Deimel et al.\textsuperscript{48} The small figures show enacted grasps of the Feix taxonomy, using empirically determined actuation patterns.\textsuperscript{49} Grasps are numbered according to the Feix taxonomy; the hand failed to replicate grasps 5 (Light Tool) and 19 (Distal Type, Scissors). This picture was printed with permission of the SAGE Publications.

The adaption of the elastomeric material to its surrounding is only one benefit resulting from soft machine manufacturing. By incorporating soft materials into the design, an entire new dimension is added to machine manufacturing.\textsuperscript{51} This dimension results in other benefits, as described by Iida and Laschi:\textsuperscript{52}

- Machine weight can be reduced
- Motion is controlled directly by the elastomer
- Mechanical self-stabilization due to the elastomer, reducing control architecture of such machines
Nevertheless, designing a machine almost entirely from soft materials changes the applicable mechanics and therefore increases the actuation complexity of such a machine. This is why the next paragraphs shall tackle a possible elastomer choice in order to understand system dynamics (described in chapter 1.3) and shall illustrate potential manufacturing techniques to actually fabricate soft machines (described in chapter 1.4).

1.3 Elastomers for soft machine manufacturing

Elastomers, also called rubbers, are materials consisting of slightly cross-linked polymer chains which preserve their elastic nature between very low temperatures (i.e. below 0°C) up to their decomposition temperature.\textsuperscript{53,54} They can undergo large and reversible deformation of up to 1000\% and a Young’s modulus of up to 2 MPa.\textsuperscript{55} Elastomers are considered to have viscoelastic properties, resulting from the fact that part of the work carried out by the elastic deformation is released as heat. Therefore, material properties depend on the actual deformation speed and the prevailing temperature. The materials can be split into two main categories, namely natural rubber produced from latex emulsion and synthetic rubber mainly produced from hydrocarbon feedstock. Scheme 1.5 illustrates the classification into their symbol groups. Prominent elastomers were highlighted from each symbol group (according to ISO 1629).\textsuperscript{56-58}
Scheme 1.5. Overview on prominent elastomers, grouped according to ISO 1629.\textsuperscript{56-58} Elastomers used in large quantity are highlighted in blue, whereas elastomers mainly used for manufacturing of soft machines are highlighted in green.
This standard divides synthetic elastomers into rubbers,

(i) having saturated chains of the polymethylene type ("M" group)
(ii) having carbon and oxygen in the polymer chain ("O" group)
(iii) having the name of the substituent group on the polymer chain prior to silicone designation ("Q" group)
(iv) having the name of the monomer(s) from which the rubber was made from in front of the letter “R”. This letter signifies the conjugated diene form of the polymer chain ("R" group)
(v) having carbon, oxygen and sulfur in the polymer chain ("T" group)
(vi) having carbon, oxygen and nitrogen in the polymer chain ("U" group), and
(vii) having phosphorous and nitrogen in the polymer chain ("Z" group)

A very interesting group of elastomers for soft machine manufacturing are polysiloxanes. These polymers consist of a Si-O backbone endowing it with a variety of fascinating properties.\(^{59}\) They are temperature stable and therefore suitable for high temperature applications such as combustion-driven soft machines.\(^{60,61}\) Depending on used side chains and due to the nature of the bonding, polysiloxanes have a low surface free energy, resulting in desirable surface properties for coatings and biomedical materials. The Si-O bond length (1.64 Å) is significantly longer than the C-C bond length (1.53 Å). Also, the O-Si-O angle is much larger (~150°) than the usual tetrahedral value (~ 110°) and can even readily pass through the liner 180° state.\(^{62,63}\) The result is a very flexible polymer chain, which is also reflected by high elongation at break (i.e. over 1000 %).\(^{64}\) These properties of silicone elastomers help soft machines to perform well with their cyclic expanding behavior.

As already mentioned above, elastomers not only need good properties for soft machine manufacturing but must also be workable in order to manufacture these machines. Therefore, the synthesis of these polymers is of great importance. Siloxane polymer is generally produced by reacting organodichlorosilanes with water.\(^{65,66}\) The obtained mixture is further converted into cyclic or linear polysiloxanes. These feedstocks then can undergo anionic, cationic or ring-opening polymerization in order to form a pre-polymer of higher degree of polymerization. The polymerization degree is controlled by the concentration of chain stoppers. These units terminate the polymerization process with typical end groups (i.e. methyl, hydroxyl, vinyl or hydrogen).
Then, silicone pre-polymer needs to be cross-linked in order to give the desired rubbery material. This can be done by radical curing involving peroxides\textsuperscript{67,68}, by hydrosilylation curing of Si-H groups to C=\!C double bonds\textsuperscript{69}, condensation curing of a Si-OH with Si-OC(O)CH\textsubscript{3}\textsuperscript{70,71}, by radiation curing using ultraviolet (UV) light\textsuperscript{72} or by oxidative coupling of thiol end groups in the presence of metal catalysts to form disulfide bridges\textsuperscript{73}. The crosslinking, also called vulcanization, can be performed at different temperatures of up to 230°C. This is why silicones can be categorized into high temperature vulcanizing (HTV) and room temperature vulcanizing (RTV) rubbers. Further, these rubbers can be differentiated according to their processing into one- or two component systems. One component systems are almost exclusively based on a condensation mechanism, curing upon exposure to atmospheric moisture. Two component systems are mainly implemented for injection molding and can use almost any of the before mentioned curing systems. Summarizing, silicone rubbers have a broad property range, can be processed in many different ways and are therefore suitable for soft machine manufacturing.

1.4 Lost-wax casting of soft machines

Lost-wax casting, also called investment casting, is a manufacturing process first used in the Bronze Age in order to manufacture tools or weapons. The following section describes this process at the example of an arrow tip, as shown in Scheme 1.6. First, a wax pattern of an arrow tip is formed (also plastics, such as polystyrene, can be used). The tip is then dipped into a slurry of refractory material. Nowadays, this material consists of silica and a fine binder. In order to increase the mold thickness, the dipping is repeated several times. After drying at the air, the mold is heated to a temperature of 90°C to 175°C. Due to this heat, the wax can be melted out. Then, the mold is fired to 650°C to 1050°C in order to burn off residual wax and to drive off residual water. Following, liquid metal is poured into the mold. After the metal has solidified again, the mold is destroyed and the casting is removed.
Scheme 1.6. Lost-wax casting process as used in the Bronze Age to produce an arrow tip. First, a wax pattern of a tip is formed. Then, the pattern dipped into a slurry of refractory material. After the coating has dried, the wax pattern is removed by burning it off. The mold is then filled by pouring in molten metal. After cooling and metal solidification, the mold is destroyed thereby freeing the metal arrow tip.

Lost-wax casting is one possibility to build single parts without any connecting joints. The process is capable of producing parts with a good surface finish and close dimensional tolerances. This is why we adapted this process for elastomer casting in order to build single part soft machines which can be driven by combustion. For illustration purposes, Scheme 1.7 highlights this adapted manufacturing process using the example of an arrow tip made from silicone rubber. Instead of forming a wax pattern of the arrow tip, the tip is designed by computer-aided design (CAD) software. The design tip is then inverted in order to form a virtual mold. The data is converted such that the mold can be printed using 3D printing. There, the mold is built up layer by layer. After removing any residual support structures, the mold can be filled with two component silicone pre-polymer. In other words, the silicone rubber is injected into the mold and cured at room or elevated temperature. After curing, the mold material is removed by dissolving the thermoplastic mold material in a solvent bath. This then frees the silicone arrow tip.
Scheme 1.7. Virtual lost-wax casting process in order to produce a soft arrow tip. The arrow tip is designed by using computer-aided design (CAD) software. The mold is then formed by virtually inverting the tip design. The resulting virtual mold is then manufactured by 3D printing. Uncured elastomer is then poured into the mold. After the elastomer cured, the mold is removed by dissolving it in solvent. This then frees the soft arrow tip.

1.5 Conclusion

In this thesis, the emphasis is on the impact of combustion-driven soft actuators produced by the lost-wax casting process. As mentioned before, nature designs its machines in such a way that the transition between materials and their function is interconnected. This interconnection takes place at a very small scale level and is barely reproducible with traditional manufacturing techniques. Manufacturing machines with the developed casting technology exploit the elastic behavior of rubbers to mimic the interconnection of machine parts and their functions. The strength of this technology is shown with the example of transforming fuel into locomotion. Unlike traditional combustion engines, combustion-driven soft machines show a way to fulfill this difficult task without the necessity of numerous machine parts. They thereby challenge the ongoing miniaturization trend of numerous machine parts by offering an original way to manufacture fewer but multi-functional parts.

The benefits of this technology are demonstrated for three different soft machines. The first soft machine analyses combustion behavior and efficiency for the task of conveying liquid. Further, the impact of an outer fabric layer on the combustion efficiency and the thermal stress on the silicone rubber are investigated. The second soft machine analyses the use of this casting principle in order to produce an untethered jumping robot. This robot revealed further investigations on fuel selection for a long term powering of such machines. Lastly, a liquid
conveying soft pump inspired by mammalian veins demonstrates the design possibilities with this casting technology. This machine also shows the possibility to visualize valve contractions in real time with state-of-the-art diagnostic devices used in medical examinations (e.g. sonography).
2. Design, Performance and Reinforcement of Bearing-Free Soft Silicone Combustion-Driven Pumps

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Author contribution:
M.L., C.M.S. and W.J.S. elaborated the concept. M.L. and C.M.S. designed the machine and the experiments. M.L. and C.M.S. performed the experiments. M.L., C.M.S. and W.J.S. wrote the manuscript with contributions and review from all authors.
2.1 Introduction

Soft machines have received considerable attention to face today’s engineering challenges in system manipulation and locomotion. Unlike classical “hard” machines working within dimensionally tight and preset boundaries, they are capable of fast adaption to the operation environment. These machines do not depend on fully functional bearings and transmission axes to transform energy carriers such as hydrocarbons into directed mechanical work. Furthermore, soft machines show low impact sensitivity and cope well with high degrees of mechanical deformation while maintaining full operability. Soft machines are often manufactured on the base of elastomeric structures which can be actuated by electrical charge, pneumatic expansion, gas combustion, or magnetic fields. Although built as very simple structures (i.e., no need of a lubricant system compared to hard machines with similar capabilities) soft machines performing rather complex movements have been shown in several studies. Lin et al. presented a soft caterpillar capable of rolling locomotion by stimulating incorporated shape memory alloys. Shepherd et al. designed a soft robot capable of sophisticated locomotion by specific pressure regulation inside various soft-silicone body segments. Key advantages such as high robustness and an almost wear-resistant actuation (compared to traditional mechanic transmission) are believed to fill a gap in future engineering approaches. Additionally, low-cost producibility and lightweight manufacturing are further reasons why soft machines have gained general attention. As already pointed out by Whitesides et al., combustion driven soft systems bring in a substantial enhancement in power characteristics compared to others. This has effectively been demonstrated by the ignition of an oxygen–methane mixture inside a tripodal soft robot created by soft lithography. The overall energy efficiency of this machine was calculated to be around 0.7% and it was able to jump over 30 times to a height of 30 cm (corresponding to more than 30 times its own height). The benefits of soft machines can also be transferred to standard equipment, such as pumps. Fuhrer et al. presented a peristaltic pump operated by repetitive application of a magnetic field to magnetic elastomer tubing. This pump is now being investigated for prospective blood pumping in human beings. However, less attention has been paid to system applicability and stability. These are the most critical parameters for future application. Earlier this year, we presented a silicone monoblock soft pump using lost-wax casting technology. This soft machine shows pumping capability at increased robustness and high combustion power loadings. The fluid pumping device designed by Schumacher et al. was able to
pump for over 10 000 combustion cycles without any structural rupture but failed yet to pump under application relevant conditions (i.e., no fluid conveyance to reasonable heights).\textsuperscript{43}

In the here presented work, we provide a design investigation for highly durable soft pumps, which are reliably cooled by the conveyed liquid and that are capable of pumping water at flow rates up to 240 mL/min or to considerable heights (up to 13 meters). These pumps were able to withstand over 30 000 combustion cycles. Using already established lost-wax casting technology, we demonstrate simple fabrication of durable soft silicone pumps. Incorporation of fabrics into soft structures has already been shown to improve soft machine robustness.\textsuperscript{76} Here, control and yield of combustion power inside the system is increased by incorporating aramid fabric layers into the outer pump shell. To better understand combustion force transmittance, pump designs are slightly varied. The resulting impact of the design changes is then measured as a function of the overall pump mass flow rate. Our study also shows investigations on combustible gas mixtures, their flow rates, and actuation frequencies. The resulting best design demonstrated reliable pumping up to heights of 13 meters.

2.2 Experimental Section

2.2.1 Soft pump design and manufacturing

Soft pumps were manufactured by producing lost-wax casted soft silicone monoblocks as shown earlier.\textsuperscript{43} In detail, we designed rotationally symmetric pumps consisting of an inner combustion chamber (volume of 94 cm\textsuperscript{3}) and an outer liquid pump chamber (volume of 220 cm\textsuperscript{3}). A schematic overview of these pump designs is shown in Figure 2.1a.
Figure 2.1. (a) Schematic overview of a fabric reinforced soft pump. The three layered outer shell consists of an inner and outer silicone layer with an intermediate aramid fabric layer. (b) A schematic cut-through of the pump indicated with all necessary pump parts such as spark igniter, and liquid and gas check valves. The asymmetric chamber design is illustrated by the displaced center of gravity (COG) of the inner combustion chamber. (c) Photograph of the installed soft pump with gas and electricity connection at the bottom, as well as the additionally inserted metal exhaust pipe at the top.

Upon combustion inside the inner chamber, the resulting gas expansion stretches the soft actuation barrier (thickness of 3 mm) within the two chambers. The resulting compression wave displaces the liquid inside the pumping chamber. By the use of check valves, the resulting flow is guided unidirectionally which enables clocked pumping. The alignment of all necessary components is schematically illustrated in Figure 2.1b. To design a pump, computer aided design
software (CAD) was used to create a three-dimensional injection mold. This virtual mold was then printed using commercially available 3D printers (HP Designjet 3D, Hewlett-Packard or uPrint SE Plus, Stratasys, both United States). We used a layer resolution of 0.22 mm, smart layer arrangement and low density printing options. The resulting 3D models consisted of acrylonitrile–butadiene–styrene (ABS) model material and polylactic acid (PLA) support structures.

After dissolving all PLA support structures inside the ABS mold in an alkaline bath, we filled all voids with a mixture of room temperature vulcanizing silicone (RTV 1701, 95 wt % monomer, 5 wt % cross-linking agent, both Neukasil, Altropol, Germany). This was performed by pressing the silicone into the molds with an in-house made press. When the silicone mixture was completely cured at room temperature, the filled ABS mold was dissolved in an acetone bath until no plastic material was left. The resulting soft pump was then air-cleaned to remove residual acetone prior to further design steps. As previously suggested, the outer layer can be reinforced to increase energy efficiency. Therefore, we wrapped fabrics made from polyaramid (Fibermax composites, United Kingdom) around the soft pump to increase expansion stiffness. In detail, two fabric layers were produced by wrapping two pre-cut fabric pieces around the pump for each layer. Superglue (Superflex, UHU, Germany) was used for fixation. Then, a second layer of silicone was applied by adding the same monomer cross-linking mixture into a second ABS shell (assembled from two identical halves) enclosing the soft pump. The photo line of the most important manufacturing substeps is shown in Figure 2.2.
Figure 2.2. Step-by-step presentation of the soft pump design with (a) CAD design, (b) printed ABS mold, (c) silicone pump, (d) fabric layer reinforcement without second silicone layer and (e) with the second silicone layer, as well as (f) the finally installed soft pump.

After curing, the pump was again washed in acetone and air-cleaned. Overall, such a soft pump was produced in less than 10 days, also shown in Table 2.1. The corresponding dimensions of all produced soft pumps are summarized in Table 2.2.

Table 2.1. Overview of the time demand of the different manufacturing steps.

<table>
<thead>
<tr>
<th>Step</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D printing lost-wax mold</td>
<td>30 h</td>
</tr>
<tr>
<td>Alkaline bath removing support PLA</td>
<td>100 h</td>
</tr>
<tr>
<td>Silicon filling &amp; curing</td>
<td>12 h</td>
</tr>
<tr>
<td>Acetone bath removing ABS model structure</td>
<td>48 h</td>
</tr>
<tr>
<td>Second ABS shell printing</td>
<td>12 h</td>
</tr>
<tr>
<td>Fabric layer reinforcement, filling &amp; curing</td>
<td>14 h</td>
</tr>
<tr>
<td>Acetone bath removing ABS model structure</td>
<td>12 h</td>
</tr>
<tr>
<td>Total production time</td>
<td>228 h</td>
</tr>
</tbody>
</table>
Table 2.2. Overview of all produced soft pumps including their dimensions. 

<table>
<thead>
<tr>
<th>Design no.</th>
<th>Fabric layer</th>
<th>Outer wall thickness, inner layer (mm)</th>
<th>Total outer wall thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>no</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>no</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>3</td>
<td>yes</td>
<td>6</td>
<td>14</td>
</tr>
<tr>
<td>4</td>
<td>yes</td>
<td>15</td>
<td>24</td>
</tr>
</tbody>
</table>

*The first dimension denotes the wall thickness resulting from the first molding process. For fabric reinforced soft pumps, the total outer wall thickness is increased due to an additional silicon layer surrounding the fabric.*

2.2.2 Pump installation

To power the soft pump, additional components were installed. On the liquid side, polyvinyl chloride (PVC) tubing was connected to the pumping chamber inlets. Inside PVC tubing, check valves (spring pressure 0.17 psi, SmartProducts, United States) were placed as close as possible to the pump chamber inlets. On the combustion side, in-house-made gas injection nozzles and spark gap igniters were connected to the combustion chamber inlet. A combustible environment was established by flushing an air–methane mixture through the chamber. Mass flow controllers (MFC) connected to a programmable logic controller (PLC) regulated the flow rates and the ratio of combustibles to oxidants. Overall volumetric gas flow rates ranged from 1 to 12 L/min and volumetric air–methane ratios ranged from 9:1 to 14:1. The PLC also triggered the ignition spark of the spark gap igniter at frequencies ranging from 0.25 to 1 Hz. As an exhaust, simple stainless steel tubing with an inner diameter of 1.5 mm was used to create physical resistance to the outgoing flow. This is also shown in Figure 2.1c.

2.2.3 Design and operation parameter investigation

Various parameters were investigated in order to find an optimized pump design. In a first step, the effect of the outer wall stiffness on the overall mass pump rate was studied. Therefore, different grades of stiffness (regarding outer wall expansion) were created by manufacturing four pumps with two different outer wall thicknesses (see again Table 2.2). Two pumps of each thickness were then reinforced using the above-mentioned fabric layer. Pumping rates were
investigated by gravimetrically determining the conveyed liquid mass over a time period of two minutes at pumping height levels between 0.1 and 9 m. The operation settings of all pumps were adjusted to an ignition frequency of 0.5 Hz to ensure sufficient flushing time at a corresponding air–methane ratio of 10:1 (Air 3 L/min, methane 0.3 L/min). As a next step, the impact of the ignition frequency at different methane flow rates was investigated. Therefore, the pumping height was kept constant at two meters while each of the before mentioned parameters were screened one by one. During investigation of the methane flow dependency, ignition frequencies were set to 0.25, 0.5, and 1 Hz. Again, the resulting mass pump flow rate was gravimetrically determined over a time period of two minutes (one minute for 1 Hz setting). At last, air–methane ratios were varied. Therefore, the volume flow of methane was kept constant at 0.3 L/min while the air volume flow was varied within 2.7 to 4.2 L/min (corresponding to ratios of 9:1 to 14:1). The ignition frequency was kept at 0.5 Hz. The mass flow rate of the conveyed liquid was again gravimetrically determined.

2.2.4 Exhaust gas tracking

To determine the degree of spilled combustibles by constant fuel gas flushing through the combustion chamber, exhaust gases were analyzed using online mass spectroscopy (MS) (Pfeiffer Vacuum, Germany). In detail, the metallic exhaust tubing was connected to a larger silicone tube to reduce the gas flow velocity and to smooth pressure shock waves resulting from the combustion. Ion counts were recorded for methane, nitrogen, water, carbon dioxide, and oxygen during all experiments. Since the device was only able to sample the selected gases every 0.5 s, two experimental paces were chosen. First, the ignition frequency was set to 0.5 Hz at an air–methane ratio of 10:1 (air 3 L/min, methane 0.3 L/min). Then, the frequency was changed to 0.05 Hz with adapted flow rates of 0.5 L/min for air and 0.05 L/min for methane. The PLC was set to run a three-step ignition procedure for each experiment. At the beginning, air flow was switched on until all ion counts stabilized. Then, methane flow was added until all counts stabilized again. Last, the ignition was triggered. These obtained base levels were used as reference points for further discussion.

2.2.5 Long-term stability analysis

To persist in most applications, continuous operation over many cycles is one of the key demands. Therefore, we tested the thin walled soft pump for a period over 30 000 combustion
cycles. This design showed the highest overall deformation upon combustion and represents the most fragile of the tested pumps. Therefore, it represents a good long-term stability benchmark. Water was pumped in a closed loop starting from a common water tank. To ensure a nearly constant water inlet temperature and to generate additional stress by even higher temperature gradients across the combustion chamber actuation walls, ice was added to the water tank. The pump height was chosen around one meter. This represents a normal pump load situation for the design. For convenience and to create thermally even more demanding conditions, we switched the ignition frequency of the pump to 1 Hz. Thus, the methane flow was adapted to 0.6 L/min to ensure a proper combustion environment. The air–methane ratio was kept at 10:1. All ignition pulses were counted by the PLC during the experiment. In the case of an unsteady ignition behavior of the soft pump, pumping was paused to clean the primitive spark gap igniters removing any deposits with a brass brush. Then, the igniter was reinserted, and pumping was continued. After the occurrence of a total pump failure (i.e., gas bubbling on the liquid side), the pump was cut open. This procedure was documented using video recording (Legria HF G25, Canon, Japan). One half was then analyzed further by reflected-light microscopy (Axio Imager.M2m, Zeiss, Switzerland) in dark field mode.

2.2.6 Pump stress analysis

To investigate the behavior of the best design, we physically stressed the soft pump by pumping to a height of 13 meters (equals three floors in a common building). Therefore, the fabric reinforced pump was again connected to polymer tubing. Within each floor, this tubing was secured with a clamp to prevent any slipping. At the most elevated point, tubing was guided back into the water tank to close the pumping loop. For increased observability, the pump water was colored using red food dye. Then, the pump experiment started with an ignition frequency of 0.5 Hz, and an air–methane ratio of 10:1 at a methane flow rate of 0.3 L/min. The procedure was recorded by video. After the liquid reached its target level, the back flow was measured gravimetrically over a period of 10 min. To generate a second conveyance rate data point, the flow rate was also measured at a pumping frequency of 1 Hz. Thus, the methane flow rate was adapted to 0.6 L/min while the air–methane ratio was kept constant again. The resulting temperature profile along the soft pump after conveying the liquid to the highest point was captured by an infrared camera (i7, FLIR, United States).
2.3 Results and Discussion

2.3.1 Outer wall stiffness

Figure 2.3a shows the conveyance rates of the different pumps. The pump designs with fabric layers show highly increased flow rates at equal pump height compared to the designs without any fabric layer. This confirms the expected positive effect of the outer wall expansion stiffness regarding flow rates. During combustion, the intermediate actuation barrier expands toward the pumped fluid and thereby increases the fluid pressure. Owing to the undirected nature of pressure, the generated force propagates to the outer-wall layer and to the fluid check valves. Pumping force then starts stretching the outer wall. The valve will open as soon as the actual force gets higher than the spring force of the outbound check valve and the associated force applied by the hydrostatic pumping head. Hence, a stiffer outer wall allows pressure to build up faster without being damped by its elasticity. This increase forces the valve to open earlier and more fluid can be ejected. The valve then closes when spring pressure and pressure resulting from the hydrostatic head surpass the pump chamber pressure again. The consequential order of the above-mentioned process would rank the highest pump rate to the thin-walled but fabric-reinforced soft pump. In this design, the outer wall has a considerable stiffness toward expansion due to the fabric layer (see Movie A1.1 for stiffness comparison).
Figure 2.3. (a) Pump rates for all four designs are given as a function of the pump height. Dashed lines with empty symbols represent the two soft pump designs with no fabric layer, whereas the solid lines with the filled symbols denote the two reinforced designs. (b) Energy efficiency is given as the dimensionless ratio of the generated potential and the combustion energy of the soft pump. This efficiency ratio is shown again as a function of the pump height.

There is also a second effect, which explains the increased performance of the thin-walled but fabric-layered soft pump compared to the thicker fabric version. The inward directed silicon part of the outer wall causes additional damping. Hence, a thicker inner layer damps the force propagating from the combustion process. As a consequence, the second best design would be the thick reinforced pump. Since the remaining two designs are missing a fabric layer, the outer wall thickness is the main parameter influencing the pump rates. The thicker outer wall has increased expansion stiffness and would thereby also exhibit higher pump rates. Hence, the thin-walled soft pump without reinforcement shows the most flexible outer wall and would therefore have the smallest pump rate. This is exactly what our results show (see again Figure 2.3a).
As suggested earlier, characterizing soft pumps by their energy efficiency is a simple way to gain insight into the optimal pump operation range. We adapted these energy efficiency calculations to our system as follows:

\[
EE = \frac{Q_{pot}}{Q_{comb} \cdot n_{CH4} \cdot \Delta H_{comb} \cdot f} = \frac{[J/s]}{[J/s]}
\]  

(eq. 2.1)

where \( m_{pump} \) represents the mass flow rate (kg/s), \( g \) is the gravitational constant (m/s\(^2\)), \( h_{pump} \) is the pumping height (m), \( n_{CH4} \) is the amount of methane (mol) inside the pump at the time of the ignition, \( \Delta H_{comb} \) is the heat of methane combustion (J/mol), and \( f \) is the ignition frequency (1/s). This modification of the energy efficiency calculation is an improvement to the one presented earlier since we here use online mass spectroscopy (MS) to determine the amount of actually used methane inside the combustion chamber (i.e., loss of methane in off-gas stream is partially considered; for calibration and detailed calculations see Appendix A1.2 and Table A1.1). Resulting energy efficiencies are shown in Figure 2.3b for all tested soft pumps. Considering this figure, we immediately identify the best operation regions for soft pumps without fabric layer reinforcement. Both efficiency curves experience a maximum. In the case of the thin-walled soft pump, one meter seems to be the optimal pump height, whereas for the thick walled pump, 1.5 meters of height is a more suited pumping load in terms of energy usage. At these operation conditions, the pumping force is retained best regarding the balance between expulsed volume and surmounted height. We also expect both fabric layered designs to undergo an efficiency maximum at a given point. Because of increased stiffness of the outer walls, these maxima probably lie at higher pumping levels than we were able to test.

2.3.2 Ignition frequency, methane flow rate and the impact of air–methane ratio

After characterizing the influence of the outer-wall stiffness on the pumping performance, combustion processes inside the soft pump were investigated. On the basis of the foregoing results, we only tested advanced soft pump designs with fabric layer reinforcement. First, we powered these pumps within methane rich or methane lean gas mixture regions. The resulting pump flow rates are shown in Figure 2.4a.
Figure 2.4. (a) Pump flow rates are shown as a function of different air–methane ratios for the thick (solid symbols) and thin (empty symbols) fabric reinforced soft pumps. Upper (UEL) and lower (LEL) explosion limits indicate the boundaries of the combustible region. The dashed line represents the point of the stoichiometric mixture (SM) (b) Pump flow rates at different ignition frequencies are shown as a function of the methane flow rate (at an air methane volumetric ratio of 10:1).

The above-described difference regarding pumping performance for the two designs is again observed. Further, pump rates tend to be elevated in the stoichiometric region. If there is more methane added to the feed stream, the resulting mixture will combust rather violently and will decrease the overall pump rate as a consequence of the very short actuation pulses. As the upper explosion limit (UEL) is approached, ignition does not cause any combustion anymore. When less methane is added (i.e., lean methane mixture), almost no impact on the pump rate is observed. The more the lower explosion limit (LEL) is approached, the higher was the misfiring rate of the spark igniter. Table 2.3 shows an overview of the data used for explosion limits.
Hence, powering combustion soft pumps around their stoichiometric ratio is most effective (for correlations used and detailed calculations see Appendix A1.1).

**Table 2.3. Overview on upper and lower explosion limits, as well as stoichiometric mixture.**

<table>
<thead>
<tr>
<th></th>
<th>Upper explosion limit</th>
<th>Stoichiometric mixture</th>
<th>Lower explosion limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>UEL</td>
<td>SM</td>
<td>LEL</td>
</tr>
<tr>
<td>mol %</td>
<td>15.6</td>
<td>9.1</td>
<td>4.6</td>
</tr>
<tr>
<td>vol %</td>
<td>15.7</td>
<td>9.1</td>
<td>4.6</td>
</tr>
<tr>
<td>(\text{V}<em>{\text{air}}/\text{V}</em>{\text{methane}})</td>
<td>5.4</td>
<td>9.9</td>
<td>20.6</td>
</tr>
</tbody>
</table>

\(^a\) Mole fraction for UEL and LEL were calculated from correlations given in the literature.

Details on correlations and calculations are also shown Appendix A1.1.

Beyond, the pumping rates are also dependent on the feed gas flows as becomes evident in Figure 2.4b. The minimal inflow rates that must be chosen in order to achieve a successful ignition lies around 80% volumetric recharging of the combustion chamber between two pumping pulses. Below, residual exhaust products dilute the incoming gas mixture and prevent the buildup of a combustible atmosphere. Above this level, gas mixtures begin to combust more readily and gaseous waste products are flushed out more efficiently by the entering fuel gas. As a consequence, the pumped liquid volume per pulse rises. Lower pumping rates (i.e., at identical gas feed rates but higher ignition frequencies) can be explained exactly on this account. The fact that the pumped volumes still get higher at flow rates allowing complete gas replacement can most notably be addressed to backmixing and channeling effects inside the combustion chamber. Elevated feed rates compared to the required volumetric fuel gas amount therefore result in a better pumping performance per pulse. A second effect becomes apparent at very high feed flow rates and at a pumping frequency of 1 Hz (especially for the thin-walled pump). In this regime, the conveyed volumes per pulse nonlinearly increase. This behavior can be explained by the thin exhaust gas tubing, which adds more and more resistance to the escaping gases. Like this, the combustion chamber experiences increased pre-expansion and thus contains a higher amount of combustible gas than normally. The effect is a performance boost caused by the higher specific energy release upon ignition. However, it is obvious that the excessive feed rates result in disproportional spillage of combustibles compared to the added pump output. Still, the findings
highlight the possibility to adapt power characteristics of a soft combustion machine in very flexible ways.

2.3.3 Exhaust gas tracking

The obtained data for off gas tracking by online MS was not only used for calibration purposes, but also gave good insight into the combustion process. After an ignition event, combustibles and oxidant concentration reduce as a consequence of the combustion reaction. Thus, combustion product concentration will increase. During the ignition lag time, the combustion chamber is flushed with fresh feed gas that contains new combustibles and oxidant. This flushing allows concentration profiles to relax and to readily ignite again. This behavior was observed in our exhaust gas tracking, as shown in Figure 2.5a.

![Figure 2.5a](image)

**Figure 2.5.** (a) Exhaust gas tracking by online mass spectroscopy detection of methane (black), oxygen (red), and carbon dioxide (blue) at a decelerated ignition frequency of 0.05 Hz. Methane and oxygen tracking shows in phase oscillation, whereas methane and carbon dioxide show antiphase oscillation. Initial methane concentration (IC\textsubscript{CH\textsubscript{4}}, green, dotted) and relaxed methane concentration (RC\textsubscript{CH\textsubscript{4}}, green, dashed) are shown as well. (b) Exhaust gas tracking at the usual ignition frequency of 0.5 Hz (used throughout most of the experiments). In-phase oscillation of methane–oxygen tracking, as well as antiphase oscillation of methane–carbon dioxide tracking is still observable.
Further, methane and carbon dioxide are flushed out by air at the end of each experiment (i.e., ignition and methane flow were stopped). For already mentioned reasons, ignition frequency had to be decreased in order to track fine resolved off gas concentration paths. Thus, methane and oxygen concentration have more time to approach their initial concentration. Since most of the experiments were run at 0.5 Hz ignition frequency, an air–methane ratio of 10:1 at 0.3 L/min methane flow, off gas tracking was also performed for this setting. Still, in-phase behavior of methane and oxygen, as well as antiphase behavior of carbon dioxide and oxygen are observable. This is shown in Figure 2.5b. By comparing both tracked paths at 0.05 and 0.5 Hz, one can see that a faster ignition frequency decreases the flushing time. Hence, the difference between initial methane concentration (IC\textsubscript{CH\textsubscript{4}}) and methane concentration after relaxation (RC\textsubscript{CH\textsubscript{4}}) is smaller at 0.05 Hz ignition frequency than at 0.5 Hz. This behavior can also be observed when the carbon dioxide tracking at the mentioned frequencies is considered. In Figure 2.5a, we see that carbon dioxide flushes out and behaves as a tracer being pulsed into a continuously stirred tank reactor (CSTR). The actual amount of tracer inside the CSTR then varies depending on the pulse frequency. In Figure 2.5b, we then observe that carbon dioxide accumulates inside the pump upon an increase of the combustion frequency. These effects have a strong impact on the combustible regions of the soft pumps and might also explain the fact that the UEL and LEL cannot be reached under the here studied operation conditions.

2.3.4 Long-term stability

After 30 000 combustion cycles, the pump suddenly showed feed gas bubbling through the liquid pumping chamber. Hence, a weak spot was detected. Figure 2.6 shows the tested soft pump after cutting it in two halves. Immediately, the perforated spot was detected by the bare eye. On the basis of the gap propagation, this spot was determined to originate from repeated insertion of the feed gas nozzle and the spark igniter into the chamber opening (see Figure 2.6a for details). The soft pump itself still shows unaltered elasticity. This was tested by stretching the outer wall (for further analysis, see Movie A1.2). Nevertheless, the inner combustion chamber section of both shell sides shows thermal and mechanical damage. By bending these combustion shell halves, damage becomes visible in the form of distributed cracks (see Figure 2.6b,c, Figure 2.7 and see Movie A1.2).
Figure 2.6. Photographic overview of long-term stability analysis is shown by the thin layered soft pump after 30 000 combustion cycles. (a) Damaged region caused by periodical reinsertion of the injection nozzle. This damage was identified to be the cause of the pump failure. (b) Observable, thermal damage caused by the combustion process. Material shows already severe cracks which would probably have ended up in a pumping failure. (c) The crack pattern is also observed on the other shell side of the cut-through.

Their depth reaches 2 mm. Compared to the actuation membrane thickness of 3 mm, this represents a 66% cut through the material. Even if damage by nozzle reinsertion is reduced, this pump would most probably have broken down within the next 15 000 cycles (if a linear degeneration rate is assumed). Since the here-used silicone is only optimized for high elongation at break values, it does not offer a very high thermal stability. According to our experience, we expect that the long-term stability could be substantially enhanced by extensive, thermal resistivity silicones. One of the drawbacks of these thermally more stable materials is their comparably low elongation at break values. Hence, future research should target optimized silicone mixtures or alternative elastomers for the manufacture of soft combustion machines. This
breakdown also shows potential for future soft pump manufacturing. Cracks most frequently occurred in regions where 3D printing mold created a step joint due to the spaghetti-like printing of ABS model and PLA support structure. At such a step, mechanical stress is increased. Once tearing occurred at these spots by the mentioned thermal and additional mechanical stress, the crack propagates. Hence, higher resolved 3D printing may lead to an increased load bearing capacity.

![Damage investigation on a combustion chamber wall after 30 000 combustion cycles.](image)

(a) Macroscopic overview over the combustion wall halve shows the cracks formed by thermal and mechanical stress. (b) Magnification under the microscope shows that the surface not only cracked but also debris has formed. (c) Using Z-Stack, a topological view of the crack was established. (d) Further magnification of the wall surface revealed that the silicone was also stressed thermally.

### 2.3.5 Pump stress analysis

On the basis of the observations made earlier, the thin-walled and fabric reinforced soft pump was considered to be the best design. Hence, this pump was tested under extreme conditions by pumping water up to a level of 13 meters. This test allows an industrially relevant comparison of the soft pump performance to that of commercially available gasoline water pumps. (see Table 2.4).
Table 2.4. Comparison of commercially available fluid pumps with similar engine displacements to the thin-walled and fabric layered soft pump design. The value in parenthesis represents the weight of the monoblock pump without any connected parts (i.e. Mass flow controllers, igniters, etc.)

<table>
<thead>
<tr>
<th>Type</th>
<th>Displacement cm³</th>
<th>Weight kg</th>
<th>Total Head m</th>
<th>Max. Flow Rate L/min</th>
<th>Flow Rate / Mass L/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>T.I.P. LTP 40/10</td>
<td>97.7</td>
<td>12.6</td>
<td>15</td>
<td>167</td>
<td>13.25</td>
</tr>
<tr>
<td>Honda WB 20XT</td>
<td>120</td>
<td>21</td>
<td>32</td>
<td>600</td>
<td>28.75</td>
</tr>
<tr>
<td>Soft Pump</td>
<td>94</td>
<td>2.5 /(0.6)</td>
<td>13</td>
<td>0.5</td>
<td>0.2 /(0.83)</td>
</tr>
</tbody>
</table>

Figure 2.8 shows the corresponding scenario schematically (see Movie A1.3 for a detailed pumping scenario).

Figure 2.8. (a) Schematic overview of the extreme pumping scenario is shown in scale. Floor safety clamps (FS) prevented ascending tubing from back slipping. (b) Pumping scenario at the zero level of altitude. (c) Thermal stress caused by the combustion process recorded with IR camera for ignition frequency of 0.5 Hz.

Although the flow rates strongly decreased, a stable conveyance loop was established. Despite the low liquid transport rates, the outflow temperature did not surpass the inlet
temperature by more than 3 °C at any time, and the pump did not heat up to more than 110 °C for 0.5 Hz pumping and 140 °C for 1 Hz pumping at any external spot as shown in Figure 2.9.

![Temperature profile](image)

Figure 2.9. Temperature profile along the thin walled, but fabric layer reinforced soft pump after reaching an altitude of 13 m for an ignition frequency of 0.5 Hz. The outlet temperature (left image) and the inlet temperature (right image) resulted in a temperature difference of approximately 3 °C.

Surprisingly, we found that the forwarded amount of water per pulse at 1 Hz pumping rate was higher than at 0.5 Hz (2.4 g vs 1.6 g per pulse) despite the gas flow rates remaining below the combustion chamber expansion regime as described above. An explanation for this behavior could be the cooling effects of the combustion chamber walls, since the latter is cooled by the exterior liquid. A lower pumping rate also results in lower inner operation temperatures during combustion. Gas near the actuation walls cannot expand as much due to lower temperature progression in these regions. Despite this effect being cumbersome to be verified experimentally, it would mean that a lower overall expansion of the combustion chamber occurs at lower actuation rates. These circumstances suggest that elevated operation temperatures can be beneficial for the performance (similar to a conventional combustion engine). Thus, a balance between heat generation in favor of performance and thermal wear should be established for optimal running characteristics.
2.4 Conclusion

We demonstrated improved designs and manufacturing of pumping-fluid-cooled soft silicone combustion pumps, which are able to pump to a height of 13 meters (42 feet, corresponding to a back pressure of 1.3 bar) without any sealing issues and good wear resistance. This performance was possible without any involved concepts of torque transmission, which would imply the use of bearings and other light engineering. Our findings show that partial flexibility blocking (here expansion) by, for example, incorporated aramid fabrics results in highly increased operation performances compared to non-modified soft motors. We further investigated a broad range of operational parameters such as the influences of different fuel gas mixtures, feeding, and ignition rates. These results represent a base for future soft pump design. We believe that the here presented design could enhance pumping in situations, where weight or replacement time are limiting factors. The here-presented soft pump design investigation would not have been possible without 3D printing, which can produce lost-wax molds within reasonable time frames (less than 10 days to build a complete soft pump). Despite that these pumps can now be used for relevant pressure conditions, their conveyance rates are still comparatively low. One of the main issues is the short and relatively abrupt pressure development in the combustion chamber. When the water in the pumping chamber is displaced very rapidly, it causes more resistance and there is only a short time frame within which it can be expelled through the liquid check valve at the outlet. Consequently, only around 5% of the contained liquid volume was maximally ejected per pulse and the energy efficiencies are still low. Therefore, one of the main targets of future work is smoother actuation (i.e., slower combustion) in order to achieve better energy yields. This can be attained by actually modeling the residence time of the exhaust gases to further understand the combustion process. With this information, PLC could be programmed to flush and charge the soft pump with the exact gas amount needed for such a smooth actuation. Another important finding of this study is the result of charging the soft combustion chambers with variable volumetric amounts of combustibles. Totally inflatable chambers could allow highly adaptive output ratings. Despite stable operability over 30 000 combustion cycles sounding impressive at first sight, this is an insignificantly low value from an application perspective. The pump shows a highly reliable operational and structural integrity, whereas the material clearly failed due to the mentioned combination of mechanical and thermal degradation during operation of the here-used silicone. More robust silicones and other elastomers exist, but they usually exhibit lower
elongation at break values. Hence, soft machines must either be designed with respect to these properties or elastomers could be blended in order to optimize the properties for future long-term application. We believe that continuous operation during days to weeks should be reached following our concepts.
3. An Untethered, Jumping Roly-Poly Soft Robot Driven by Combustion

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Michael Loepfe, Christoph M. Schumacher, Urs B. Lustenberger, Wendelin J. Stark,
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Author contribution:
M.L., C.M.S. and W.J.S. elaborated the concept. M.L. designed the machine. M.L. and C.M.S. designed and performed the experiments with the support from U.B.L. M.L., C.M.S. and W.J.S. wrote the manuscript with contributions and review from all authors.
3.1 Introduction

Recent progress on machines made almost entirely from elastomeric material has extended an engineer's possibilities to actuate systems. Driven by magnetic fields, electrical charge, combustion, or pneumatic expansion, these systems offer already a large application variety. Soft robots are an upcoming class within this field. They have shown their capability to perform complex manipulation or locomotion (tentacles, grabbers, and others). Most of these systems are driven pneumatically via a flexible drive line, transferring gas from a stationary source into an actuation chamber (i.e., a Pneu-Net). Although such a drive line has no direct disadvantages associated with its use, outdoor applications become unfavorable due to risk of tearing the line. For demanding tasks (i.e., during rescue operations) soft robots need to act untethered, fast, and independent in rough terrain. Stokes et al. presented a first step toward an untethered design by combining a mobile, but hard robot (diameter \( \sim 30 \text{ cm} \)) with a tethered and soft grabbing unit (length 11 cm). Tolley et al. then presented an untethered design of their quadrupedal soft robot (length 65 cm, weight \( \sim 4 \text{ kg} \)) being actuated by two mini air compressors (MAC) attached on its top. This soft robot showed reliable performance under different circumstances (i.e., exposed to fire, to a snow storm, and others). Nevertheless, pneumatically driven systems do not have comparably high peak energy densities as combustion-driven systems. This is particularly important if fast movement is required (i.e., on outdoor recon missions).

Shepherd et al. presented a tethered tripedal soft robot (length 13 cm, weight \( \sim 40 \text{ g} \)) driven by combustion of hydrocarbons. Although this jumping robot already showed impressive performance (i.e., maximum velocity of \( \sim 3.6 \text{ m/s} \), jump height of 30 cm, peak power generation of up to 35 W), its design would need further improvement in terms of stability (the robot lasted only for 30 jumps). Tolley et al. then presented a design of a larger soft robot (diameter 15 cm, weight 0.5 kg), able of a single but controlled jump. This robot already advances fast (0.6 m with a single jump) but further design optimizations are needed in order to achieve multiple jumps.

Here, we have developed an untethered jumping soft robot (diameter 18 cm, weight 2.1 kg) largely made from soft material, using 3D printed lost wax molds. Our combustion-driven robot imitates a roly-poly toy and is therefore intrinsically able to perform reorientation into an
upright position after a jump event. Hence, this geometry should favor repetitive jumping events. Fuel and oxidant tanks placed inside the soft robot represent a quick and high-density energy access, which is important in rough terrain. Also, their use can increase operation time in the field. The robot further consists of a thin and flexible actuation wall, which separates the gas combustion chamber from the environment (i.e., the wall facing the ground). This allows adaptation to the soil surface due to the elastomeric properties. We performed tests on different terrains (i.e., smooth concrete and a dirt road) and at different fuel flow rates to evaluate our design for fast and long-lasting locomotion. Video analysis enabled us to track the robots actuation paths. We further analyzed this paths and calculated jumping tendencies (i.e., speed and direction) of the here presented soft robot design.

3.2 Experimental Design

The design presented here is a prototype of a combustion-driven soft robot. Nevertheless, all necessary elements for an untethered system are present. The robot consists of an elastomeric body with an incorporated combustion chamber for actuation. Small gas tanks store combustibles or oxidant. A set of valves and tubing assures proper mixing of the gasses and subsequently fills the combustion chamber with a ready-to-ignite mixture. A radio-controlled (RC) circuit then ignites the mix on demand. A schematic depiction of this jumping soft robot is shown in Figure 3.1 (for more details, see Movie A2.1). All selected material parts result from tradeoffs between simplicity and system control, as well as weight and space demand. Therefore, this design represents a broadening step within the family of untethered soft robots.
Figure 3.1. (a and b) Schematic cross section of the roly-poly soft robot with all used parts. The control unit containing radio-controlled (RC) systems is only symbolically indicated. The red point denotes the center of the hemisphere, which is above all heavy parts. Panel (c) illustrates the lift of the center of mass during displacement. Panel (d) shows the final jumping soft robot with the two views of the cross sections A and B.

3.2.1 Design and manufacturing of the soft robot shell

The design of the here-presented jumping soft robot is based on the principle of a roly-poly toy. This should help the robot (1) to reorientate itself without the need of additional equipment; (2) to save weight; and (3) to increase the robots actuation speed, because potential energy can be converted into a rolling motion. In our design, the rotation center of the semi-hemispherical body thus has to be above the center of gravity to enable reorientation into an upright position after each jump (see Figure 3.1a and b). We added an inner bump to prevent the combustion chamber from collapsing under its own weight. An inner mold sheltered the ignition
control and the fuel power source. We designed the mold as deep as possible in order to further lower the center of gravity. Thereafter, the same manufacturing technique to produce 3D printed and lost-wax-casted soft silicone devices as reported earlier was applied. More in detail, we first designed a virtual mold using computer-assisted design software (NX; Siemens). This design was then 3D printed with acrylonitrile–butadiene–styrene (ABS) model material and polylactic acid (PLA) support structures (uPrint SE Plus; Stratasys).

The resulting mold is washed in an alkaline bath to remove PLA support structures and filled with a room-temperature vulcanizing (RTV) silicone (RTV 1701; Altropol). After elastomer curing, the ABS mold was dissolved in acetone until no residual ABS model material is left. We further added thread connections to mount a wooden plate. This plate enabled an easy attachment of control units (i.e. by sticking them to plate). To install the threads, small cut outs with a size larger than the thread were made into the silicone. Then the threads were placed into cut offs and filled up with uncured RTV silicone mixture. Also a bottom slide made from polyvinylchloride (PVC) was glued into the silicone mold in order to increase the physical separation from the combustion chamber to the control unit and the fuel power source. The glue in this case was again a mixture of uncured silicone. The soft robot is then covered with a lightweight and synthetic bowl in order to protect the inner workings. Further, a camera (eco HD 1080p, 96° lens; ACME FlyCamOne) was installed to record the movement from the robot's point of view (see Figure 3.1c) if needed.

3.2.2 Soft robot control circuit

The schematic control unit (as shown in Figure 3.1a) consisted of two battery packs at different voltages. The first pack (6 V, NiMH 1700 mAh; Conrad) was used to power the RC circuit and the video camera as shown in Figure 3.2. This circuit consists of an RC receiver (6-channel FS-R6B 2.4 GHz FSK with JR plug-in system; Modelcraft) connected to an RC switch (2-channel switch; Conrad). Upon actuation of the RC control (6-channel FS-T6 2.4 GHz; Modelcraft) the RC switch closes and powers a spark transformer (4.8 V; SparkFun) connected to an in-house-made spark igniter. The spark transformer itself is powered by the second battery pack (7.4 V, LiPo, 2700 mAh; Conrad). For reasons of system stability, this circuit was physically separated from the first.
Figure 3.2. (a) Circuit diagram for the here-presented soft robot. All implemented parts are schematically shown. (b) The pneumatic system is shown with all standard symbolic part descriptions. The content of the tank is indicated directly on the scheme. Tanks and injection nozzle correspond to start and end point of the flow direction.

3.2.3 Fuel power source

Our recently presented soft pumps are based on air–methane mixtures, which are continuously fed to combustion chambers and periodically ignited.\textsuperscript{43,80} Exhaust gases are flushed out after each ignition by the inflow of fresh gas mixture. For an untethered setup, we would have either needed a passive gas exchange system or an active one, which would incorporate a MAC as used by Tolley et al.\textsuperscript{83} Since our contribution intends to illustrate also the application of fuels to directly power a soft machine, we incorporated gas tanks. These pressurized tanks store
combustibles or oxidant and allow passive mixing using downstream equipment (i.e., flow regulators).

We adapted our system to combustion mixtures of nitrous oxide, propane, and butane, having the following advantages in mind: (1) higher energy density (reaction energy per inflow volume, in J/cm$^3$) for a propane/butane–air mixture compared to methane–air mixture (see Table 3.1); (2) fluid property inside the gas tank changes from compressed gas (for methane) to a vapor–liquid equilibrium (for propane/butane) and therefore allows a higher tank filling at lower pressure (thereby also reducing tank wall thickness and weight); (3) direct reaction with nitrous oxide instead of air further increases energy density (see again Table 3.1); and (4) both nitrous oxide and propane/butane mixtures are simply available as whipped cream chargers or as refill gas for lighters. Energy densities $\rho_{\text{energy}}$ were calculated upon division of the reaction energy $E_r$ by the volume $V$ of the involved reaction gases. Reaction energy was calculated by multiplying the standard reaction enthalpy $\Delta_rH^\circ$ times the amount of combustible $n_{\text{comb}}$ ($n_{\text{comb}} = 1$ mol). The inflow volume was calculated by multiplying the molar volume $V_m$ at standard pressure and temperature ($V_m = 22.71$ dm$^3$/mol) times the molar amount of the inflow gas $n_{\text{in}}$ according to the reaction equation:

$$\rho_{\text{energy}} = \frac{E_r}{V} = \frac{\Delta_rH^\circ \cdot n_{\text{comb}}}{V_m \cdot n_{\text{in}}} = \frac{[\text{J/cm}^3]}{[\text{mol}]}$$  \hspace{1cm} (eq 3.1)

**Table 3.1.** Overview on calculated energy densities $\rho_{\text{energy}}$ for methane-air, propane-air, propane-$N_2O$, butane-air and butane-$N_2O$ gas systems.

<table>
<thead>
<tr>
<th>Reaction</th>
<th>$\Delta_rH^\circ$</th>
<th>$n_{\text{in}}$</th>
<th>$\rho_{\text{energy}}$</th>
<th>$\rho_{\text{energy}}/\rho_{\text{energy,CH}_4}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{CH}_4 + 2 \text{O}_2 + 8 \text{N}_2 \rightarrow \text{CO}_2 + 2 \text{H}_2\text{O} + 8 \text{N}_2$</td>
<td>-802 kJ/mol</td>
<td>11</td>
<td>-3.2</td>
<td>100</td>
</tr>
<tr>
<td>$\text{C}_3\text{H}_8 + 5 \text{O}_2 + 20 \text{N}_2 \rightarrow 3 \text{CO}_2 + 4 \text{H}_2\text{O} + 20 \text{N}_2$</td>
<td>-2043</td>
<td>26</td>
<td>-3.5</td>
<td>108</td>
</tr>
<tr>
<td>$\text{C}_3\text{H}_8 + 10 \text{N}_2\text{O} \rightarrow 3 \text{CO}_2 + 4 \text{H}_2\text{O} + 10 \text{N}_2$</td>
<td>-2864</td>
<td>11</td>
<td>-11.5</td>
<td>357</td>
</tr>
<tr>
<td>$\text{C}<em>4\text{H}</em>{10} + 6.5 \text{O}_2 + 26 \text{N}_2 \rightarrow 4 \text{CO}_2 + 5 \text{H}_2\text{O} + 26 \text{N}_2$</td>
<td>-2658</td>
<td>33.5</td>
<td>-3.5</td>
<td>109</td>
</tr>
<tr>
<td>$\text{C}<em>4\text{H}</em>{10} + 13 \text{N}_2\text{O} \rightarrow 4 \text{CO}_2 + 5 \text{H}_2\text{O} + 13 \text{N}_2$</td>
<td>-3724</td>
<td>14</td>
<td>-11.7</td>
<td>365</td>
</tr>
</tbody>
</table>

**Figure 3.2b** shows the pneumatic system used to power the soft robot. In-house-made gas tanks were pressurized using either nitrous oxide chargers (Kisag AG) or refill gas for lighters.
containing a mix of propane and butane (25% propane, 75% butane; Rotfill Super 100; Rothenberger Industrial). The gas tanks contain a pressure regulator (part nr 7300; Parker Legris; all subsequent pneumatic parts were bought from the same company), followed by a flow regulator (part nr 7660) to adjust gas flows manually. A 2/2 manual switch (part nr 7802) then allows simple on–off switching. Last, the two gases are mixed with a Y connector (part nr 3140). A check valve (part nr 7996) prior to the in-house-made injection nozzle prevents any backdrafts. **Figure 3.3** shows the exact positioning of the elements inside the soft robot.

![Figure 3.3. Mounting of all elements inside the soft robot starting from (a) battery packs and spark transformer, followed by (b) the fuel power source, (c) RC switch and video camera, as well as (d) attachment of the RC receiver on the hard, wooden plate.](image)

The fuel power source uses manually adjustable valves. These valves are adjustable with a screw driver. First, the pressure regulator screw was adjusted. Therefore, the tank was filled
with either oxidant or fuel gas. Starting from the end flange, the screw was loosening until gas bubbled through attached tubing. Then, the flow regulator and the manual switch were mounted to the system. The flow regulator valve was then adjusted in a similar way. To measure the actual flow rate, the outflow volume was measured inside a water-filled cylinder over a time period of 5 s. This adjustment task was performed for both tanks. Then, the power source and the control unit were mounted inside the soft robot as shown in Figure 3.3. The parts were mounted as follows:

1. Battery packs and spark transformer were placed inside the mold. Care had to be taken to avoid large bending of the already mounted spark igniter.
2. Fuel power source was smoothly inserted and pushed down to the very end, without bending the already mounted fuel injection nozzle.
3. Then, the video camera was attached to the battery. To mount the RC switch, we had to slightly lift gas tubing and insert the switch underneath. Again care had to be taken to avoid any large bending of all switch wires.
4. Last, the RC receiver was placed in such a way that the antenna still was outside the robot. Only right before the experiment started, the video camera was set on record mode and the two manual switches were put on. Then, the synthetic bowl was placed on top of the robot and secured with a rubber band.

3.3 Results

3.3.1 Jump height and material choice

So far, combustion-driven soft robots capable of repetitive jumping were only reported at a small scale (i.e., robot mass of less than 30 g and less than 10 cm³ volume of the combustion chamber). Our system includes a combustion chamber of 65 cm³ at a robot weight of roughly 2.1 kg. This is a clear dimensional increase compared to the values given above for the tripod and tethered robot presented by Shepherd et al. We therefore investigated our robot regarding its jumping capability and its energy efficiency. The fuel power source was manually adjusted to a combustible flow rate of around 1 cm³/s and an oxidant to combustible ratio 5. Jump heights were recorded on video (Legria HF G25; Canon) with a resolution of 2 megapixels and at a speed of 25 frames/s. The ignition was triggered after 20 s. Thus, the combustion chamber was flushed at
least 1.5 times with combustible fresh-gas mixture. To further understand the impact of the oxidant-to-fuel ratio, we recorded jumps with a ratio varying between 5 and 9. Jumps resulting from misfire (i.e., if the injection nozzle was not sufficiently tightened to the soft part) were not included. Jump heights were then evaluated using motion-tracking software (V 0.8.15; Kinovea open source project).

**Figure 3.4a** shows the result of this jump height analysis. The corresponding energy conversion efficiency was calculated for each data point to gain further insight into the jumping behavior. The efficiency corresponds to the ratio of potential energy gained by the jump height and the energy of combustion. In other words, the following equation was used,

\[
EE = \frac{E_{pot}}{E_{comb}} = \frac{m \cdot g \cdot h_{jump}}{n_{gas} \cdot \Delta r_{H_{gas}}}
\]

(eq 3.2)

where \(m\) represents the mass of the soft robot (in kg), \(g\) the gravity of the earth (with \(9.81 \text{ m}^2/\text{s}\)), \(h_{jump}\) the jump height (in m), \(n_{gas}\) the amount of combustible (in mol) and \(\Delta r_{H_{gas}}\) the reaction enthalpy of the combustible (in kJ/mol). We further assumed that the amount of combustible inside the combustion chamber (\(V_{comb} = 65 \text{ cm}^3\)) correlates to the oxidant to fuel ratio (\(V_{ox}/V_{fuel}\)) as follows:

\[
n_{gas} = \frac{V_{comb} \cdot \rho_{gas}}{(1 + V_{ox}/V_{fuel}) \cdot MW_{gas}}
\]

(eq 3.3)

Note that gas density \(\rho\) (in kg/m\(^3\)), molar weight (in kg/mol) and reaction enthalpy correspond to an average value for a given mixture of propane and butane. For a mixture of 25% propane and 75% butane, we calculated an average gas density of 2.7 kg/m\(^3\) and an average molar weight of 0.055 kg/mol. Used standard enthalpies for the different reactions are given in **Table 3.1**. The average jump height for an oxidant-to-fuel ratio of 5 (\(n=4\)) was measured to be 20.1±2.9 cm. Thus, the robot is able to jump on rough terrain (i.e., a dirt road). The robot can gain enough height to partially transform jump energy into a rolling motion after elastic landing (see **Movie A2.2**).
Figure 3.4. (a) Jump heights and jump efficiencies are shown for different oxidant-to-fuel ratios. Average and standard deviation (n=4) were calculated for an oxidant-to-fuel ratio of 5. One recognizes the steeper curve for the energy efficiency. This is due to the reduced relative fuel flow rate at an elevated ratio. Therefore, combustion energy also decreases, which correspond to a positive impact on the energy efficiency. (b) Deformation of the combustion chamber during combustion. Change in angle and displacement lead to a more than fivefold increase in combustion chamber volume.

Due to the thin actuation wall (i.e., a thickness of 3 mm), ignition events were simply observable as a light-flash through the (in principle nontransparent) material. To further illustrate the stress imposed on the material after a combustion event, the soft robot was turned upside down onto its flat top (see Movie A2.3). The corresponding displacement is shown in Figure 3.4b. From the obtained data, we estimated an increase of the original chamber volume of more than five times. This is why we have chosen an RTV silicone with an elongation at break of roughly 1000%. The deformation demonstrates the considerable energy stored already in small amounts of hydrocarbons (i.e., 760 J for an oxidant-to-fuel ratio of 5). This is particularly important for long-running systems.
3.3.2 Roly-poly behavior

Since our soft robot is based on a roly-poly toy, we also investigated the reorientation behavior in a realistic environment. First, combustible flow rate was set to 1.3 cm$^3$/s with a corresponding oxidant-to-fuel ratio of 5 to have similar flow conditions as for the jumping height investigation. Then, we recorded the robot's jumping behavior on video on a dirt road. Ignition times were triggered in such a way that the soft machine had only little kinetic energy left from its last jump (usually 5 s after each jump). The video analysis showed that the before-mentioned reorientation behavior was indeed observable (Figure. 3.5 and Movie A2.1). Such a jump event starts with the ignition of the gas mixture. Depending on the soil surface and the remaining pendulum motion from the last jump, the contact area of the combustion chamber differs. This area contains the normal vector being the direction of the next jump. Then, the combustion energy is transmitted to the ground and the robot jumps.

Figure 3.5. Equilibration process of the roly-poly-like soft robot. The picture series starts at the point of ignition. The inset denotes the adaption of the combustion chamber membrane to the soil surface. Due to remaining impulse from the last jumping event, the soft robot gets further orientated. The red flash then indicates the resulting normal vector, which is the direction of the next jump. The subsequent images then show the landing and equilibrium processes.
Upon landing, the machine equilibrates back into a stable position and thereby rolls, swings, and rotates around. We also observed that there are few landing positions that prevent the robot from any reorientation and therefore stop the equilibration process (see Movie A2.2). This is mainly associated with the fact that the covering bowl has no perfectly spherical geometry, which would allow a reorientation from any position. In principle, a semispherical bowl could be made from elastomeric material with a high hardness. This would still protect the inner parts but might allow better torque transmission to increase rolling motion. However, the roly-poly geometry prevents us from employing further reorientation equipment and therefore allows repetitive jumping. Further, the rolling behavior can help to cover a longer distance, which is important for a fast movement.

### 3.3.3 Jump speed

After demonstrating the ability to jump and reorientate, we further investigated the soft robot's moving behavior. Hence, we recorded the movement of the soft robot from an upright position within an area of 1 m$^2$ on a flat concrete surface. This allowed us to track the robots jump paths in a two-dimensional area. Ignition was triggered after equilibration of the robot's position, which was approximately after 5 s to the precedent jump. We first recorded paths for a fuel flow rate of 1 cm$^3$/s and an oxidant-to-fuel ratio of 5. This corresponds to an actuation chamber volume exchange of roughly 50% and results in smoother combustion due to residual exhaust gases from precedent ignitions. For each run the robot was placed in the center of the analysis section. Then, the soft machine was powered until it either left the area or ignition stopped due to a lack of fuel. If the robot was not able to reorientate after an unsuccessful landing, the recording was stopped as well. In total, we recorded 5 runs with over 50 ignition events. These runs were then evaluated with a video analysis software, including a path-tracking (PT) software for evaluation of the total path. By comparing adjacent video frames the software recognizes movement within a given sector of the frame. The obtained value is then converted from pixel into unit of scale by selecting a reference size (in our case the side length of our square area). We tracked runs of up to 10 meters of length.

Note, however, that the PT software includes the equilibration movements of the robot such as rolling, rotating, and swinging. We therefore further analyzed the videos by manually comparing start and end positions after each ignition. Thus, we neglect nonlinear path
contribution and assume linear locomotion. More specifically, a perspective grid was added to the square test area. Then, all ignitions were located on this grid by flagging the robots position on the particular video frame. The obtained coordinates were then normalized such that the first ignition point is the point of origin for each run. Since these points represent start or end point of jump direction vectors, the magnitude of all vectors could be calculated (see Table 3.2). We measured linear path lengths up to one meter by summing up all magnitudes for a run. This result highlights the before-mentioned deviation between actual path covered and calculated linear path. Nevertheless, linear paths cope better with our understanding of locomotion because we observe the progressive motion of the robot on the terrain. We therefore calculate an average, linear velocity of $0.89\pm0.33$ cm/s with the data from Table 3.2.

**Table 3.2. Summary of runs for a combustible flow rate of 1 cm$^3$/s and an oxidant-to-fuel-ratio of 5.**

<table>
<thead>
<tr>
<th>Run no.</th>
<th>Duration</th>
<th>Cause of stop</th>
<th>Number of Ignition</th>
<th>Total PT$^a$</th>
<th>Linear PT$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>81</td>
<td>No fuel</td>
<td>17</td>
<td>989</td>
<td>96</td>
</tr>
<tr>
<td>2</td>
<td>90</td>
<td>No fuel</td>
<td>12</td>
<td>800</td>
<td>107</td>
</tr>
<tr>
<td>3</td>
<td>73</td>
<td>Bound</td>
<td>8</td>
<td>418</td>
<td>56</td>
</tr>
<tr>
<td>4</td>
<td>64</td>
<td>No fuel</td>
<td>10</td>
<td>423</td>
<td>59</td>
</tr>
<tr>
<td>5</td>
<td>66</td>
<td>No fuel</td>
<td>5</td>
<td>316</td>
<td>26</td>
</tr>
</tbody>
</table>

$^a$ Values obtained by software path tracking (PT).

$^b$ Values obtained by manual path evaluation.

We further investigated the impact of the combustible flow rate on the jump behavior. Thus, we tracked the path for a fuel flow rate of 2 cm$^3$/s at the same oxidant-to-fuel ratio (complete combustion chamber refilling after each ignition). Video analysis revealed that the spatial boundary of the test area was reached after 3 s and one single jump (combustion event). The second jump pushed the robot out of the measurement area into a nearby wall, stopping the experiment after 7 s. Figure 3.6 shows the impact of the fuel flow rate on the jump behavior. Again, we observe the before-mentioned deviation between actual path covered and calculated linear path. The jump distance increased for higher flow rates. For comparison purposes, we also included an additional data point at high flow rate recording (robot left the analysis square right after the second ignition and bumped into the wall). The magnitude between these last two points
is already much larger than for any jump vector of the low flow rate runs. This observation demonstrates the possibility of covering a long distance with a single jump (i.e., three times the robots body diameter) by adjusting fuel flow rates.

**Figure 3.6.** Video analysis of the jumping soft robot on a 1 m² area with a grid size of 12.5 cm per sector field with (a) software-assisted (red) and manual linear PT (blue, combustible flow rate 1 cm³/s, oxidant-to-fuel ratio of 5.5) and (b) increased combustible flow rate of 2 cm³/s. To facilitate reading, linear PT was highlighted only at the beginning (both left), whereas software PT was highlighted at the end (both right).

### 3.3.4 Jump length and direction

This section investigates the observed motions to identify possible preferences in direction. We therefore analyzed the data obtained in the jump section for lower fuel flow in more detail (see Figure 3.7).
Next to magnitude, we calculated the corresponding angles from the jump vectors (Figure 3.8a). Since we did not change any settings between individual ignitions and we allowed sufficient time to re-equilibrate after each jump, we assume that jump events are independent. We then tested whether jumps are biased in a preferred direction. A Rayleigh test (n=52) using circular statistics (CircStat-toolbox; MATLAB\textsuperscript{85}) found that, at the 0.05 significance level, the null hypothesis (no preference) could be rejected with high significance (p=2.7×10\textsuperscript{−7}).
Figure 3.8. (a) The obtained paths (i.e., 5 runs) were translated into a two-dimensional representation containing only the points of ignition (n=52) and the corresponding jump distance. (b) Transformation of the x–y system into polar coordinates. A 0° angle corresponds to a straight-forward jump, positive or negative angle deviation to right, or a left jump direction. The average jump angle and length is indicated as a red line with filled center, whereas standard deviations for distance and angle are indicated by the red area.

We may alternatively analyze changes in direction between a precedent and next jump. A jump angle of 180° corresponds to a straight forward movement. Angles of less or more than 180° correspond to left or right jumping, respectively. Subtracting 180° from the obtained angles
results in the jump angle deviation. Together with the magnitude for the next jump, the data can be represented in polar coordinates, representing the jump length and direction (Figure 3.8b). We calculate an average jump length of 6.4±3.3 cm (n=47) and an average jump angle deviation of 10±45°. This is in agreement with the previous analysis and confirms a bias of the robot in a chosen direction.

3.4 Discussion

We have characterized the performance of a combustion-driven soft robot that is able to reorientate itself into an upright position after a jumping event. Thus, the robot is capable of repetitive jumping. The roly-poly-like reorientation was generally observed, even though there are unfavorable landing spots with a subsequent interruption of this equilibration process. The presented robot has an acceptable size (i.e., almost 20 cm in height with a total weight of 2.1 kg) and copes well with demanding environments. Jump heights were measured to be around 20 cm (body height for an oxidant-to-fuel ratio of 5). An average linear velocity of 1 cm/s was recorded for low gas flow rates. Increasing the flow rate allowed covering of larger distances (i.e., 0.5 m) by a hop-and-roll motion using a single combustion, further demonstrating the promising possibilities of the jumping-based locomotion (see Figure 3.6 and Movie A2.2). We have addressed to the mechanical impact of a combustion event on the used soft silicone and thereby support the demand for high performance elastomers.

In terms of running time, this soft robot still has to be further improved. Operation time was no longer than two minutes, which is clearly unacceptable for potential outdoor use. This short time period is mainly assigned to the insufficient filling behavior of the gas tanks. We have built these tanks using commercially available systems (i.e., charging valves for cream whippers using nitrous oxide chargers). This allowed quick iterations regarding the design of the soft robot. Nevertheless, upon filling such a tank with liquid hydrocarbons at low pressure (again with commercially available gas to refill lighters), the used valves showed unsuitable sealing properties. The liquid fuel only passes the sealing under large human effort. Therefore, integration of hydrocarbon friendly valves would help to significantly improve the filling rate of the tanks.
The need of nitrous oxide should also be put into perspective in combustion-powered soft robots. The gain of approximately $8 \text{ J/cm}^3$ in energy density (or $800 \text{ kJ/mol}$ in reaction enthalpy) justifies the initial gas choice. Nevertheless, a robust gas tank is needed to store the oxidant. Due to usual oxidant-to-fuel ratio of about 5, this tank should store much more gas in order to prevent a limiting situation. Therefore, a simpler hydrocarbon–air mix would not only omit an additional tank, but also extend the theoretical operation time of such a robot. Similarly, another possibility would be switching to other oxidants (i.e., producing oxygen from a liquid–solid system such as $\text{H}_2\text{O}_2/\text{MnO}_2$). This is important for applications where long running times are required on a rough terrain to cover a large area (i.e., mine clearance).

Last, we would like to address the choice of powering soft robots by hydrocarbons instead of electricity: combustion-driven systems take advantage of the gas expansion due to the sharp temperature increase after an ignition event. This expansion is reversed by heat dissipation through transport across silicone walls, as well as heating of inert gases. Hence, high combustion wall surface-to-chamber volume ratio and inert gas content (for air, predominately $\text{N}_2$) cause the total expanded gas volume to compress fast after an ignition. Since the surface-to-volume ratio gets smaller with increasing system size, meter-sized systems should in principle less suffer from subsequent compression. Therefore, future intermediate-sized systems might be designed as a hybrid of electrical and fuel power source.

### 3.5 Conclusion

A long running time is a key feature for soft robots designed to operate outdoors. This time is determined by the energy capacity and the possibility of repetitive ignition. Since we were able to show a design with repetitive jumping, power consumption is the main factor on which future designs should act. This consumption rises if additional equipment is needed to control the soft robot (i.e., video camera for visual observation, GPS tracking system to locate the soft robot, and others). One possibility to account for this higher energy demand would be the addition of further battery packs. Nonetheless, this additional load also increases the power consumption on its own. Especially for large, meter-sized robots, this does not seem to be the solution. Prominent examples are the LS3, the WildCat, or the BigDog, hard robots made by Boston Dynamics. Their main power source is an electricity generator, running on gasoline. Their decision is a logic consequence of the fact that hydrocarbons have a much higher energy density than batteries.
Even a low fuel-to-electricity conversion would lead to higher energy yield compared to Li-based batteries.

We have shown an untethered system running on hydrocarbons and discussed room for improvement. By combining a battery-operated MAC with lighter or camping gas cartridges containing propane/butane, the operation time of a soft robot like ours could be drastically increased. Since the fuel supply system does not operate at a high back pressure (i.e., mostly the check valve would cause hindrance to the gas), the MAC would only need to deliver a pressure that is slightly above-ambient conditions and thereby decrease the electric power consumption drastically. Assuming a fuel flow rate of 2 cm$^3$/s and an oxidant-to-fuel ratio of 12 for our robot (i.e., 7.7 vol% propane in air is regarded as a deflagrative explosion), a standard 240 g cartridge would last for roughly 16.5 h of operation. This value corresponds to about 12,000 combustion events, which is feasible for the here-used RTV silicone as shown earlier on (for an ignition frequency of 0.2 Hz).

Our robot design is currently not aiming on directionally controlled jump events and thus might be used, where larger areas needed to be covered (i.e., mine clearance). So far, the system can be adapted regarding a rough jump direction and jump length. Further adaptations are needed in order to control jump direction in a precise and target-oriented manner. A feasible way to achieve this would include geometric adaptations accommodating several combustion chambers that can be actuated independently. This is further improving the robot's jumping capabilities, which could be particularly advantageous in terrains with demanding obstacles (i.e., recon missions in debris fields).
4. Contrast Agent Incorporation into Silicone Enables Real-Time Flow-Structure Analysis of Mammalian Vein-Inspired Soft Pumps

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Author contribution:

M.L., C.M.S. and W.J.S. elaborated the concept. M.L. designed the machine with support from C.H.B. M.L. and C.M.S. designed and performed the experiments with the support from C.H.B. M.L., C.M.S. and W.J.S. wrote the manuscript with contributions and review from all authors.
4.1 Introduction

Recent development in soft mechanics has yielded machines that are capable of performing very complex movements. They can grab like a human hand, roll like a caterpillar or swim like a fish. Powered by inflating pneu-net actuators, by applying voltage to shape memory alloys or by burning off hydrocarbons, soft machines can use many different actuation principles. The soft principle can be further used to build more basic structures such as soft pumps. These pumps offer several advantages compared with rigid machines, which work within dimensionally tight and preset boundaries. They are lightweight and run even under deformational stress, without the need of a lubrication system. Regardless of the machine, all these systems take advantage of the elastomeric nature of their building material in order to generate actuation. However, none of these systems yet exploited silicone elasticity to gain further information on the running characteristics of these machines.

Noninvasive examination techniques are well known for soft tissues. Using magnetic resonance imaging (MRI), computer tomography or ultrasonography, medical professionals examine human tissue on a daily basis to explore the inner life of their patient's bodies. Especially, ultrasonic imaging is widely applied in medical care due to comparably low handling costs and versatile applicability (i.e., most parts of the human body can be investigated). Ultrasonography is also part of the online-monitoring equipment during heart surgery. There, these devices take advantage of the Doppler Effect to detect flow within the scanned area. So-called color Doppler imaging (CDI) forms a standard procedure to evaluate the integrity of i.e., a heart valve by measuring any undesirable backflow. Research has already combined this technique with computational fluid dynamics (CFD). CDI delivers reliable flow information, which can be implemented as boundary conditions into numerical models of CFD calculations. Nevertheless, these investigations yet only focused on biomedically relevant systems, such as mitral regurgitation (insufficiency of the left atrioventricular valve to close properly). Combining medical imaging with traditional soft mechanics would therefore create a technique, allowing the gain of flow information for directly applicable systems.

Medical professionals train many years in order to measure, analyze, and interpret obtained data of such noninvasive imaging methods. One of their main focuses in ultrasonography is the development of the spatial sense to recognize tissue structures, nerves
blood vessels or bones during an examination session.\textsuperscript{102} To facilitate interpretation, contrast agents are widely applied.\textsuperscript{103} Introduced into a patient, they enhance backscattering due to material properties of the contrast agent. Usually, vessel-rich body parts benefit from this enhancement, because the fluid phase contains the contrast agent. However, this enhancement could also take place in an elastomeric phase, enhancing wall contrast. This would allow better recognition of elastomer movement in soft actuation, especially for untrained examiners.

In this work, we demonstrate the successful flow analysis inside a soft pump by CDI after contrast agent incorporation into elastomer, as well as reliable fluid pumping using different pump actuation patterns. Based on lost-wax casting of virtually designed and 3D printed injection molds, we produce a soft pump analogue inspired by the functionality of mammalian veins.\textsuperscript{43,80,104} Our modular mold approach enables production of monoblock pumps with different, segmented length (up to 80 cm), approaching the general design of a human vein (i.e., several valves inside an entire vein). Incorporation of a simple contrast agent modifies the used room temperature vulcanizing (RTV) silicone elastomer, and thereby enhances imaging properties. This modified silicone is then injected into the molds. Actuation by internal gas combustion or by pressurized air is evaluated for a reliable pumping. We image internal flow at common actuation regimes by CDI for different pumping fluids, such as water, silicone oil, ketchup or starch-water mixture. Simple parameter analysis evaluates the impact of pump frequency and pressure on the obtained pump rate, optimizing the soft system to pump up to five meters of height at a flow of over 500 mL min\textsuperscript{-1}.

4.2 Design of Venous Soft Pump Analogue

Blood vessels are commonly examined by medical professionals using ultrasonic imaging. We designed a vein-like soft pump, capable of peristaltic pumping of fluids. This pump follows the same pumping principle as in mammalian veins (Scheme 4.1). Upon relaxation of adjacent muscles or pulsating arteries, the squeezed vein creates a slight vacuum. The vacuum then forces the valve lips facing the squeezed vein section to open. This segment fills with blood until pressure difference is equalized or another contraction incident occurs. Such a contraction incident squeezes the vein again and increases the pressure inside. The valve facing its lips toward the pressurized segment now closes. The other valve is forced to open and thereby
expulses blood into the next vein section. Refilling of one segment is usually supported by expulsion of an adjacent segment, enabling this passive transport.

**Scheme 4.1.** (a) Operating mode of mammalian veins. Upon relaxation of adjacent muscles or pulsating arteries, a slight vacuum is created inside a vein segment (a, left). This leads to an opening of the valve lips, facing the segment of interest. The segment is now filled again with blood. Subsequent muscle contractions or artery pulses then also contract the venous segment again (a, right). The increasing pressure forces the left valve (facing the segment) to close, whereas it pushes the right valve (valve lips point to next segment) to open. (b) Design of our soft pump analogue. The fluid chamber (lumen) contains vein-like valves. Contraction of muscles or artery pulses is simulated by a pressure chamber, which can be driven pneumatically or by gas combustion.

Our pump comprises a pressure chamber, which imitates the contraction of adjacent muscles or pulsating arteries (**Scheme 4.1b**). This chamber jackets the tubular pump chamber, leaving space for a thin actuation membrane within (**Figure 4.1**). Upon a pressure increase inside
the pressure chamber (by pneumatics or by gas combustion), the fluid phase is squeezed like in a mammalian vein. Adjacent valves with similar, natural shape then guide the flow.

Figure 4.1. Design sketches are shown for the pump module. Important dimensions are indicated. The module has the function of a lost-wax mold, which was virtually inverted and 3D printed. Cross section parts are indicated with hatching. All sizes are given in mm.
Subsequent pressure release (i.e., by pressure relief valve opening) relaxes the soft pump and allows refilling of the fluid chamber (Figure 4.2). For direct inflation of the pressure chamber, aramid fabrics are used as an outer shell. The pump can be driven by pneumatic expansion or gas combustion. In both cases, a programmable logic controller (PLC) controls valves (and respectively an electric spark ignition for gas combustion operation). Since we are mainly going to use pneumatics to power this soft pump, we explain its design more in detail. In a relaxed state, the pump has closed inlet- and open outlet pressure chamber valves. When the PLC triggers a pumping pulse, the inlet valve opens and the outlet valve closes. The jacket now fills for a pre-set time and pressure. This consequently increases fluid pressure and expels the pump fluid as described above. To relax, the PLC closes or opens inlet and outlet valves, respectively. The jacket relaxes and fluid pressure decreases.

![Figure 4.2](image)

**Figure 4.2.** Design sketches are shown for the vein valve module. Important dimensions are indicated. The module has the function of a lost-wax mold, which was virtually inverted and 3D printed. Cross section parts are indicated with hatching. All sizes are given in mm.
Our blood vessels usually contain several valves within a limb. For instance, the great leg vein, *V. saphena magna*, contains roughly eight valves in an adult person. One of our pump designs therefore consists of several pump segments to mimic such veins. Recently, we produced soft pumps by injection of RTV silicone mixtures into 3D-printed molds, designed by virtual lost-wax casting. For this soft machine type however, pump size would be limited to available 3D printing dimensions size. Thus, we designed valve modules and pump modules, which can be assembled together in a repetitive way to form a larger injection mold. These molds can be filled with modified silicone containing a contrast agent. The contrast agent enables enhanced imaging properties for non-invasive examination techniques, such as medical sonography or MRI.

### 4.3 Material and Methods

#### 4.3.1 Silicone modification

Silicone modification was performed by incorporating different types of soda lime glass beads (Cospheric LLC). Incorporated bead types were SL11 (d$_{50}$ = 5 µm), SL15 (d$_{50}$ = 11 µm), SL50 (d$_{50}$ = 36 µm) and SL75 (d$_{50}$ = 69 µm). Incorporation was performed by mixing prepolymer (Neukasil RTV 23, Altropol) with 1 wt% of glass beads in a dual asymmetric centrifuge (SpeedMixer DAC 150 FVZ) for 3 min at 3400 rpm. To get lower loadings of incorporated glass beads, the mixture was further diluted with pre-polymer. Each dilution step was followed by centrifugation to give silicone-bead mixtures at 1000, 100 and 10 ppm, respectively. The highly viscous doped silicone mixtures were then mixed with 30 wt% crosslinking agent (VN A7, Altropol) with respect to pre-polymer mass by mixing each mixture with the dual asymmetric centrifuge (0.5 min at 3400 rpm). Silicone mixtures were either prepared for subsequent injection molding, tensile testing or cured directly at room temperature to form imaging test specimen. Reflected-light microscopy (Axio Imager.M2m, Zeiss) and stress-strain curves were recorded to further characterize the impact of silicone modification on the tensile properties.

Tensile tests (n=5) were conducted according to DIN 53504 on a Shimadzu Universal Testing Instrument AGS-X equipped with a 500 N load cell. According to the standard specimen design, a mold was fabricated by milling its dimensions into an aluminum plate (Figure 4.3). Uncured silicone mixtures with or without glass beads were then poured into this mold to produce tensile specimen. After curing, the thickness of each specimen was measured threefold
within the given section by the DIN standard. Average thickness per specimen was then entered into the evaluation software. Tensile specimens were clamped into tensile testing device whereupon the test was carried out at a pre-stress of 0.01 MPa and a chart speed of 200 mm/min. Images from light microscopy were obtained by calculation from a measured focus stacking (Z-stacking). This allowed broadening of the focal zone, which increases visibility of the incorporated beads in the cured silicone. Z-Stack was measured in bright field mode.

![Figure 4.3](image)

**Figure 4.3.** a) Dimensions of the tensile test specimen S2 are shown according to DIN 53504 standard. B) Milled aluminum mold is presented for test specimen production. This mold was then filled with uncured silicone mixtures. After curing, specimens were dug out carefully.

### 4.3.2 Production of material test valve

The design of the material test valve is shown in **Figure 4.4**. The tester was produced by virtual lost-wax casting of 3D printed injection molds as shown elsewhere. More in detail, our design was virtually inverted using computer aided design software (NX 8.5, Siemens) and then 3D printed (uPrint SE, Stratasys or Design Jet Color, HP). The printed mold made from acrylonitrile-butadiene-styrene (ABS) copolymer as model and polylactic acid (PLA) as a support polymer was then washed in an in-house built alkaline bath to remove PLA support structures. After drying at 60 °C, the mold was either filled with the before mentioned silicone mixture or
with unmodified silicone mixtures and cured at room temperature for 24 h. Unmodified silicones were degassed (20 mbar) by evacuation to remove dissolved gas. ABS was then dissolved in an acetone bath. Last, check valve and positioning tubing were attached to the material tester (1/4” with spring pressure 0.09 psi, SmartProducts).

**Figure 4.4.** Design sketches are shown for the material test valve. Important dimensions are indicated. Cross section parts are indicated with hatching. All sizes are given in mm.

### 4.3.3 Production of venous soft pump analogues

The design of the venous soft pump analog is given in Figures 4.1, 4.2, 4.5 - 4.7. Few additional steps were needed to produce venous soft pumps compared to material test valve production. Depending on later pump size, different numbers of modules (i.e. valve, pump, bottom or top module) were 3D printed. A one segmented pump mold consists of a bottom, a top, two valve and one pump module. A four segmented soft pump consists of a bottom, a top, five valve and four pump modules. After printing, PLA support structure was removed in the in-house made alkaline bath. To combine the modules, stacking was applied (Figure 4.8). All modules had to be glued together prior to filling (Kontaktkraftkleber Flüssig, UHU). Further sealing of possible leaking spots was performed by hot glue injection as a precaution.
Figure 4.5. Design sketches are shown for the bottom module, where silicone injection starts. The module has the function of a lost-wax mold, which was virtually inverted and 3D printed. Important dimensions are indicated. Cross section parts are indicated with hatching. All sizes are given in mm.
Figure 4.6 Design sketches are shown for the top module, where silicone last flows through just before entering reservoir tubing. The module has the function of a lost-wax mold, which was virtually inverted and 3D printed. Important dimensions are indicated. Cross section parts are indicated with hatching. All sizes are given in mm.
Figure 4.7. Assembly sketches are shown for a four segment soft pump analogue. Bottom and top module are not shown in this figure. Important dimensions are indicated. Cross section parts are indicated with hatching. All sizes are given in mm.
Figure 4.8. Process is shown of gluing different modules together to form composite mold (from left to right). The build-up starts with a bottom and a first vein valve module. Then, pump and valve module are repeatedly added until the desired size is reached. The composite mold is then closed with the top module. To produce single segment pumps, top module would be glued already on the composite mold, shown in the third picture from the left.

We pressed the silicone into the mold by repetitive filling and pressurization of an u-bend, glued to the filling inlet (Figure 4.9). Filling was performed until the reservoir tube, a tube glued into the top module, started filling. This reservoir helped to compensate for any silicone losses, caused by the slightly permeable structure of the ABS walls. Depending on the pump design (one segment or four segments), we injected a blend of two silicone mixtures (Neukasil RTV 23 with 30 wt% VN A7, Protosil RTV 240 with 10 wt% Compound B in a 50:50 mixture of the two RTV-systems, all Altropol). The one segment pump contained incorporated soda lime glass beads at a concentration of 100 ppm, incorporated by speed mixing (mentioned before).
Figure 4.9. Module composite mold is shown before (left) and after (right) silicone injection. Effluent lines (silicone tubing) were closed after silicone filled part of the line. Plug (red dot) was used to seal filling tube in order to prevent any back silicone reflux.

After ABS removal in an acetone bath, further production steps were needed to build a fully functional soft pump with an outer fabric shell. Therefore we cut aramid fabric to the size of the soft pump and wrapped the fabric around. To fixate, a fast curing RTV silicone (RTV 22, equal amount of pre-polymer and crosslinking agent) was brushed into the fabric layer without
previous evacuation of the silicone mixture. After curing the pump was suspended in straight-up way (i.e. pumping direction faced the floor), where each valve was fixated with a clamp. Upon tightening, each clamp would seal its valve. We then filled RTV 22 into the most upper valve. The silicone started dripping through the pump. When silicone dripped out of the last valve, all clamps were tightened to seal. After curing valves were cut open by an in-house made scalpel. To reach inner valves, endoscopic guidance assisted valve opening (Findoo MicroCam, dnt).

4.3.4 Ultrasonic Imaging

Ultrasonic imaging of all silicone-bead blends, either present as imaging test specimen, material test valves or venous soft pump analogues, was performed with a linear array transducer (VF10-5, Siemens) connected to a medical ultrasound scanner (Antares, 2008, Siemens). Imaging test specimen and material test valves were analyzed in deionized water, containing 500 ppm soap to avoid air bubble attachment on silicone surfaces. Then, the linear transducer was slightly immersed such that the array was completely under the water level. Venous soft pump analogues were imaged by applying sonogel (Cubitainer, Gello GmbH) between transducer and pump. This enhanced transmittance of sonic waves into the pump. 2D mode images were recorded at 5.71 MHz to penetrate deeply into the pump structure. CDI was performed by switching within different linear velocity ranges. Highest linear flow rate was determined by visual inspection of all recorded videos. Silicone oil (350 mPa s), ketchup (57, Heinz) and corn starch-water-mixture (1:1-mixture) were used.

4.3.5 Pumping of venous soft pump analogues

Combustion-driven pumping was performed as follows. The gas was mixed using two flow controllers connected to a Y-mixer, one for air and one for methane (Red-y compact series, Vögtlin), at a flow rate ratio of 10:1. An overall flow rate of 6.6 L/min was applied. Combustible gas mixture was filled into the pressure chamber. In order to prevent any backdraft, a safety valve (RF53 N for combustible gases, PanGas) was installed directly in front if the pressure chamber. Gas ignition was performed using spark gap igniters (4.8 V, SparkFun Electronics) addressed by the PLC. The PLC not only controlled ignition, but also exhaust valve (VX245JE, SMC). The valve closed for 25% of the cycle time upon an ignition event. Ignition frequency was set to 1 Hz.
Pneumatic-driven pumping was performed similar to combustion-driven pumping. Instead of mass flow controllers, an inlet valve controlled gas filling (VDW20HA, SMC) per pump segment. Inlet and outlet valves were again controlled by the PLC. We used compressed air without any pre-treatment (i.e. filtration) to fill pressure chambers. Pumping for imaging purposes was performed at a hydrostatic head of one meter, pressure of 1.5 bar and pump frequencies of 0.5 or 1 Hz. Filling and ventilation times were set to 25% and 75% per cycle time, respectively.

Pneumatic-driven pumping for pattern analysis was performed at a hydrostatic head of two meters with pressures ranging from 0.7 to 2 bar with frequencies of 0.5, 1 and 2 Hz. Filling and ventilation times were again set to 25% and 75% per cycle time, respectively. We analyzed four different pumping patterns regarding maximum pump capacity for a quad segmented (X-X-X-X) soft pump analogue. Pattern 1 and 2 represent subsequent (1-2-1-2) and simultaneous (1-1-2-2) pumping in a double segment, whereas pattern 3 and 4 represent subsequent pumping without overlap (1-2-3-4) and with overlap (4/1-1/2-2/3-3/4) of the entire quad segmented pump. These patterns were investigated fivefold.

Pattern 3 was then applied to pump up to five meters of hydrostatic head, varying again pressure within 0.8 and 2.2 bar and frequency between 0.5 and 2 Hz to get the impact on the pumping rate in more details. We also used a second exhaust per pump segment including a second exhaust valve (Magnetic valve 82 510, Busch Jost). Filling and ventilation times were switched to 50% per cycle time each.

### 4.3.6 Calculation of pump energy and pump efficiency

The simplest way to get a rough estimation of the resulting pump energy is achieved by considering the actual amount of water, which is displaced by a jacket squeezing step. We therefore calculated the pump energy as follows,

\[
E_{pump} = p \cdot dV = p \cdot \frac{dm}{\rho} = p \cdot \frac{m}{\rho} \cdot f
\]  
(eq. 4.1)

where \(p\) represents the applied pressure in [Pa], \(m\) the mass flow in [kg/s], \(\rho\) the fluid density in [kg/m\(^3\)] and \(f\) the pump frequency in [1/s]. We calculated the pump energy to be 2 J for a pressure of 1.8 bar, a frequency of 1.25 Hz and a pump rate of 8.75 g/s and a density of 1000 [kg/m\(^3\)].
To calculate actual energy efficiency, we need an estimation of the energy put into our system. We calculated the energy input similar to \textbf{Equation 4.1}, but this time, the volume difference was obtained by measuring the volume of the exhausted gas (i.e. gas spilled out after exhaust valve opening within a single pump cycle):

\[ E_{in} = p \cdot dV = p \cdot V_{exh} \]  \hspace{1cm} (eq 4.2)

where \( V_{exh} \) is the exhausted gas volume \([m^3]\). We calculated the input energy to be 200 J for a pressure of 1.8 bar and a measured exhausted volume of 1.1 L. The energy efficiency is then calculated by the ratio of these values:

\[ EE = \frac{E_{pump}}{E_{in}} = 0.01 = 1\% \]  \hspace{1cm} (eq. 4.3)

\textbf{4.4 Results and Discussion}

\textbf{4.4.1 Silicone Modification}

The most important effect of contrast agents in medical sonography is their ability to enhance backscattering (echogenicity). This is usually achieved by introduction of free or encapsulated gas bubbles, colloidal suspensions or emulsions, which can be applied intravenously. These agents effectively increase scattering cross-section of a small scatterer, which is given by the expression from Ophir et al\textsuperscript{106}.

\[ \sigma = \frac{4\pi}{9} k^4 a^6 \cdot f(\kappa_s - \kappa, \rho_s - \rho) \]  \hspace{1cm} (eq. 4.4)

where \( k = 2\pi\lambda^{-1} \) = wave number and \( \lambda \) is the wavelength; \( a \) = radius of scatterer, much smaller than \( \lambda \kappa = \) compressibility difference between scatterer and tissue; and \( \rho = \) density difference between scatterer and tissue.

Since we want to incorporate contrast agents to increase echogenicity of our soft pumps, our agents should be simple and manageable for silicone modification. Gases are very well soluble in silicone and would correspondingly hinder a size-controlled incorporation of gaseous contrast agents\textsuperscript{107}. Further, our modification should not negatively influence material properties. Otherwise we would create soft pumps, which would not reflect the actual behavior of the structure and the material upon actuation. Modifying agents should therefore be low concentrated
in order to maintain material properties. Thus, fluid–fluid or fluid–solid systems might be challenging as well, because properties are likely to get lost during incorporation (i.e., phase dilution).

Spherical soda lime glass beads are a simple and manageable contrast agent. For such solid back scatterer (i.e., $\kappa_s \ll \kappa$ and $\rho_s \gg \rho$), this function term reduces to a single-digit value, which simplifies Equation 4.4 to

$$\sigma \approx a^6$$

when we measure at a constant wave length. The effective scattering cross-section then results from the number of scatterers, $m$ and the individual scattering cross-section $\sigma$

$$\sigma_{eff} \approx m \cdot \sigma \rightarrow \sigma_{eff} \approx m \cdot a^6$$

(eq. 4.6)

In other words, our contrast agent needs to be as large as possible to get good echogenicity but also as diluted as possible to maintain material properties. It becomes obvious that there is a size optimum of the used beads.

This size effect is shown in Figure 4.10, where we recorded sonograms of soft silicone with different bead loadings and sizes. We immediately see that for a given size, backscattering decreases for smaller bead concentrations. Further, we observe decreased backscattering for decreasing bead sizes (Figure 4.10b–d) as Equation 4.6 suggests. This trend is best observed by considering a single particle (i.e., at high dilution) to see its actual backscattering. Hence, we have demonstrated successful incorporation of soda lime glass beads into silicone to control backscattering for ultrasound imaging.
Figure 4.10. (a) Embedded material test specimens are shown for backscattering analysis. Selected data is illustrated with a red dashed square. Longitudinal (L) and transversal (T) imaging planes are indicated on a test specimen. (b–d) Recorded sonograms for different particle sizes at different bead loading. For a better visualization, recorded scans were inversed in grayscale. Solid, black line represents immersion depth of specimen embedment. Dilution factors are given per mass of prepolymer used. Dashed red circle illustrate achieved backscattering by single particles.

However, the mechanical properties should not change significantly as mentioned earlier. From the obtained backscattering matrix (Figure 4.10), we therefore concluded that the largest bead size (SL75) at a concentration of 0.1 wt% per mass of prepolymer would suit best for our
purposes. We therefore tested tensile properties according to DIN 53405 for unmodified and modified silicone (Figure 4.3). The results of this test are shown in Table 4.1. No significant changes of the tensile strain properties were found. A two-tailed t-test further showed no deviations for a significance level of 5%. Therefore, we can conclude that the material modification does not substantially change our systems and we can expect comparable acting of our soft pumps upon imaging.

**Table 4.1.** Results of stress–strain test for silicone with and without soda lime glass beads incorporation. Data was obtained according to DIN 53504 and for a sample number of \( n = 5 \).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Stress ( \text{MPa} )</th>
<th>Strain ( % )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicone</td>
<td>0.79 ± 0.13</td>
<td>674 ± 67</td>
</tr>
<tr>
<td>Silicone with SL75 beads</td>
<td>0.83 ± 0.12</td>
<td>680 ± 87</td>
</tr>
</tbody>
</table>

### 4.4.2 Ultrasound imaging and flow visualization

Powering soft pumps by pneumatics or by gas combustion has deviating consequences for subsequent analysis. Combustion results in sharp pressure increases, which can however not be maintained for long time periods (less than 10 ms)\(^4\). Pneumatic actuation therefore results in different actuation behavior. The pressure increase takes much longer due to mass transport limitation imposed by the small pressure chamber inlet diameters, but the pressure can be kept for long time periods due to the isothermal behavior. In order to visualize these effects, we pumped to a hydrostatic head of two meters using a single segmented pump, either driven by methane combustion or compressed air. Gravimetrical flow analysis for a pump frequency of 0.5 Hz showed that pneumatics performed superior (4.4 g s\(^{-1}\)) compared with gas combustion (1.5 g s\(^{-1}\)). Hence, we focused on pneumatically driven systems in this work, allowing a simpler control mechanism.

Being able to modify soft silicones by incorporating soda lime glass beads (Figure 4.11), we focused on ultrasound imaging. A simple material test valve was designed, which can be actuated by manual squeezing of a bulb-shaped posterior part (Figure 4.4). Upon actuation, sonography is able to record all valve movements. Imaging was performed in a water bucket
containing 500 ppm of liquid soap. The soap changes water surface tension and correspondingly reduces air bubble attachment on silicone (i.e., less signal attenuation). The array transducer was placed in such a way that the imaging plane results in a cut through the valve axis (Figure 4.11b). We were able to observe valve movement by squeezing the bulb (Movie A3.1). Ultrasonic scans showed again that our silicone modification with contrast agent increased backscattering. This can be seen by comparing the wall contrast for modified and unmodified silicone (Figure 4.11b,c). We further analyzed contrast in the motion mode (M-mode). This technique resolves a single pixel line in time and thereby gives insight into the motion behavior of a moving system (i.e., a valve flap). We again observed increased backscattering in 2D-mode, which helped to identify a promising movement. After switching into M-mode, the observation was focused on the movement and therefore, the increased backscattering was less important.
Figure 4.11. (a) Reflected-light microscopy images of incorporated soda lime glass beads in silicone. For a better visualization, several slices were recorded to form a z-stack projection of the material. (b) Cross-section scheme of the material test valve design is shown with further illustration of the imaging process. The bulb part of the tester can be used to manually pump fluid through the tester. Sonograms of material testers with and without incorporated beads are shown in the red squares below. Increased backscattering is observed within material walls compared with unmodified silicone. (c) Material test valves are shown with all additional connections (check valve and positioning tubing).

As already mentioned in the introduction, the Doppler effect can be used to visualize flow dynamics in soft tissues. Depending on incident, backscattered waves, flow speed, and direction can be assigned (flow toward or away from the probe section). A color map then visualizes this information on the recorded 2D image in real time for a chosen section. We applied this standard
medical examination technique to our soft pumps. The material tester was investigated at different squeezing velocities. Color Doppler recordings show different linear velocities, which cope well with applied squeezing (Movie A2.2). Hence, flow visualization is possible for our small sized material test valves.

Our aim however is to visualize flow in larger pumps. We performed ultrasound imaging and flow visualization for a single segmented version. This version has not yet the size of the final multisegment pump, but already uses the same valve and basic pump design (Figure 4.12). The single segment also delivers reasonable flow information on internal currents during pumping action (Figure 4.13). Regions of interest for venous flow analysis are, besides junctions, located directly after a valve. In mammals, currents can create blood stagnation at these locations that may support formation of varicose veins. This is why we also expect most interesting flow currents in the region of the vein pocket, located after the valve.

Figure 4.12. Overview on the produced soft pumps. (a) Material test valve designed for initial imaging tests. (b) Single segment venous soft pump analogue with schematically visualized cross-section. The upper valve part has no reinforcing fabric layer to allow direct application of the ultrasonic transducer array on the pump. (c) Quadruple-segmented venous soft pump analogue with schematically visualized cross-section
Figure 4.13. Flow visualization in a venous soft pump analogue using color Doppler imaging. The region of interest is set to the valve pockets, where most interesting currents are expected. For a better understanding, pump walls and valves are highlighted by a schematic, semitransparent layer. (a–d) show flows of different pump fluids. Valve movement is shown on the right side of each fluid system by a time-resolved motion line. Flow dynamics of (a) water and (b) silicone oil (both Newtonian fluids) are shown at a pump frequency of 1 Hz. Non-Newtonian fluids as (c) ketchup and (d) starch–water mixture were pumped at a lower frequency of 0.5 Hz.

We evaluated flow characteristics for different fluids (i.e., water, silicone oil, ketchup, and starch-water mixture) to get further understanding of fluid properties impacting our pump characteristics. Water pumping showed the fastest currents with a linear velocity of roughly 6 cm s\(^{-1}\) (Figure 4.13a). Video evaluation suggests that there is only a single current zone induced by the valve movement within the pocket (Movie A3.3). When the valve is pushed open by a pressure front, the adjacent fluid elements in the pocket give way by moving on one single side. Subsequent wall relaxation then turns pocket currents around. Another good impression of flow characteristics is again given by the M-mode to gain insight into the motion behavior of a valve. We did observe sharp and symmetrical pulse amplitudes with a relaxation width (i.e., dead time) of roughly 0.5 s between two pulses. This suggests that the pump frequency might be increased to have less dead time between two contractions. Also, symmetrical pulse
characteristics suggest good valve opening and closing time (i.e., silicone valve can open and close without experiencing severe resistance by the fluid).

By increasing fluid density and viscosity (i.e., pumping silicone oil), we observe decreased linear velocities only up to $4 \text{ cm s}^{-1}$ (Figure 4.13b). Also, there seems to be a second current zone close to the pocket neck with an inversed flow (Movie A3.3). The recorded motion line showed an unsymmetrical pulse shape, flattening on the right-hand side with no observable dead time. Hence, pumping frequency is at an optimal point but the flattening suggests improving of the valve design for fluids with higher viscosities (i.e., increasing valve stiffness) to enable faster closing times. Pumping non-Newtonian fluids revealed further flow characteristics (Figure 4.13c, d). Ketchup, a shear-thinning fluid, showed even lower linear velocities up to $2 \text{ cm s}^{-1}$, while again observing a second current zone (Movie A3.3). Pulse profiles were flat but symmetrical, thereby suggesting that the pump had difficulties to displace fluid elements in the valve pocket. Dead time was estimated to be roughly 1 s, thus frequency could have been increased to 1 instead of 0.5 Hz. We observed similar flow characteristics for a shear-thickening starch-water mixture. A starch-water mixture incorporates many small starch grains, which cause a lot of signal attenuation. For this reason, it was difficult to penetrate deeply into the soft pump while imaging.

Even though our flow analysis is rather approximate than detailed, we were able to gain real-time insight into the soft pumps. We strongly believe that with further improvements, considerably wider comprehension could be possible. This would be of great interest to research areas employing CFD and fluid-structure interaction. Usually their computational models are in need of appropriate boundary conditions in order to converge. By designing soft analogues of the systems of interest (i.e., industrial valves), this information would be accessible. Combining CFD with noninvasive flow measurements was so far performed for medically relevant systems, such as heart support systems. We hereby show that data can also be obtained by CDI for venous soft pump analogues and more generally in soft machines through the incorporation of contrast agents into elastomers.

### 4.4.3 Peristaltic pumping of venous soft pump analogue

In order to investigate pumping of the four segmented soft pump, we pumped water to a hydrostatic head of two meters by applying different pressure chamber actuation patterns and
conditions. As already mentioned, gas valves were controlled by a PLC, allowing simple modification of the pump patterns. First, we analyzed two-segment patterns with single gas in- and outlet, as well as filling and ventilation times of 25% and 75% per pulse interval, respectively. These patterns consisted of subsequent or simultaneous actuation of two segments inside the four segmented soft pump. To facilitate cycle understanding of the segments, we hereby introduce an (X-X-X-X) nomenclature. Each X represents a pump segment to which, number values are assigned according to their relative actuation moment within a cycle. Therefore, the subsequent, two-segment pattern within a four segmented pump would have the notation (1-2-1-2). The simultaneous pattern would have the notation (1-1-2-2). We then evaluated mass pump rate by gravimetrical analysis for pneumatic pressures of 0.7–2 bars with pump frequencies of 0.5, 1 or 2 Hz (Figure 4.14a). Flows were normalized by the pump frequency to get mass flow rates per pump beat.
Figure 4.14. Influence of pressure and pump frequency on mass pump rate is shown for two and four-segment pump patterns. Single in and outlet was used with filling and ventilation times of 25% and 75% per pulse interval, respectively. All measurements were taken fivefold. Resulting standard deviations are indicated in the two plots. (a) Two-segment patterns are shown for a subsequent (1–2–1–2) and simultaneous (1–1–2–2) segment actuation. Visualization for both patterns is given on the right. (b) Four-segment patterns are shown for direct (1–2–3–4) and for an overlapping pulse propagation (4/1–1/2–2/3–3/4). Pattern visualization is again given on the right.

We observed that the (1-1-2-2) pattern mostly results in higher pump rates than the (1-2-1-2) pattern, regardless of applied pressure and frequency. This might be explained by a beneficial interaction of the two adjacent pump segments for the (1-1-2-2) pattern. There, the interconnected valve might not seal completely upon contraction and thus extends the pumping segment. However, at a pressure/frequency-configuration of 1.5 bar and 0.5 Hz, the (1–1–2–2) pattern had lower mass flow rates than the (1–2–1–2) pattern. Since all pumping rates were measured fivefold with a corresponding measurement deviation of typically lower than 1% (for flow rates larger 2 g beat\(^{-1}\)), these phenomena are significant. A possible explanation might be
that the interconnecting vein valve now starts to seal more rigorously due to an increased fluid pressure and thereby, prevents any additional fluid exchange during the compression phase (such that segment extension is annulled). M-mode analysis of the interconnected valve then helped to identify that the flap opens more violently when pumping with the (1-1-2-2) pattern than for the (1-2-1-2) pattern (Figure 4.15). This can be explained by the pressure increase resulting from the upper segment due to the simultaneous contraction. Thus, the valve is closed more rigorously and has a delayed opening, which also reduces the pump rate. The compression phase at 0.5 Hz takes 0.5 s (25% fill time per 2 s cycle) and is therefore the longest amongst all frequencies. A higher pneumatic inlet pressure also favors a higher final jacket pressure, thus increasing fluid pressure as well. Nevertheless, the beneficial segment interaction of the simultaneous pattern also suggests that the fluid needs a start-up time to reach higher pump rates.

**Figure 4.15.** M-mode analysis of different pump patterns in a four-segment pump without contrast agent enhancement at 1.5 bar, 0.5 Hz and a pump height of 2m. a) Schematic representation of the interconnected valve and the resolved pixel line is shown. b) M-mode recording for a 1-1-2-2 and c) for a 1-2-1-2 pattern are shown.
To reach longer flow start-up times, we switched to four-segment pumping. Two possible patterns were evaluated. The first pattern used a pulse propagating through the entire four segments of the pump (1–2–3–4), whereas the second pattern should take advantage of pulse overlapping. This leads to the notation (4/1–1/2–2/3–3/4), which is also illustrated in Figure 4.14b. The impact of extended flow start-up times is clearly observable by comparing two-segment and four-segment patterns. We observed much higher flow rates for the same pressure and frequency condition. Further, a drop in mass pump rate was observed for the pulse overlapping pattern (4/1–1/2–2/3–3/4) at elevated pressures, regardless of the pump frequency. This might again be a pump sealing effect, where the interconnected vein valve is closed upon a higher inlet pressure as mentioned earlier. Hence, the pulse propagation pattern without overlapping (1–2–3–4) seems to be the best pattern for pumping. We then tested, if the soft machine was still able to pump upon bending. Our measurement showed that pumping was still possible with less than 10% decrease in efficiency for a bending angle of 26°.

Another interesting observation can be made by comparing pump rates at a given pressure for pattern (1–2–3–4). Before, we would like to emphasize again that pump flow rates are given per actuation cycle with units of g beat⁻¹. This allows a better comparability in terms expulsion effectiveness. At a pneumatic pressure of 1.5 bar and a frequency of 2 Hz, we observed approximately 6 g beat⁻¹. If a linear dependence of the frequency and pump rate should apply, the pump rate per beat could double for the half frequency (1 Hz) at the same pneumatic pressure. However, we observed a much higher rate (19.5 g beat⁻¹). If we further have the frequency, the rate should reach approximately 40 g beat⁻¹ but measured flow rate was much less (29 g beat⁻¹) for a frequency of 0.5 Hz. Therefore, an optimum is expected where pumping frequency meets best elastic material properties of the venous soft pump analogue.

A simple way to increase pneumatic start-up times further is performed by adding a second pressure chamber gas outlet. This allows longer liquid filling time frames without the risk of gas accumulation, causing the pump to collapse (i.e., permanent sealing of pump chamber). We therefore changed the filling and ventilation times to 50% each per pulse interval. Further, pump height was increased to five meters to generate demanding operation conditions (i.e., pumping from a flooded basement). Again, we measured at different frequency/pressure-configurations to screen for an optimal operation parameter set (Figure 4.16, Movie A3.4). Since we are interested in the overall performance, pump rates are now given in g s⁻¹. We observe a
local maximum around a frequency of 1.25 Hz and a pressure of 1.8 bar, yielding a flow rate of 8.75 g s\(^{-1}\). The shape of the frequency plane of 1.25 Hz clearly shows that there seems to be an optimal operation point, where pump actuation and material characteristics best match together.

**Figure 4.16.** (a) Detailed investigation of parameter impact on mass pump flow rate is shown for a frequency range of 0.5–2 Hz and pneumatic pressures of 0.8–2.2 bar for the (1–2–3–4) pump pattern. To increase flow start-up times, two exhausts were used with filling and ventilation times to 50% each per pulse interval. The frequency plain containing the highest mass flow rate is further emphasized with blue points. Only one measurement was taken per frequency/pressure-point due to previously shown very small measurement errors. (b) The pump setting is shown for this parameter investigation. The inset shows the installed four-segment venous soft pump analogue.

With the obtained knowledge, we are able to classify the pump design and compare its properties to already available systems. The presented design entirely separates pump fluid and actuation jacket. Due to this partition, the pump requires no sliding planes, which usually need
extensive care to minimize damage caused by small debris in the fluid (i.e., a filter unit to remove debris in oil pumps). Thus, the mammalian vein-inspired soft pump can be compared with traditional peristaltic pumps. However, the pump has a substantial difference in terms of manufacturing and storing. The injected silicone not only produces lightweight and elastic pumps but also has other consequences based on this material choice. Silicones are known to have high elongation at break, are UV and temperature insensitive (i.e., –20 to 300 °C without loss of elasticity), and are moisture resistant. Therefore, our soft machine was still able to pump after the frozen fluid phase had melted again (Figure 4.17). This could be useful, where low weight and good storage properties are required (i.e., on-site pumps in buildings of possible flooding areas or outdoor pumps, where temperature is likely to drop below 0 °C).

![Frozen water in soft pump](image)

**Figure 4.17.** Completely frozen four-segment soft pump with photo details of the pump reservoir, tube adapter and tubing.

The presented pump design can be further improved, particularly if comparing its weight (2.1 kg for a four-segment pump) and its energy efficiency (1%, see Appendix A3.1 for detailed calculations) to the resulting pump rate (500 mL min\(^{-1}\)) and head (5 m, corresponding to 0.5 bar). Nevertheless, these facts should again be compared with the resulting robustness, the storage properties and the ability of real-time flow analysis within a soft pump design.

### 4.5 Conclusion

The application of the recently presented virtual lost-wax casting technology using RTV soft silicones has opened versatile perspectives toward soft machines and robots with complex
geometries. A key advantage of these machines is that they are essentially made from one single part. This avoids weak spots caused by material discontinuities such as bonding surfaces. In this work, we thoroughly investigated the characteristics of single- and multi-segment mammalian vein-inspired soft pumps with a special focus on real-time analysis of the inner workings regarding fluid-structure analysis. Based on mold stacking, we were able to show successful production of single part pumps with different segment lengths (up to 80 cm).

We managed to operate our pumps both by gas-combustion as well as pneumatically and introduced the concept of in situ observation of such systems by the use of well-established medical tools such as ultrasonic imaging. In order to enhance the observability of flow phenomenon and mechanical operation principles, we modified the used soft silicones with suitable contrast agents (soda lime glass microspheres). The specific pump capabilities are dependent on the mechanical properties of the used soft material as well as external input parameters, such actuation patterns, frequency, and pressurization. Hence, we conducted detailed parameter optimization studies to find ideal operation constraints.

Due to the use of silicone, we can further exploit material properties (i.e., UV and temperature insensitivity, as well as moisture resistance) to identify a potential field of application, where long lasting and light weight properties are required even under low temperature conditions (i.e., on-site pumps or outdoor pumps). Driving such pumps by combustion instead of pressurized air might lead to further improvement due to the higher energy density of hydrocarbons. This would also lead to a direct pumping because the combustion would occur in the pressure jacket, which is adjacent to the fluid chamber.

Our findings point out the potential of structure pervading imaging tools after simple material modification for a deeper understanding of the inner motions of soft machines. Such information was before only accessible by numerically expensive calculations and did not represent the actual motion behavior (i.e., based on boundary values and model simplifications). This helps to shape future optimization strategies and design approaches for an interdisciplinary research field in material science, computational science, and mechanical engineering.
5. Conclusion and Outlook
In this thesis at hand, the production of soft machines manufactured with the presented lost-wax-casting principle is shown using the example of three different devices. The casting process proved its potential to build very versatile, large and complex structures which can even go beyond the available 3D-printing volume (i.e. by mold stacking). This novel manufacturing technique also decreased the necessary part number for machine manufacturing compared to conventional combustion engines. Due to the monoblock design as well as the used silicone rubber with its fascinating properties, these machines could be driven by the combustion of hydrocarbons. Some of the designs could withstand cyclic expansion of over 30’000 repetitions. Thereby, these soft machines clearly demonstrate that traditional torque transmission principles can be questioned and that other ways of transforming energy into motion can be found. Further, these actuators proved themselves to be versatile under extreme conditions (i.e. deformation, freezing). Expansion could be controlled by the incorporation of aramid fiber and resulted in higher pump pressures for the two soft pump designs of up to 1.3 bar. Also, these machines demonstrated that elastomer-based machine parts can be multi-functional. As seen with the example of the soft robot, the silicone part gave structural integrity to the device while hosting the expansion chamber for the jumping motion.

Combustion-driven soft machines have therefore shown their potential to keep up with traditionally engineered machines. All presented machine designs in this thesis were developed to directly fulfill a traditional task such as fluid pumping or locomotion. However, there are machines which can fulfill these tasks with much more output and precision. The many objectives of this thesis was to understand in which areas soft machines can have a significant advantage. An obvious consequence of this elaboration is that soft machines are not going to substitute traditional machines. Soft machines are intended to enlarge the possibilities of an engineer. Therefore, these machines might become a useful tool when low part numbers and soft structures are desired. The next subchapters address arising challenges for soft machines.

5.1 Combustion vs. expansion

Combustion actuation has shown to be very powerful. However, the actual actuation time is limited by the very short combustion interval (i.e. around 15 milliseconds) and the necessary flushing time (i.e. to reestablish a combustible mixture inside the soft machine). In principle, these time frames can be changed by optimizing the gas exchange system in order to allow a
faster reestablishment. Further, the combustible mixture could contain some kind of a thermal damper which allows to transform heat into volumetric expansion. This could help to prolong the actual combustion interval and would increase the low energy efficiency. Also the elastomer itself has shown to have a certain tolerable cycle time so that the material can completely expand and relax. By optimizing the chamber design, this might be changed to a faster possible actuation as well. Nevertheless, combustion is unlikely to replace expansion-driven soft actuators because they cannot hold a deformational state. Vice versa, expansion-driven actuators do not offer the possibility to actuate with high velocity. Therefore soft actuators combining both, combustion and expansion-driven, actuation principles can result in superior performance. This can help to enlarge actuation possibilities for one single soft actuator.

5.2 Elastomer choice and processing

Silicone has many properties which enable the manufacturing of soft actuators. Yet, there are also properties that prevent longer cycle time for combustion-driven soft machines. Due to lower fracture toughness of most silicone rubbers, a soft actuator can rupture immediately upon a single crack. Taking the example of car tires, tire rubber can withstand many rolling cycles over several years. Thus, elastomers have already shown that they can be produced for long-term applications. The smart use of different and interconnected rubber layers accounting for the forthcoming use is the reason why a tire is so robust. Hence, the elastomer choice as well as its processing will be of great importance to improve the lifetime of soft machines, especially combustion-driven actuators.

5.3 Soft implants as an example for potential use

As mentioned before, soft machines can be a useful alternative when a low number of parts and elastomeric machine behavior are desired. One very prominent sector requiring these properties are human implants such as an artificial heart. There, the risk of machine failure is most critical because it can lead to the decease of a patient. In Chapter 1 the risk of machine failure was, amongst others, assigned to the number of involved machine parts. With the technology of soft machines, actuators consisting of few parts can be built and therefore account for the inherent safety of a device. Also the elastomeric material has a beneficial impact on its
incorporation into a human body. Human beings have the urge to move. Therefore, an implant adapting to the movement of its patient would result in less impairment.

The field of soft machines has grown rapidly during the last decade and is certainly driven by the desire to mimic nature in more detail. Nevertheless, as the outlook has shown, the potential of this field has not been completely reached. This is why more research in this area is necessary in order to grasp the full potential of this emerging field.
Appendix A: Supplementary material
A.1 Supplementary data for chapter 2

A.1.1 Calculation of upper and lower explosion limit

We calculated the upper (UEL) and lower explosion limit (LEL) using the temperature function presented by Gieras et al. for methane in air as follows:77

\[
UEL(\%) = 0.0108 \cdot T(K) + 12.4 \quad \text{(eq A1.1)}
\]

\[
LEL(\%) = -0.00436 \cdot T(K) + 5.86 \quad \text{(eq A1.2)}
\]

The resulting UEL and LEL are then given as a methane molar fraction in air (%). Due to the subsequent flushing of the explosion chamber with combustible gas mixture, we assumed that ignition occurs around the inlet temperature of the gas mixture (at room temperature). Hence, the limits are calculated to be 4.6 mol % for the LEL and 15.6 mol % for the UEL at a temperature of 300 K. Methane volumetric fraction (vol %) and the volumetric air to methane ratio (Vair/VCH4) were then calculated of each limit using the molecular weights for methane and air, as well as their densities (0.66 kg/m³ for methane and 1.20 kg/m³ for air) at 298 K.

A.1.2 Calculation of upper and lower explosion limit

We calculated the energy efficiency according to the mentioned formula in the article:

\[
EE = \frac{Q_{pot}}{Q_{comb}} = \frac{m_{\text{pump}} \cdot g \cdot h_{\text{pump}}}{m_{\text{CH}_4} \cdot \Delta H_{\text{comb}} \cdot f} \quad \text{[J/s][J/s]} \quad \text{(eq A1.3)}
\]

\(Q_{pot}\) was calculated by using the obtained, already shown data. To get \(Q_{comb}\), calibration data was needed. Therefore, we used online mass spectrometry (MS) tracking data for an ignition frequency of 0.5 Hz, a methane flow rate of 0.5 L/min and an air-methane ratio of 10:1. The total ion count (TIC) per sample cycle (j) was calculated by summing up all ion counts (IC) for every component (i) per cycle:

\[
TIC_{ij} = \sum_{i} IC_{i} \quad \forall i = \text{CH}_4, \text{N}_2, \ldots \quad \text{(eq A1.4)}
\]

Then, the IC’s of all compounds were normalized using the corresponding TIC’s. This data was then considered to be the mole fraction of each component. Now, we used the data region right before our first ignition for calibration purpose. There, the feed flow was able to reach its steady state concentration. We then averaged each component’s mole fraction within a time region of
roughly 50 seconds right before the first ignition. Therefore, we obtained the following calibration data, as shown in Table A1.1.

**Table A1.1. Calibration data, obtained by MS tracking, used mole weights and used densities.**

<table>
<thead>
<tr>
<th>Component</th>
<th>xᵢ</th>
<th>MWᵢ g/mol</th>
<th>ρ kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₄</td>
<td>0.1338</td>
<td>16.04</td>
<td>0.66</td>
</tr>
<tr>
<td>O₂</td>
<td>0.1698</td>
<td>32</td>
<td></td>
</tr>
<tr>
<td>CO₂</td>
<td>0.0006</td>
<td>44.01</td>
<td></td>
</tr>
<tr>
<td>H₂O</td>
<td>0.0077</td>
<td>18.02</td>
<td>1.20</td>
</tr>
<tr>
<td>N₂</td>
<td>0.6881</td>
<td>28.02</td>
<td></td>
</tr>
</tbody>
</table>

Using the mole fractions, we calculated a weighted average for the feed mixture density and for the molecular weight. Then, we used these values to calculate the corresponding amount of moles associated to the feed mixture inside the combustion chamber of 94 cm³:

\[
n_{\text{CH}_4} = x_{\text{CH}_4} \cdot n_{\text{tot}} = \frac{x_{\text{CH}_4} V_{\text{comb}} \rho_{\text{av}}}{\text{MW}_{\text{av}}} \approx 526 \mu\text{mol} \quad \text{(eq A1.5)}
\]

Multiplying the value obtained in (3) with the molar enthalpy of combustion of methane and the ignition frequency, we then get the power of the soft pump:

\[
P = n_{\text{CH}_4} \cdot \Delta H_{\text{comb}} \cdot f = 526 \mu\text{mol} \cdot 890.7 \frac{kJ}{\text{mol}} \cdot 0.5 \text{ s}^{-1} \approx 234 \text{ W} \quad \text{(eq A1.6)}
\]
A.1.3 Movies

Movie A1.1. Different stretching strengths of the outer wall.

Movie A1.2. Video analysis of the thin walled soft pump after 30’000 combustion cycles.

Movie A1.3. Recording of the extreme scenario with pumping to a height of 13 meters.
A.2 Supplementary data for chapter 3

Movie A2.1. Reorientation with roly-poly geometry. This video shows the reorientation of the soft robot after several combustion events. The robot gets back into an upright position and therefore shows the expected roly-poly behavior.

Movie A2.2. Roly-poly equilibrium stop. This video shows a weak spot of the soft robot due to the non-optimal bowl geometry. After the combustion event, the soft robot lands on its side in such a way that no reorientation is possible anymore. The video shows also an inset from the second camera inside the robot.
Movie A2.3. Jump behavior and material stress. This video focuses on the jump event itself. A close-up shows also the repelling force developed by the combustion event in slow motion. Then the robot is turned upside down to demonstrate the stress imposed on the material during such a combustion event.
A.3 Supplementary data for chapter 4

A.3.1 Calculation of Pump Energy and Pump

The simplest way to get a rough estimation of the resulting pump energy is achieved by considering the actual amount of water, which is displaced by a jacket squeezing step. We therefore calculated the pump energy as follows,

\[ E_{pump} = p \cdot dV = p \cdot \frac{dm}{\rho} = p \cdot \frac{m}{\rho} \cdot f \]  

(eq A3.1)

where \( p \) represents the applied pressure in [Pa], \( m \) the mass flow in [kg/s], \( \rho \) the fluid density in [kg/m\(^3\)] and \( f \) the pump frequency in [1/s]. We calculated the pump energy to be 2 J for a pressure of 1.8 bar, a frequency of 1.25 Hz and a pump rate of 8.75 g/s and a density of 1000 [kg/m\(^3\)].

To calculate actual energy efficiency, we need an estimation of the energy put in to our system. We calculated the energy input similar to Equation A3.1, but this time, the volume difference was obtained by measuring the volume of the exhausted gas (i.e. gas spilled out after exhaust valve opening within a single pump cycle):

\[ E_{in} = p \cdot dV = p \cdot V_{exh} \]  

(eq A3.2)

where \( V_{exh} \) is the exhausted gas volume [m\(^3\)]. We calculated the input energy to be 200 J for a pressure of 1.8 bar and a measured exhausted volume of 1.1 L. The energy efficiency is then calculated by the ratio of these values:

\[ EE = \frac{E_{pump}}{E_{in}} = 0.01 = 1\% \]  

(eq A3.3)
A.3.2 Movies

Movie A3.1. Sonograms of the material test valves are shown with and without contrast agent incorporation. Modified silicone shows increased backscattering within silicone walls compared to unmodified silicones.

Movie A3.2. CDI recordings are shown for different squeezing velocities of the material test valves.
**Movie A3.3.** CDI recordings of different fluids are shown to illustrate density and viscosity impacts on ultrasonic imaging. Pumped fluids are water, silicone oil, ketchup and a starch-water mixture.

**Movie A3.4.** Four segmented soft pump is shown from the inside. Squeezing mechanism is also recorded from the inside. Last, the four segmented pump pumps up to 5 m with a frequency of 6 Hz, using pattern (1-2-3-4).
References


70. Ceyzeriat, L. Composition organopolysiloxaniques vulcanisables. *FR 1,198,749*, **1958**.


