Acta Polytechnica CTU Proceedings **25**:83–88, 2019
doi:10.14311/APP.2019.25.0083
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FEASIBILITY STUDY OF MICRO-LATTICE STRUCTURES BY MULTIPHOTON LITHOGRAPHY

Markus Wimmer*, Zoltan Major

Johannes Kepler University Linz, Institute of Polymer Product Engineering, Altenberger Strasse 69, 4040 Linz, Austria

* corresponding author: markus.wimmer@outlook.at

Abstract. The paper describes the possibilities of additive manufacturing with multiphoton lithography. The basis of this technology is that a laser beam (with a certain wavelength) is fired into the mixture of a monomer and a photo-initiator. When the energy of the laser is high enough, the latter acts as a catalyser for the polymerization of the monomer compound. This study focuses on the influences of certain parameters of the multiphoton lithography process. One of the important aspects is the choice of the solvent for the post processing. In sequence to the solvent problem, the influence of the layer height is examined. Furthermore the limits and possibilities of the setup in use are investigated. As an example the differences in fabrication with the laser firing with "constant frequency" and "constant density" were subject of this investigation. The second goal of the study was to compare three different structures consisting of periodically repeating elements, scaled in size and number of elements per side.

Keywords: Multiphoton lithography, lattice structures.

1. Introduction

The technology used for this research is "femtosecond laser multiphoton polymerization". It is an auspicious technology that can be used for the fabrication of micro structures in fields like bio medicine (cellgrowing), or photonics (photonics crystals) [1]. To generate a 3D nano structure with this system, a mixture of a photo initiator and a monomer is required. A laser with a certain wavelength (in this case 1028nm) is firing in this mixture and the energy inserted cracks open the photo initiator chains. The so formed radicals start the process of polymerization by cross linking the surrounding monomer. This effect occurs locally around the focus point of the laser.

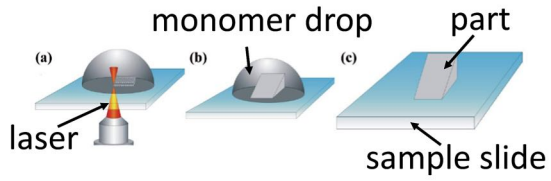

Figure 1. Schematic: multiphoton lithography [2]

To move from a dot to a defined 3D structure, the desired geometry has to be prepared by a slicer. The slicer disassembles the model into a number of slices with constant height. A smaller height results in a better resolution.

The downside of small layer heights is, that the production time is proportional to the height. Half the layer-thickness results in a twice as long production time. Another problem could be that the contact area between the layers might be too small which causes the mechanical properties to suffer, or in the worst case, the structure collapses. The sliced model is than written by the laser. Therefore it moves along the area of each slice and polymerizes the material around. This continues for each from the slicer prepared layers. All the layers combined give the part its shape (Figure 1 (a)). The so created model is still covered in unpolymerized monomer(Figure 1 (b)). To get rid of the monomer drop around it, the probe has to get developed. This means, that the sample slide gets washed with ethanol or acetone. Due to this, the unused monomer gets washed away from the polymerized structure and the written part stays on the sample-slide (Figure 1(c)). There is a variety of resins available to use for this process. There is a variety of resins (consisting of a photoinitiator and a monomer) available to use for this process. Some examples are shown in Figure 2. The monomer in use for this investigation is PETA.

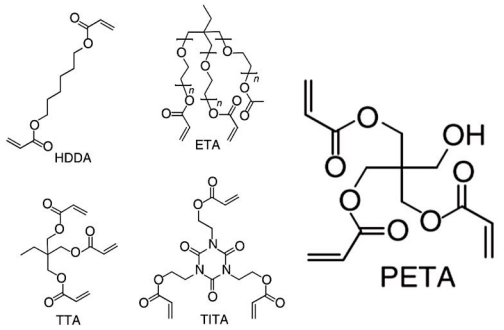

Figure 2. Common Monomers [3]

2. Examined influences

The following images were taken on the scanning electron microscope "Philips SEM-525M". The samples are prepared by sputtering (applying a thin layer of gold dust to the structures to ensure a conductive connection between probe and stage of the microscope). The sputter coater in use is the model "SCD 005 Cool Sputter Coater" from BAL-TEC.

2.1. Solvents

The first aspect to analyze is the influence of the solvent used for the development process. Ethanol and acetone are the two options. The structures designed consisted of 4 separate clusters with 5 micro cells in each direction in plane and one micro cell in height (structures were designed by Kerstin Lehner(JKU) in her master thesis). Four of those clusters are stitched together to generate the structure shown here.
As can be seen in Figure 3, the structure developed with ethanol suffered heavy damage during the developing process. Therefore the following samples were all developed with acetone.

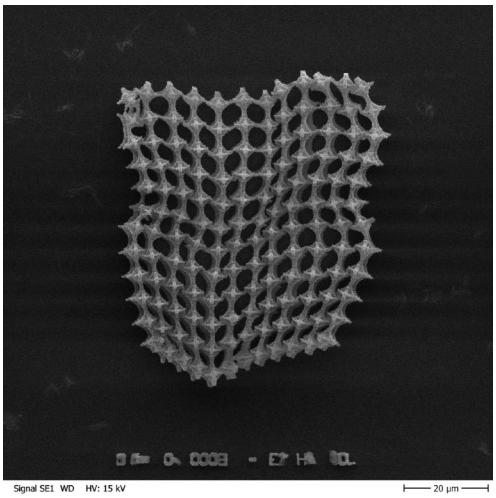

Figure 3. Structure developed with ethanol

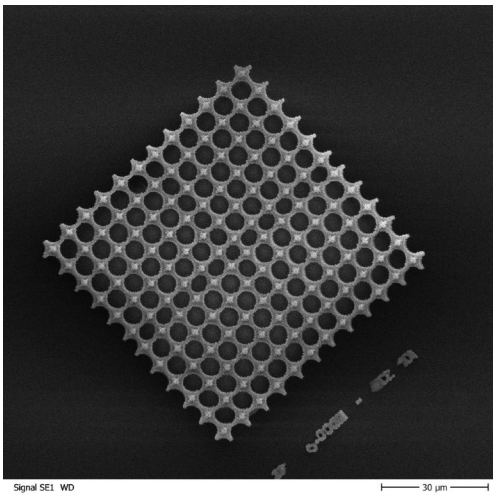

Figure 4. Structure developed with acetone

2.2. Layer height

As in most additive manufacturing processes, the layer height is expected to have a major influence on the results of the samples as well as on the fabrication time. A small layer height leads to a more defined structure normal to the sample slide (Z - direction). The fabrication time is directly influenced by the layer height. Therefore a twice as large layer height results in half the time needed for the production.

The samples displayed in Figures 5 & 6 show the expected impact of an increased layer height. The structure gets bulkier and has a reduced grade of details.

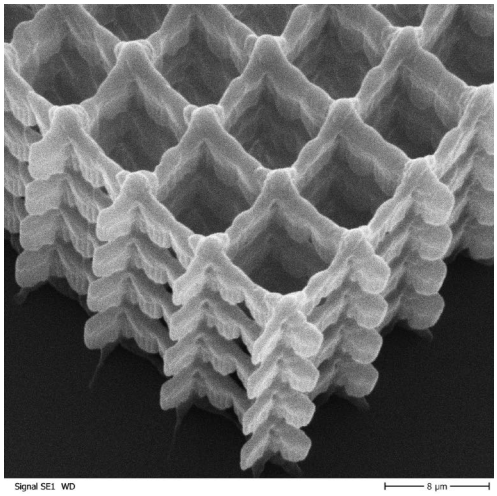

Figure 5. Structure with 90nm layer height

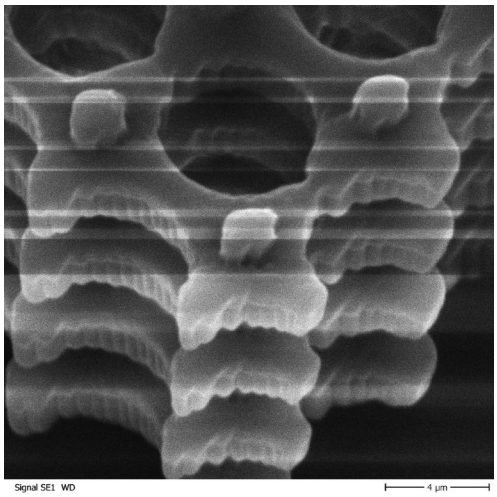

Figure 6. Structure with 200nm layer height

2.3. Constant Density

The following samples are not written with the standard "constant frequency" mode but instead with "constant density". The theory behind this is, that with a constant frequency, the pulse of the laser is a sole function of the time, not considering the acceleration

of the stage or galvoscanner. Therefore the distance between the impacts of the photons varies depending on the speed of the galvoscanner and the stage. The galvoscanner (Figure 7) is basically a fast moving mirror that directs the laser with a multiple of the stages velocity and acceleration but with a limited field of view (operating area).

FIGURE 7. Galvoscanner [4]

In the "constant density" mode or PSO (Position Synchronized Output) the pulse is not dependent on the time, but on the position of the laser. Therefore the frequency increases with the speed of the stage or galvoscanner and guarantees a constant distance between the pulses, even in time of acceleration. To improve the stability of the structure, there is also the option to use a "pulse burst". That means that every time the laser gets triggered, multiple pulses are fired. Those are still dependent on the position. This should prevent an overlapping of pulses during low speeds. Overlapping can lead to so called "micro explosions" which can damage the surrounding structures.[5]

With the increase of the stage- or galvospeed, the pulse density has to be reduced in order to stay beneath the maximum possible frequency of 920 kHz which is the maximum the machine is capable of.

The results of the sample look very disappointing. Instead of an improvement due to the constant allocation of the pulses, the structure seems to be consisting of horizontal rods with barely any cohesion at all. The unusual shape of the layers might be connected to the pulse burst in use. Those two in rapid frequency fired pulses might result in those patterns. The fact that they are all oriented in the same direction supports this theory.

2.4. MAXIMISING STRUCTURE SIZE

Another point of interest was to Figure out which fabrication size is possible in a reasonable time. Due to extrapolating of the writing time of smaller parts, a 35x35x35 micro cell large cube is the maximum that is possible in a time frame of 12 hours. This represents a side length of 0.28mm, Therefore, the layer height is increased to the maximum that was tested so far (800nm).

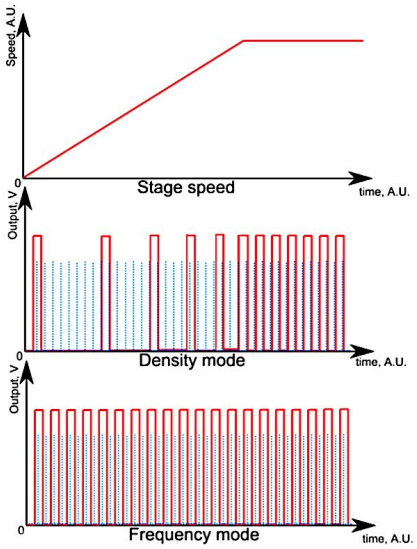

FIGURE 8. Density and Frequency Graph (A.U. stands for arbitrary unit)[5]

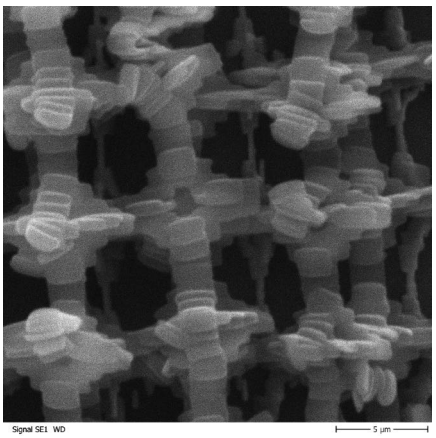

FIGURE 9. Constant density

The galvoscanner is used in the way that the stage positioned in the center of a 5x5 cluster and the galvoscanner writes the micro cells. As a result of the faster acceleration and velocity limit of the galvoscanner, this reduces the production time per cluster to 1/15 in comparison to stage-only writing. Due to the limited operating radius of the galvoscanner, the stage has to re-position after each cluster to the center of the next one.

The structure, as seen in Figures 10 to 13, got only slight geometric shortcomings. As seen in Figure 12 ,there is a stitching error between the 5x5 clusters. Another anomaly are the shining white spots between some of the micro cells. A possible explanation for the existence of these spots is that there is unpolymerized PETA stuck in the holes between the structures. The radiation from the electron microscope might have stimulated it and that is the reason for the glowing. In Figure 13 there is also a piece of dust that got into the sample and now sticks on the side.

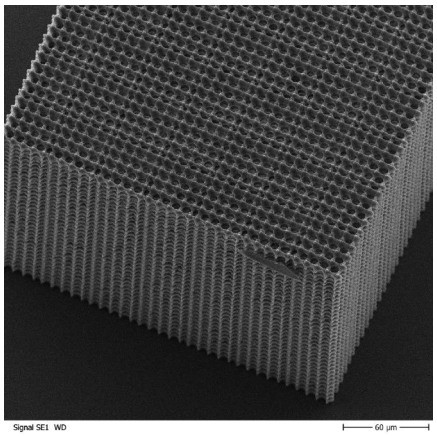

Figure 10. Structure with 35x35x35 microcells overview (0.28mm side length)

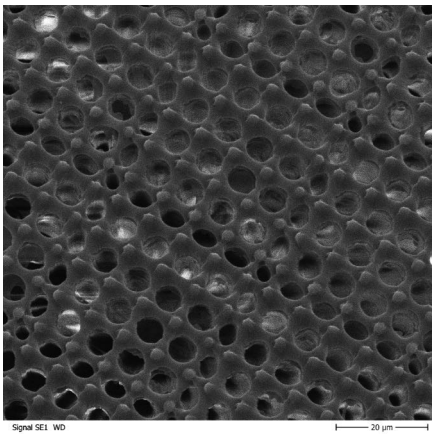

Figure 12. Structure with 35x35x35 micro cells, Detail 1

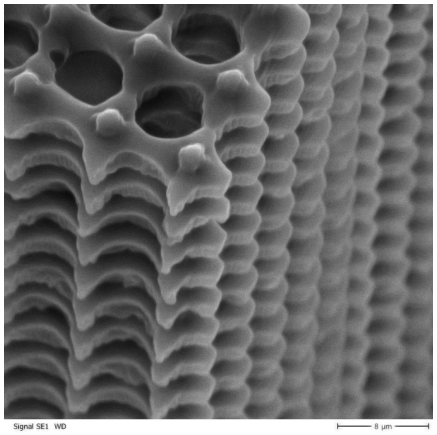

Figure 11. Structure with 35x35x35 microcells closeup (0.28mm side length)

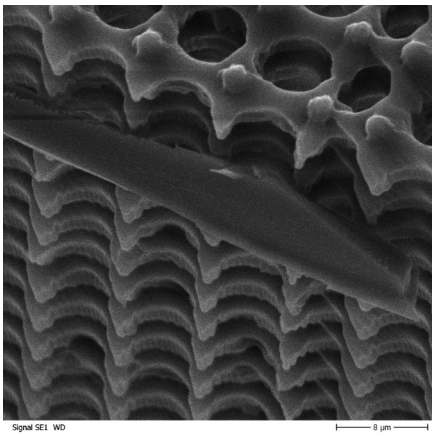

Figure 13. Structure with 35x35x35 micro cells, Detail 2

2.5. Variation of Micro Cell Structures

In this part of the research, three different micro-cells (Figure 14) are compared in two different ways.

2.5.1. quantity scaling

At first the elements got scaled in number with a ratio of 1:2:4. This ratio results 5, 10 and 20 micro cells per side.

The inspection of the results shows the already known stitching problem. The most plausible reason behind this is a slight inaccuracy in the stage and/or galvoscanner movement. That means that in the medium and large construct, every five micro cells there is less distance to the next micro cell than there should be.

Next in line is the octet structure. While the external dimensions still remain a cube, the interior increases in complexity. The standard size of a micro cell of this type is 10 microns and a cluster consists of 3x3 micro cells. Therefore, the side lengths vary from 30 microns to 120 microns which represents three, six and twelve microns cells per side. As there can be seen in Figure 15, the base structure is not even remotely preserved. The structures with more elements show the same results.

The last structure in comparison is the hex structure (Figure 16). The characteristic of this one is, that it got different dimensions in length and width. Therefore, the base area of the result this time is not a square. In this iteration the dimensions of one micro cell are 15 microns x 17.32 microns. A cluster consists of three of those micro cells in each direction. The overview Figure 16 show a decent quality regarding the shape and contour.

The closeup on the medium structure in Figure 17 visualizes that there is a problem with the connections within the structure. This error is reproducible and seems to be limited to big structures. Therefore it is evident that this is in connection to a software issue.

2.5.2. Dimension scaling

In the second part of the scaling process, the side lengths of the micro cells get changed. The ratio remains the same with 1:2:4. The resulting dimensions therefore are 4, 8 and 16 microns per side for the hex structure. For the octet structure the resulting side length are 5, 10 and 20 microns. The hex structure results in micro cell dimensions of 7.5 microns x 8.66 microns up to 30 microns x 34.64 microns. The structure in Figure 18 is the smallest of the three. Even

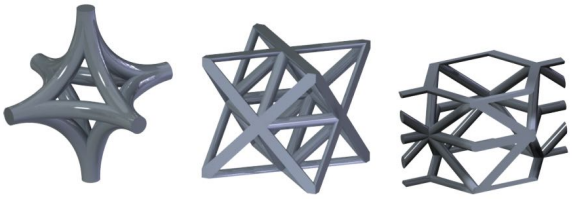

FIGURE 14. Lattice structure base elements: nonlinear thrust structure, octet structuer, hex structure (left to right)

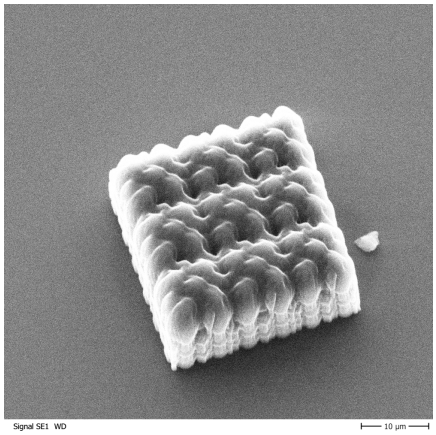

FIGURE 15. Quantity scale: octet structure

though the details are noticeably worse, the result is acceptable in respect to the dimensions.
Reasons for this might be that the microscope can not deliver a better resolution in this dimension or the writing accuracy is at its limit. But even through the blur the basic shape of the micro cell seems to be preserved. Furthermore the structure did not collapse and still shows the significant holes.

The samples of the octet structure show the same result as the one with the nonlinear thrust structure. The grade of detail of the small structure makes it blurry and unrecognisable (similar to Figure 15 that's twice the size of the structure in question).

Scaled to 16 microns per element the details of the pattern increases drastically (Figure 19). The glowing in the middle indicates, that there is still unpolymerized monomer inside of the structure that was not removed during the developing process.

The first impression of the hex results is quite satisfying. All of the structures still got recognizable micro cell shapes and none of them collapsed. The smallest one (Figure 20) shows the already known resolution boundary of the laser with the used settings and in consequence got quite bulky.

While the medium sized construct does not show any anomalies, the big structure with 30 microns x 34.64 microns element size shows the already known stitching problem. A close look reveals a line between the three clusters alongside one axis (bottom left to top right).

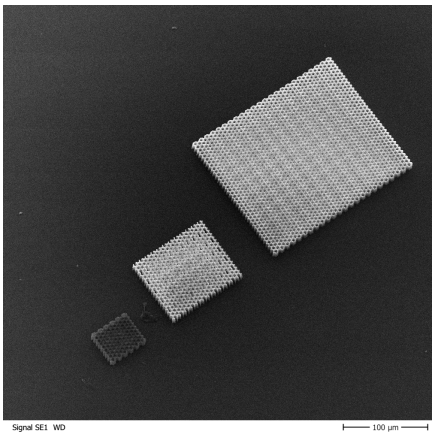

FIGURE 16. Quantity scale: hex structure

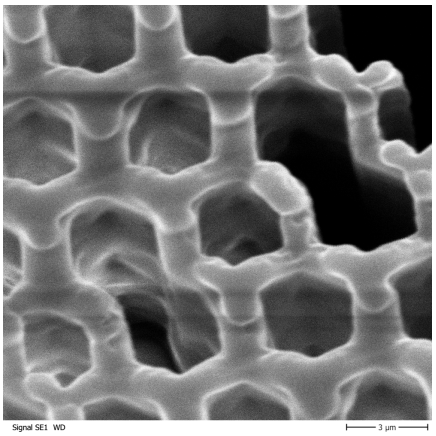

FIGURE 17. Quantity scale: hex structure: medium detail

3. Conclusion

This paper investigates the influences of manufacturing parameters on multiphoton lithography. The parameters in focus are the solvent and the height of the layers. The effect of ethanol (Figure 3) and acetone (Figure 4) as developing fluid demonstrates that acetone is the superior choice for this application. The influence of the layer height behaves similarly like in other additive manufacturing methods. Small layer heights result in a more detailed geometry with the drawback of long fabrication times (Figure 5). High layers on the other hand decrease the time needed for production but the structure gets more bulky (Figure 6). In order to produce reasonably fast and big structures, layer heights in the scale of 800 nm turned out to be ideal. For more complex structures a lower layer height is appropriate.
The tests with the "constant density" render this option useless. But the theory behind this method is promising and might turn out important for the future.
For the future I expect this technology to get essential for the field of bio medicine. There it could be used to generate the environment for cell growing.

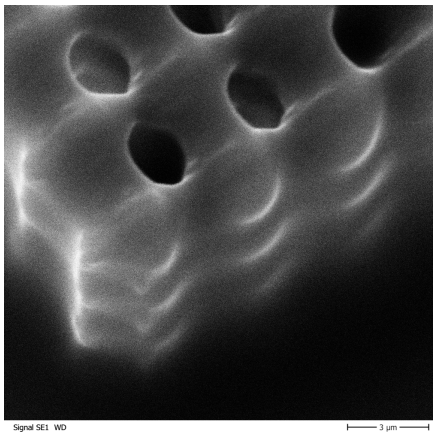

Figure 18. Dimension scale: nonlinear thrust structure (4 microns element size)

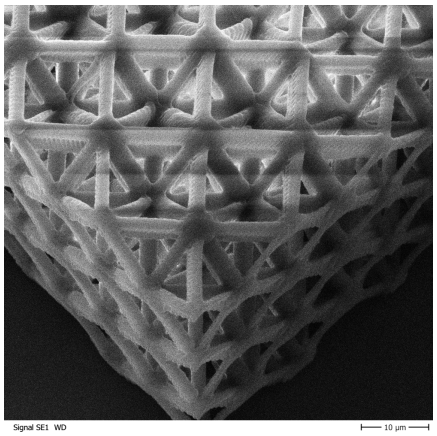

Figure 19. Dimension scale: octet structure (16 microns element size)

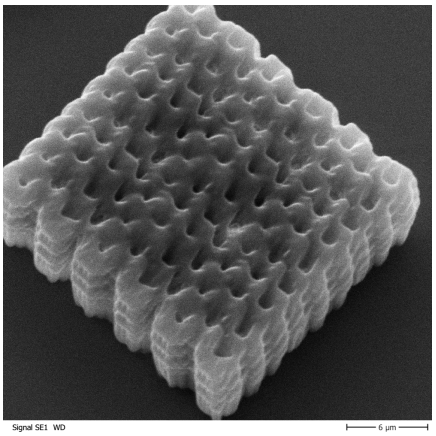

Figure 20. Dimension scale: hex structure (7.5 microns x 8.66 microns side length per element)

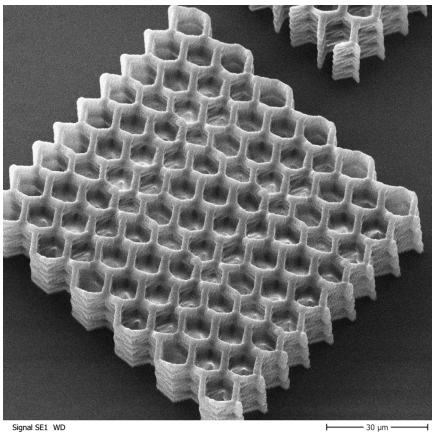

Figure 21. Dimension scale: hex structure (30 microns x 34.64 microns side length per element)

Acknowledgements

This work was supported by FH-Prof. DI Dr. Jaroslaw Jacak (Upper Austrian University of Applied Sciences), Boris Buchroithner (Institute of Applied Physics - Johannes Kepler University) and Valdemaras Juzumas (Workshop of Photonics). A special thanks to Univ.-Prof. DI Dr. Werner Baumgartner from the Institute of Biomedical Mechatronic on the Johannes Kepler University for providing the SEM.

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