Influence of steam sterilization and raster angle on the deflection of 3D printing shapes
(Rapid communication)

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Abstract: The study examined influence of steam sterilization and the raster angle (0°, 90°) on the deflection of polymer shapes obtained by 3D printing. In the case of PEEK and PLA, the FDM/FFF method was used, in the case of photocurable resin, DLP technology, and in the case of MED610, PolyJet technology. It was shown that the raster angle, type of material and sterilization have a significant impact on the strength and deformation of the tested polymers. A model of a facial bone implant was also developed and the suitability of the tested materials for obtaining this type of implants was examined. The dimensional accuracy of the implant models was highest for the MED610 model. However, due to the significant deflection of this polymer under load, its use is only possible in areas of low stress. In the case of UV resin printed in the vertical direction (90°), the samples showed the least deflection and the printed model had no visible defects. The greatest deformations occurred at the ends and narrowing's of the model.

Keywords: 3D printing, implants, sterilization, deflection.

Wpływ sterylizacji parą wodną i kąta rastra na ugięcie kształtek otrzymanych metodą druku 3D
(Komunikat szybkiego druku)

Streszczenie: W pracy zbadano wpływ sterylizacji parą wodną oraz kąta rastra (0°, 90°) na ugięcie kształtek polimerowych uzyskanych metodą druku 3D. W przypadku PEEK i PLA zastosowano metodę FDM/FFF, dla żywicy fotoutwardzalnej technologię DLP, a MED610 technologię PolyJet. Wykazano, że kąt rastra, rodzaj materiału oraz sterylizacja mają istotny wpływ na wytrzymałość i odkształcenie badanych polimerów. Opracowano także model implantu kostnego twarzoczaszki i zbadano przydatność badanych materiałów do otrzymywania tego typu implantów. Dokładność wymiarowa modeli implantów była najwyższa dla modelu MED610. Jednak ze względu na znaczne ugięcie tego polimeru pod obciążeniem jego zastosowanie jest możliwe jedynie w obszarach o małych naprężeniach. W przypadku żywicy UV drukowanej w kierunku pionowym (90°) próbki wykazywały najmniejsze ugięcie, a otrzymany model nie miał widocznych wad. Największe odkształcenia wystąpiły na końcach i zwężeniach modelu.

Słowa kluczowe: druk 3D, implanty, sterylizacja, ugięcie.

Polymer materials used in medical applications must not only be characterized by appropriate mechanical and physicochemical properties but must also properly and safely meet the biocompatibility criteria. Before use, all medical devices undergo a decontamination process. Despite the continuous development of materials engi-

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neering, metal materials are still the most frequently used materials for implants. Nowadays, polymeric materials are becoming increasingly popular in the field of medical engineering, and with the dynamic development of additive technology, increased attention is being paid to research on the mechanical strength of composite polymer structures. The emergence of new polymer materials allows for more accurate reproduction of desired medical structures, reducing costs and at the same time accelerating the processes of their creation. Understanding the relationship between printing parameters and the mechanical strength of the medical materials used will allow for the optimization of the manufacturing and design processes of components [1–4], as well as for the improvement of materials used in 3D printing [5–9].

The research conducted by the authors was created in response to the growing interest in the use of polymer materials in medical engineering and was an attempt to fill the gap in the previous scientific research which did not sufficiently focus on assessing the impact of the medical sterilization process on these materials. Most authors focus on the impact of the sterilization process on the geometric form and its maintenance after this process [2, 5].

The objective of the research was to study the effect of steam sterilization and raster angle on the deflection of 3D printing shapes obtained from PEEK, MED610, PLA and UV resin. This serves as a prelude to expanded research in this area.

EXPERIMENTAL PART

The research involved examining the deflection of polymer samples obtained using selected additive techniques and analyzing them in the aspect of medical sterilization. The tests conducted on the deflection during simple bending at a constant stress of 5 MPa were aimed at assessing the durability and load resistance of polymeric materials subjected to the sterilization process.

Materials

The shapes for bench tests were made using additive technology from polyether ether ketone (PEEK) (3DGence, Dallas, Texas, USA), MED610 (Rechovot, Israel), poly(lactic acid) (PLA) (Gliwice, Poland) and photocurable resin (UV resin, Anycubic, Shenzhen, China). The analyzed materials, classified as medical, are subject to high durability requirements and lofty standards of quality, safety, and biocompatibility [2, 5]. Table 1 presents selected properties of the used materials.

T a b l e 1. Selected mechanical properties of polymeric materials

Property	PEEK	MED610	UV resin	PLA
Tensile strength, MPa	105	50–65	36–45	47
Flexural strength, MPa	130	75–110	50–65	64
Young's modulus, MPa	4100	2200–3200	1200–1600	2600
Elongation at break, %	30	10–25	8–12	19

3D printing of samples

The samples were printed from four different materials, using four different manufacturing techniques, with different settings for each: PLA (Noctuo - UltraPLA, Gliwice, Poland) on a Prusa i3 MK3 printer (Prague, Czech Republic) using the FDM/FFF method, photocurable resin (Anycubic, Shenzhen, China) on a Photon printer (Hong Kong, China) in DLP technology, MED610 (Stratasys, Rechovot, Israel) in PolyJet technology using Object Eden260V (Stratasys, Rechovot, Israel), and PEEK on a 3DGence Industry F340 printer (Dallas, Texas, USA), which uses FDM/FFF technology, but in an actively heated printing chamber.

Sterilization process

After rinsing with water, the samples were disinfected with Medisept Viruton Bohr liquid (Medisept, Lubin, Poland), rinsed again in water, and dried on paper. The cleaned samples were separately packed into paper and foil bags with a type 4 indicator and an external type 1 control indicator attached, sealed, and placed in an Is Yeson E 12L Black class B autoclave (Is Yeson, Łódź, Poland) (Fig. 1). The parameters of saturated water vapor

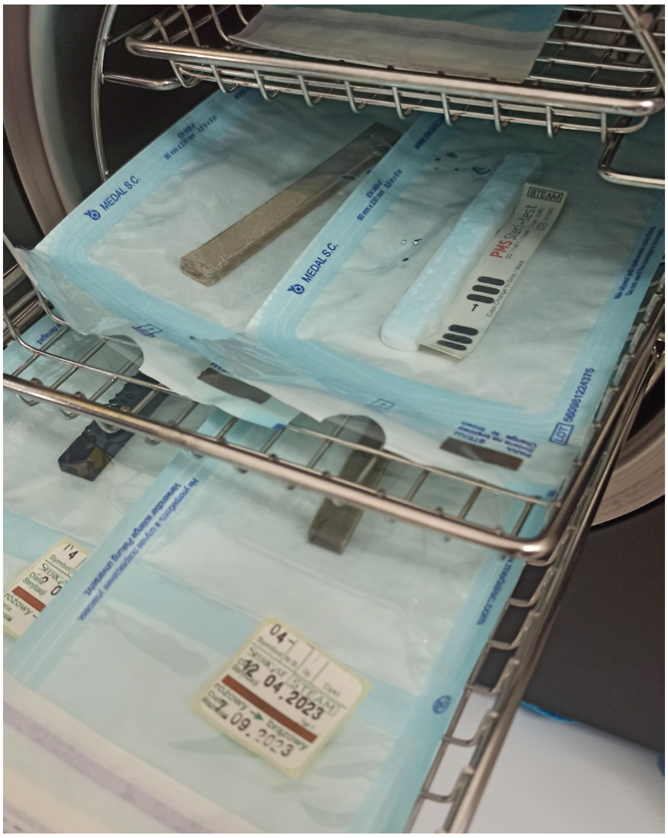

Fig. 1. Autoclave chamber containing packed and sterilized samples

were controlled, i.e., pressure (max. 116 MPa), temperature (121°C) and time (15 min).

Deflection during bending

To measure the deflection during bending, an automated force measuring stand, MultiTest-dV 2.5, from Mecmesin (Slinfold, West Sussex, United Kingdom) was used (Fig. 2). The samples with dimensions of 4 × 10 × 100 mm were subjected to a constant stress of 5 MPa for 2 months by placing them on a specially constructed station providing support points at two opposite ends of the beams and free space for hanging weights. The ambient temperature during the process was 22 ± 0.5°C and the humidity was 50 ± 5%. The value of the elastic deformation occurring under the influence of a given force exerted by the bending moment was measured and is the distance between the deformed and undeformed axis of the beam. Theoretically, the deflection arrow determines the value of the maximum absolute deflection leading to the breaking of material connections [6–9]. The research considered two printing directions and decontamination or lack thereof for each polymer material. Descriptions placed above individual beams inform about the orientation of the printout relative to the machine axis, where ⊥ means vertical position and ǁ means horizontal position.

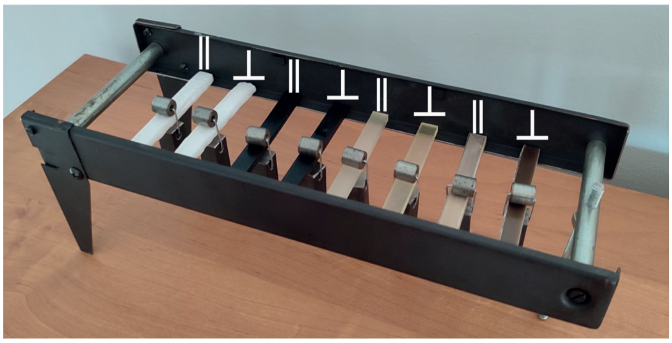

Fig. 2. Flexural test

Model of cranial-facial bone implants

A cranial-facial bone implant model was performed by geometric and dimensional analysis using the non-contact optical system Atos Triple Scan II (Carl Zeiss, Oberkochen, Germany). A detailed analysis of the measurements was performed using the ZEISS Quality Suite application (Carl Zeiss, Oberkochen, Germany).

RESULTS AND DISCUSSION

Deflection during bending

Table 2 shows the deflection arrow of unsterilized and sterilized samples depending on the material used. In the case of PEEK (horizontal printing), the deflection arrow before sterilization was 1.01 mm and after sterilization 0.74 mm. This may indicate that the print sample has become stronger after decontamination of this material. However, in the case of vertical printing, the deflection arrow before sterilization was 0.26 mm and after sterilization 0.82 mm, which suggests the opposite effect of sterilization compared to horizontal printing.

MED610 samples printed in the vertical direction, both unsterilized and sterilized, had a larger deflection arrow compared to samples printed in the horizontal direction. Without sterilization, between the two types of sample arrangement on the printer table, the deformation was twice as large, and after sterilization it was even four times greater. This indicates a visible relationship between the load resistance of MED610 and the direction of 3D printing relative to the machine axis. Due to excessive sterilization deformation, PLA samples could not be evaluated, so the relationship between sterilization and the deflection arrow could not be thoroughly analyzed. However, it can be concluded that PLA showed the greatest load resistance without sterilization in both directions. Unsterilized samples of UV photocurable resins were deformed in both directions by 0.29–0.28 mm. For samples printed in the horizontal direction, sterilization resulted in a larger deformation of 0.69 mm, which may suggest a loss of high load strength after decontamination. The sample, which was also sterilized but printed in a vertical direction, did not show any major deviations compared to the unsterilized sample.

T a b l e 2. Effect of sterilization on deflection arrow

Material	Deflection arrow, mm			
	Raster angle 0°		Raster angle 90°	
	Unsterilized	Sterilized	Unsterilized	Sterilized
PEEK	1.01	0.74	0.26	0.82
MED610	1.02	0.66	2.43	2.04
PLA	0.15	–	0.14	–
UV resin	0.29	0.69	0.28	0.22

Analysis of facial implants model

Cranioplasty is a frequently performed neurosurgical procedure that allows the repair and reconstruction of skull defects resulting from various causes. This is achieved using cranial prostheses made of materials such as titanium, autologous bone, ceramics, and polymers. The production of prostheses is often expensive and requires complex intraoperative processes. Individual fabrication of implants using 3D-CAD methods allows for precise and anatomical reconstruction in a shorter operating time compared to other conventional techniques [10–14].

The main goal of the research was to provide valuable information regarding the durability and load resistance of the materials used, as well as the appropriateness of their use in the developed facial implants (Fig. 3).

The model obtained in the 3D-CAD modeling process was compared with the reference model created at the

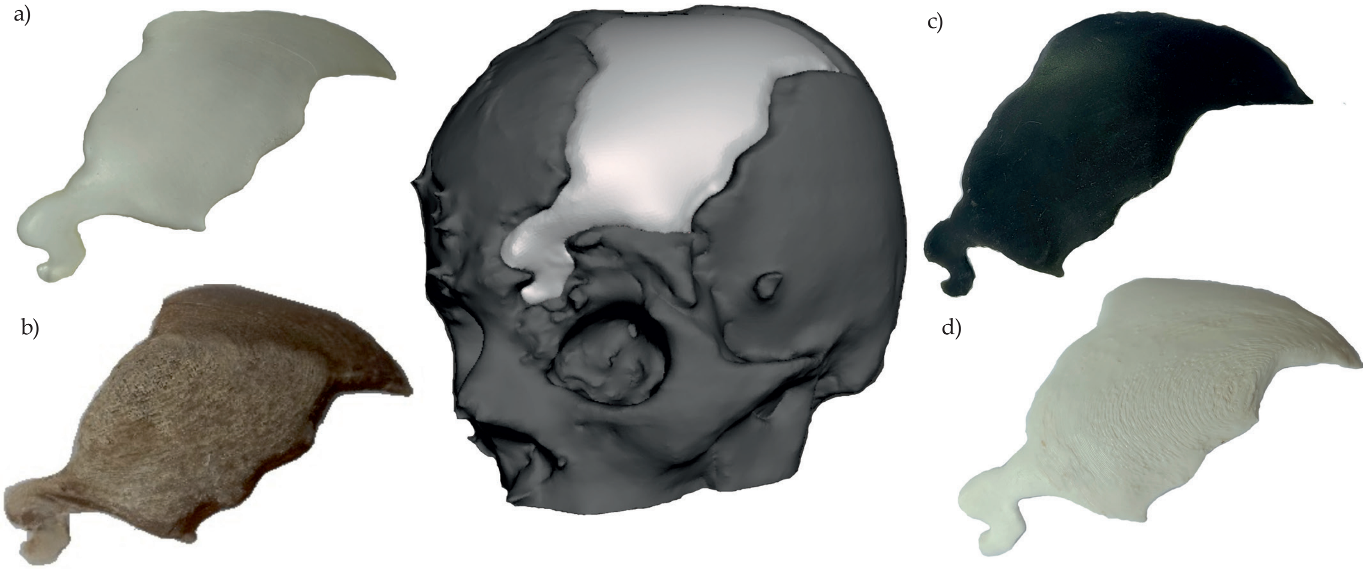

Fig. 3. 3D-CAD model of a facial defect and the implants obtained from: a) MED610, b) PEEK, c) UV resin, d) PLA

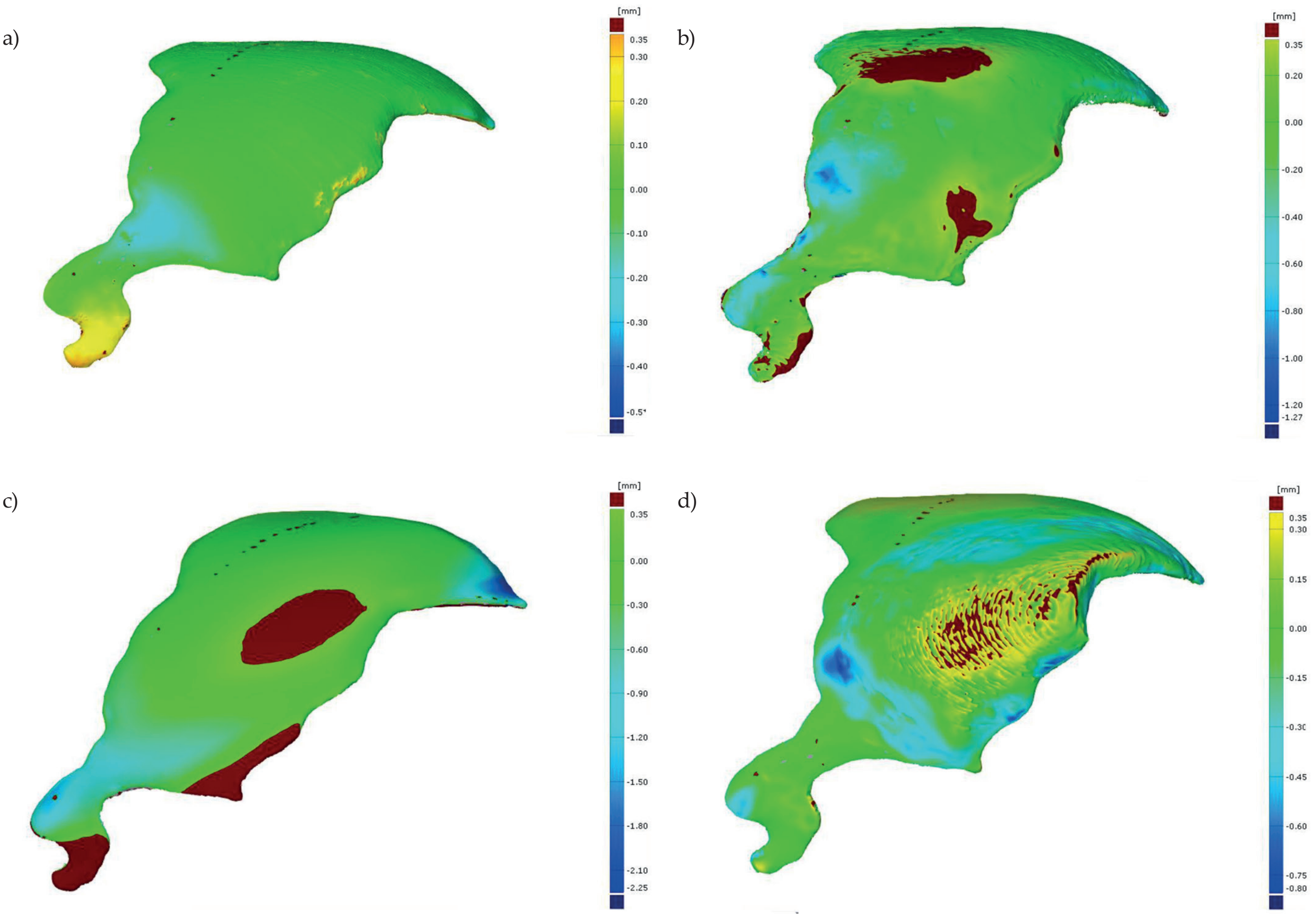

Fig. 4. 3D maps of dimensional deviations of the implants obtained from tested materials: a) MED610, b) PEEK, c) UV resin, d) PLA

measurement stage using the best-fit method. As part of the developed results, three-dimensional maps of deviations were obtained (Fig. 4).

The implant made of MED610 reproduced the CAD model with the smallest deviations. The only deviations occur in the supraorbital area. The color table only allows for an approximate determination of the deviation values.

PEEK implant analysis shows increased deviation values. The implant had visible defects. The green color indicates compliance within a tolerance range of -0.2 to +0.2 mm, blue areas reflect depressions, located where the surface is narrowed. Yellow and red areas indicate excess material, especially visible in the central part, which adhered most tightly to the supports during printing. The obtained result may indicate incomplete removal of the support material; hence the model shows visible surface defects.

Analysis of the UV resin model required a tolerance range of ±2.35 mm. Green indicates compliance from -0.5 to +0.5 mm. The greatest deformations occur at the ends and narrowing's of the model. The printed, ready model had no visible defects. However, significant differences were observed in the accuracy of the reproduction analysis. These deviations may be related to previous mesh repairs made during the measurement process.

The results of the analysis of the PLA implant indicate compliance in the range of -0.15 to +0.15 mm. Shape bulges are points of significant deformation, with the greatest deviations occurring at the edges. The polymer model made using the FDM/FFF technique shows clear distortions resulting from the characteristic arrangement of the fibers. The concentric arrangement of fibers can be observed especially in the upper part of the model.

The highest acceptable dimensional and shape accuracy, within the acceptable range, was ±0.25 mm. The largest surface area within this scale was found in the MED610 material model. This means that despite the deformations, it is closest to the intended dimensions [14–17]. The utilization of additive technologies in the production of medical models, both for direct therapeutic purposes and educational purposes, has expanded these individual areas. The accessibility of 3D printing for medicine has opened a path for the development of this field in bioengineering. Materials offered on the market strive to meet society's expectations in terms of strength and biocompatibility, but there is a lack of general information regarding these properties. Therefore, as part of the work, an attempt was made to determine and investigate the strength parameters and the influence of factors such as humidity, temperature, load, and UV radiation on selected polymers.

The dimensional accuracy of modeled facial implants is highest in the case of the MED610 model, using the PolyJet technique. However, due to significant deformations of this polymer, its application would only be possible in areas with low stress. Additionally, the storage period of implants made from this material should be limited, according to the results of aging tests. The period of use of materials has been determined by the manufacturers.

It is worth noting that there is currently no uniform, standardized procedure for sterilizing biomaterials [3, 7, 9]. The applicable standards impose instructions regarding sterilization stations, which standardize the course of processes and the selection of process parameters. Therefore, further research should follow a series of guidelines, but with an individual approach, since there are no clear practical rules that would indicate a universal decontamination method for each biomaterial. The effects associated with different sterilization methods may be acceptable in one case but contrary in another, depending on the application of the medical model. Thus, despite the complicated process, it is justified to continue attempts to select appropriate methods and specialized process factors [17-24].

CONCLUSIONS

The influence of sterilization and raster angle on the deflection under load of polymer materials used in medical engineering, e.g., in the facial implants were studied. The samples were obtained using 3D printing techniques and a raster angle of 0° and 90°. It has been shown that the printing direction (raster angle), the type of material and sterilization can have a significant impact on the deformation of polymer materials. A cranial-facial bone implant model was also developed and the suitability of the tested materials for obtaining this type of implants was examined. The dimensional accuracy of the implants model was the highest in the case of the MED610 model, which used the PolyJet technique. The only deviations occurred in the supraorbital area. However, due to the significant deflection of this polymer under load, its application is only possible in areas of low stress. In the case of UV photocurable resins printed in the vertical direction (90°), the samples showed the least deflection and the printed model had no visible defects. The greatest deformations occurred at the ends and narrowing's of the model. However, significant differences in repeatability were observed. Unsterilized PLA, regardless of the printing direction, showed the smallest deformations. Unfortunately, it was significantly deformed during sterilization. Therefore, there is no perfect, universal material suitable for all applications. The non-contact optical measurement system used turned out to be accurate and sufficient to obtain the full surface structure of the tested objects, even with their complex geometry.

Author contribution

M.Z. – research concept, methodology, testing, data curation, formal analysis, investigation, validation, visualization, writing; G.B. – research concept, development of models for research; K.G.-D. – research concept, methodology, testing, data curation, formal analysis, investigation, validation, development of models for research;

J.J. – research concept, validation; K.D. – research concept, validation; M.M. – research concept, sterilization tests; D.R. – research concept, sterilization tests.

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Conflict of interest
The authors declare no conflict of interest.

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