Development of a compression moulding process for the manufacturing of artificial polymer heart valves

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Abstract. In this study a controllable compression moulding process has been developed for the manufacturing of variable thickness polyurethane heart valves. An experimental facility was established for the compression moulding process. Additive manufactured polymer moulds (AM) were used to determine the suitable design configuration and test process parameters for the successful manufacturing of polyurethane heart valves. Experiments were carried out with a polyurethane solution (PC3595A-B20 and PC3595A) and solvents (N, N-Dimethylacetamide and Tetrahydrofuran) to investigate the effect of changing compression moulding parameters. Due to the capability of the compression mould to produce thin-walled parts with controlled thickness, experimental results demonstrated that a well-controlled compression moulding technique is a feasible alternative to the dip moulding process. The AM polymer moulds demonstrated that this process could be used in an automated experimental facility to create a working prototype polyurethane heart valve. The AM polymer moulds demonstrated that it is possible to obtain a suitable design configuration of a mould layout and to create a working prototype polyurethane heart valve.

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1 Introduction

Heart valve diseases are currently increasing and the demand for heart valve replacement is likely to increase significantly in the future [1]. Existing mechanical and biological prostheses provide solutions, but at the expense of significantly increased risk of complications. Biological valves are susceptible to fibrosis, calcification, degeneration, and immunogenic problems, while mechanical valves are more likely to produce haemorrhage and thrombosis. To solve the problems with the long-term stability and biocompatibility of artificial heart valves, it is important to develop alternative heart valves [2].

In recent years, tri-leaflet polyurethane heart valves have received significant attention due to their artificially enhanced mechanical properties [3]. Polyurethane is considered the most popular choice of material amongst different types of polymers, due to its biocompatibility and durability [3][4][5][6][7][8][9][10]. Extensive research has been conducted to improve clinical outcomes that depend on the material used in the production of polyurethane heart valves [5][6]. A great advancement in the creation of new polyurethane materials has resulted in materials with improved characteristics that are suitable for heart valve applications [7].

Several types of fabrication techniques could potentially be used for the manufacture of polyurethane heart valves, namely dip moulding, film fabrication, injection moulding, and compression moulding [1]. Each manufacturing method has distinct advantages and disadvantages. The material composition of the valve material chosen typically influences the fabrication techniques used during the production of heart valves.

Several studies have attempted to evaluate the impact of various fabrication techniques on the function and durability of the valves obtained, and they have suggested that dip mould valves have better haemodynamic performance and durability compared to film fabrication valves. However, the polymer used will also influence the available production method, as some procedures are not applicable to certain types of polymers [8][11][9].

A research team from the Central University of Technology, Free State (CUT) has made an effort to assess the functionality of a heart valve manufactured through dip moulding techniques. It was shown that by employing a uniformly polished mould and a dip moulding fabrication method, valves with excellent surface topography could be obtained. However, it is challenging to control the thickness of the leaflet geometries and to prevent air trapping during the dip-moulded process [10]. The compression moulding process is considered an alternative to the dip moulding process due to its ability to produce thin-walled parts with great dimensional accuracy.

Compression moulding is a manufacturing procedure in which a preheated, measured amount of raw material is added to a heated mould. After the material has been placed in the tool, the mould is then compressed shut, pressing the material as it melts into the whole cavity. Heat and pressure are kept constant throughout the procedure until the material has cured. The component can then be removed by opening the mould [12][4]. This procedure is illustrated in Figure 1.1 below.

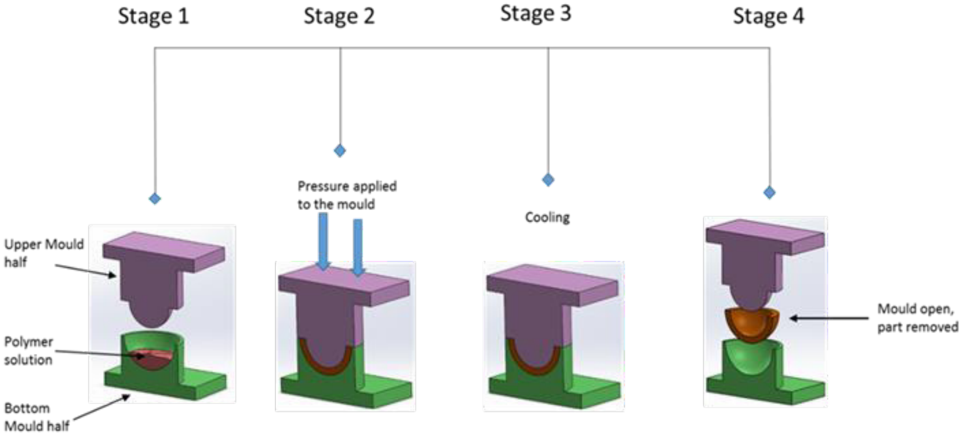

Fig. 1.1. Diagrammatic representation of the compression-moulding procedure

To enable the successful manufacturing of a component, the following process parameters and mould features must be managed and considered during the compression moulding process:

1. The correct amount of material is inserted into the mould cavity.
2. Heat the material and the mould to the temperatures specified by the material manufacturer.

3. The shortest amount of time required to preheat the material and the mould.
4. The necessary pressure must be provided to ensure that the material conforms to the contour of the mould cavity.
5. Curing temperature and time.

The compression moulding procedure should enable the manufacturing of leaflets with controlled thickness and the requisite mechanical and physical qualities by using additive manufacturing technologies to manufacture a mould (based on reverse engineering of a commercially available tissue valve). However, for medical applications, improving the surface finish of additively manufactured parts after the building process is still critical. The surface topography of polymer heart valve leaflets can influence thrombus development (clotting) in blood circulation. As a result, a smooth finish is required on both surfaces of the polyurethane leaflets. *In vivo* fatigue testing revealed reasonable endurance for polyurethane valves with leaflet thicknesses ranging from 100 to 300 µm, which serves as a baseline for acceptable heart valve leaflet thickness [13][14].

This study aims to develop a controllable experimental facility and a proven procedure that would enable repeatable and reliable production of controlled thickness polymer heart valves through the compression moulding process. The process parameters required to obtain the desired thickness and surface topography of the leaflets will be determined using cost-effective methods. The established procedure and the process parameters will be utilised to manufacture a functional prototype polyurethane heart valve.

2 Methodology

2.1 Design and manufacture of moulds

Additive manufacturing (AM) technologies were used to manufacture moulds for the compression moulding process to produce polyurethane heart valves with constant leaflet thickness. For preliminary testing, AM polymer moulds with different design configurations were manufactured using a Formlabs Form 3B desktop stereolithography (SLA) 3D printer, which is suited for the rapid production of moulds and end-use parts that require biocompatibility and sterilising compatibility. This enabled a more agile manufacturing approach, allowing the prototype of the moulds and testing different mould configurations in the most cost- and time-effective manner. During experimentation, durability of the moulds and a set of parameters suitable for the production of a polyurethane heart valve were determined.

White resin (Rigid 10K) was identified as a viable resin for mould manufacturing due to its ability to withstand extensive testing and function under pressure. This extremely glass-filled resin is the stiffest material available for use with Formlabs SLA 3D printers. The design configuration A with rigid 10K resin was used to manufacture the first mould and tested as shown in Figure 2.1 A. However, the design configuration and the mould did not perform well. As a result, the design and material selection for design configuration B were revised. Grey resin (Tough 2000) was then chosen as a replacement for its capabilities in prototyping strong and solid objects that can withstand high stress and strain. The design configuration B manufactured from Tough 2000 resin is illustrated in Figure 2.1 B.

Fig. 2.1. Design configuration A (White resin (Rigid 10K)) and B (Grey resin (Tough 2000)).

2.2 Surface roughness of the resin mould

Although AM has made significant advancements in terms of material diversity and mechanical performance, all types of AM processes are still severely constrained by their comparatively poor surface finishes when compared to traditional fabrication methods. Improving the surface finish of AM parts is essential for certain applications after the production process. As a result of the impact of the stair steps on the mould surface quality, removal of the stair steps is required. Otherwise, the leaflets produced from a mould with a poor surface finish may influence thrombus formation in the blood. Therefore, the compression-moulded leaflet must have a smooth finish on both sides. Figure 2.2 below shows the surface finish of the AM resin mould.

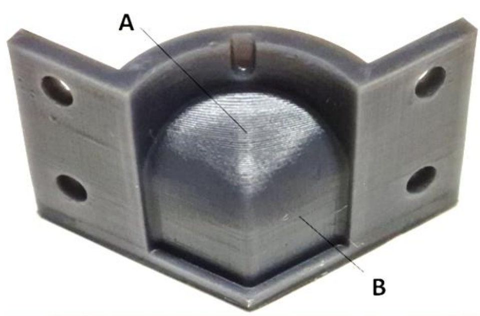

Fig. 2.2. Stair-step effect of the resin surface finish

The surface roughness of the resin mould was measured at two places, A and B, as illustrated in Figure 2.2. Surface roughness measurements were taken with Mitutoyo SURFTEST SJ-210 surface roughness measurement equipment [10]. This is portable surface roughness measuring equipment that follows the surfaces of components, measures their surface roughness using roughness standards, and displays the findings on a liquid crystal display screen. The measurement variables used to determine the surface roughness values are shown in Table 2.1.

Table 2.1. Measuring parameters and conditions.

Measuring parameters	**Values**
Roughness measurement parameter	R_a
Cutoff value or sampling length, λ_c (mm)	2.5
Number of sampling lengths, N	2, 3, 4 and 5
Evaluation length range $\boldsymbol{l_n} = \boldsymbol{\lambda_c} \times \boldsymbol{N}\ (\boldsymbol{mm})$	5 to 12.5
Measurement traversal speed (mm/s)	0.5

2.3 Preparation of polyurethane solution

Polyurethane granules PC3595A-B20 and PC3595A were dissolved in N, N-dimethylacetamide (DMAc) and tetrahydrofuran (THF) solvents to produce the solution used to fabricate polyurethane heart valve samples by compression moulding. To correctly calculate the weights of the polyurethane granules and the volume of solvents, a WTB 2000 precision balance scale and syringes were used. At room temperature, 10 grams of polyurethane granules and 15 ml of solvent were combined in a glass container. To achieve the appropriate viscosity for the compression moulding process, the mixture was placed in a preheated oven at 89 °C for 8 hours (DMAc solvent) and 4 hours (TFH solvent).

2.4 Manufacturing of polyurethane leaflets

Experiments were conducted using a solution of polyurethane (PC3595A-B20 and PC3595A) and solvents (N, N-Dimethylacetamide and Tetrahydrofuran) to analyse the effect of varying compression moulding parameters. The amount of solids in the solution determines its viscosity, which may be changed by incorporating additional solvent or polyurethane material into the mixture until the appropriate consistency is reached. According to previous studies, a 1:1.5 weight-to-volume solution of 1: 1.5 was made to suit this process appropriately.

For preheating, the moulds and the polyurethane solution were placed in two separate ovens at controlled temperatures. After the moulds and the polyurethane solution had reached the necessary temperatures (35 °C and 89 °C, respectively), 3.5 g of the solution was compressed into a mould cavity at room temperature, and then the mould was placed in an oven at 89 °C for 5 minutes to let the solution settle before being allowed to cure in a different oven at 35 °C with the holding pressure of 0.25 MPa for 3 hours.

2.5 Thickness measurement of polyurethane leaflets

The thickness of the compression moulded heart valve leaflets was measured at various points, as shown in Figure 2.3. These locations were identified as crucial areas impacting the valve's hydrodynamic performance[5][9][10].

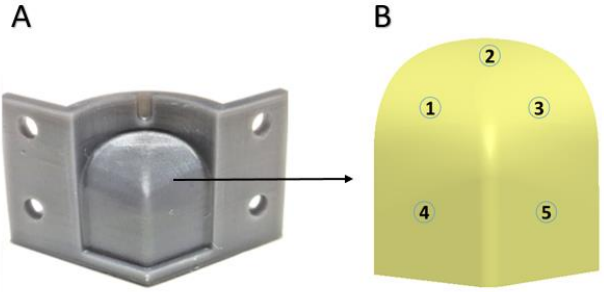

Fig. 2.3. (A) Part of the grey resin mould produces one face of a trileaflet valve. (B) Representation of thickness measurement areas on a single sheet.

The thickness measurements were performed using a Fisher Dual Scope FMP40 with FD10 probe that uses the eddy current measuring principle.

3 Results

3.1 Surface roughness

The surface roughness of the resin mould was measured at two locations, A and B, as shown in Figure 2.2. The results of these measurements are summarised in Table 3.1.

Table 3.1. Measurements of surface roughness of the resin mould surfaces

Surface finish R_a (µm)		
	Positions	
	A	B
White resin (Rigid 10K)	13.027	0.818
Grey Resin (Tough 2000)	2.700	1.566

The arithmetic average of the absolute roughness of the surface, indicated by R_a in positions A and B for the grey resin mould, varied from 1.566 µm on position B to 2.700 µm on position A. Because of the stair-step effect, the surface finish of the White Resin mould has a high R_a value (13.027 µm) on position A. Although both moulds were created using the same AM system at 0.1 mm layer thickness, the build parameters, such as chamber temperature and print speed, are automatically regulated by the Buildvolume operating system, based on the material used. The considerable difference in surface roughness at point A between the two moulds may have been influenced by the different characteristics of the resin materials and the specific process parameters used during the mould manufacturing. The rough surface topography indicated by high R_a values will result in a rough surface of heart valve leaflets, influencing the development of thrombus in blood circulation. As a result, an improvement in the surface quality of all mould surfaces was required.

Scanning electron microscopy (SEM) analyses were performed on the valves produced from the resin AM moulds to determine the surface topography of the leaflets. Figure 3.1 shows the profile shape and surface roughness of the sample leaflets examined by SEM.

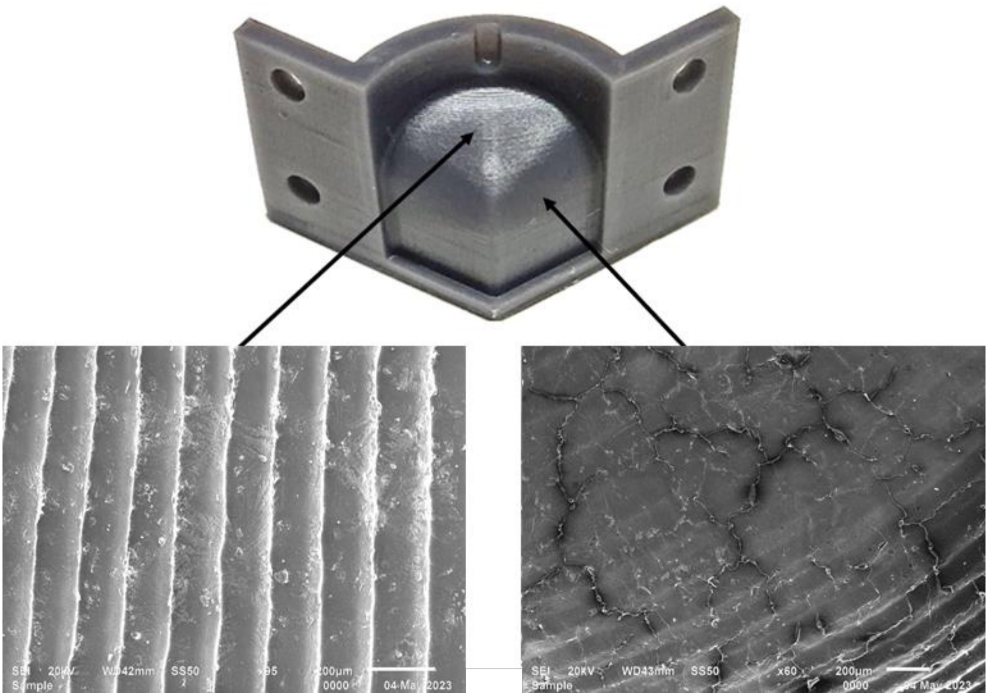

Fig. 3.1. SEM micrographs showing the surface topography of the polyurethane leaflet produced with resin mould

Due to the curved shape, the stair step results in an approximate 0.15 mm steps as shown in Figure 3.1. The roughness of the mould surfaces clearly determines the topography of the surface of the heart valves produced.

3.2 Design and manufacturing of moulds

During compression moulding trials, mould configuration A (Figure 2.2) did not produce acceptable results since the mould and solution fused, making it impossible to remove the mould valve from the mould, as shown in Figure 3.2.

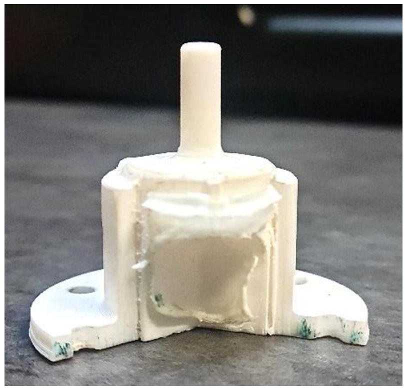

Fig. 3.2. Mould configuration A fused with polyurethane PC9535A-B20 solution

Configuration B, on the other hand, allowed for easy disassembly of the mould and removal of the tri-leaflet valve. Further testing was carried out in Configuration B and an optimised compression moulding parameters and procedure were established. Using the Ti-6Al-4V AM moulds, the established technique will be used to produce tri-leaflet valves. Ti6AI-4V is selected due to its high strength, fatigue resistance, and biocompatibility [10]. Ti6AI-4V was successfully used and proven in previous research work done at CUT [15].

3.3 Prototype

Several experiments with polyurethane granules and solvents were carried out to create the prototype heart valve. The first experiment was conducted using the conditions described in Sections 2.3 and 2.4. There was a difference in the preparation time between the N,N-DMAc, and THF solvent solutions. The polyurethane materials dissolved well with the N,N-DMAc solvent and a homogeneous solution was obtained at 89 °C in 8 hours. On the contrary, the THF solvent dissolved quickly and formed air bubbles at this temperature and duration. To create a homogeneous solution, the preparation time for the THF solution had to be adjusted. These initial parameters allowed the manufacture of prototype polyurethane heart valves; however, they still require refinement.

Figures 3.3.1 and 3.3.2 below show stentless valves produced from PC9535 and PC9535_B20 materials, respectively.

Fig. 3.3.1. Valve fabricated from PC9535A Fig. **3.3.2.** Valve fabricated from PC9535A_B2

As seen in Figure 3.3.1, some areas of the produced leaflet contained air bubbles. This is caused by the inability to allow the solution to settle at the bottom of the cavity before compressing the mould. The compression speed of the mould also has a significant impact on the development and entrapment of air bubbles. Compression moulding parameters must be properly controlled to avoid the formation of these air bubbles.

3.4 Measurement of leaflet thickness

Tables 3.4.1 and 3.4.2 summarise the results of the thickness measurements of a PC9535A polyurethane leaflets at different points on the three faces.

Table 3.4.1. Results of thickness measurements of a PC9535A polyurethane leaflets at different points on the three faces.

	Polyurethane Solution PC9535A					
Measured Point	**Thickness measurement on face 1 (µm)**	**Thickness measurement at face 2 (µm)**	**Thickness measurement at Face 3 (µm)**	**Average**	**Ideal Thickness**	**Standard Deviation**
1	309.0	310.0	307.0	308.7	100.0	104.3
2	371.0	369.0	370.0	370.0	100.0	135.0
3	310.0	309.0	309.0	309.3	100.0	104.7
4	99.0	101.0	99.8	99.9	100.0	0.8
5	99.6	100.0	99.7	99.8	100.0	0.2

Table 3.4.2. Results of thickness measurements of a PC9535A_B20 polyurethane leaflets at different points on the three faces.

	Polyurethane Solution PC9535A-B20					
Measured Point	**Thickness measurement on face 1 (µm)**	**Thickness measurement at face 2 (µm)**	**Thickness measurement at Face 3 (µm)**	**Average**	**Ideal Thickness**	**Standard Deviation**
1	325.0	326.0	325.0	325.3	100.0	112.7
2	385.0	383.0	383.0	383.7	100.0	141.8
3	328.0	326.0	326.0	326.7	100.0	113.3
4	100.0	99.8	99.3	99.7	100.0	0.3
5	101.0	100.0	99.0	100.0	100.0	0.8

Figure 3.4.2 shows a graphical representation of the results in Tables 3.4.1 and 3.4.2 is shown in Figure 3.4.2.

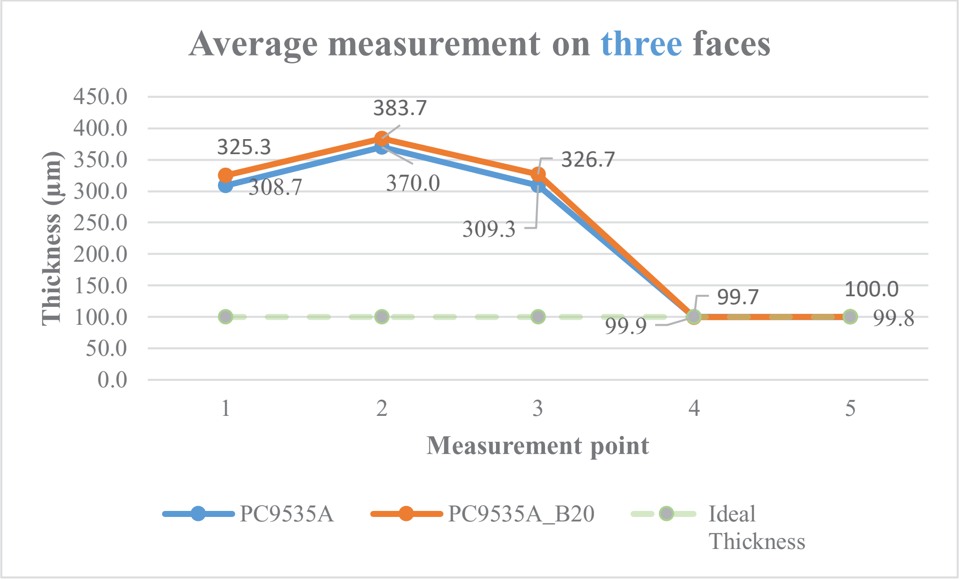

Fig. 3.4.2. Variation of thickness for different measurement points of leaflets made from materials PC9535 and PC9535_B20.

Figure 3.4.2 shows that the average thickness of the two materials leaflets (PC9535 and PC9535_B20) ranges from 308.7µm to 383.7 µm at points 1, 2, and 3, but only from 99.7 µm to 100 µm at points 4 and 5. According to the CAD drawing of the leaflet design, the intended thickness of the leaflets across all faces was 100 µm. The thickness range is not achieved at points 1 to 3, however, it is reasonable at points 4 and 5.

The significant variation in thickness from point 1 to point 5 is due to the mould's inconsistent compression pressure, particularly during the curing phase when the mould was at zero compression pressure. As a result, material from the cavity bottom and trapped air were forced upward to the convex geometry of the mould.

4 Conclusions

AM resin moulds proved that the compression moulding process may be used in an automated experimental facility to produce a prototype polyurethane heart valve. The experimental equipment and facilities to determine the factors of the compression moulding process that resulted in the production of trileaflet valves were established. It was possible to obtain a set of parameters (1:1.5 weight to volume solution, solution preheating at 89°C, mould holding pressure of 0.25 MPa for 3 hours at 35°C) and a technique that resulted in the fabrication of prototype polyurethane heart valves, although some parameters still need improvement. Experimental results showed that a well-controlled compression moulding process could be a viable alternative to the dip moulding process because of its suitability to manufacture thin-walled parts with controlled thickness.

The cost-effective and successfully used polymer moulds were sufficient to obtain certain parameters that are critical for the manufacturing of trileaflet valves using a compression moulding process.

5 Future work

It is recommended that an automated experimental facility is required to manage the compression speed and holding pressure of the mould during the curing process. The established technique will be used to produce prototype tri-leaflet valves using well-polished AM Ti6AI4V moulds. The valves produced that meet the required leaflet thickness and mechanical properties will be further tested for functionality and durability on a pulse duplicator machine.

References

1. F. Oveissi, S. Naficy, A. Lee, D. S. Winlaw, and F. Dehghani, *Mater. Today Bio*, 5, December 2019, 2020, doi: 10.1016/j.mtbio.2019.100038.
2. P. Boloori Zadeh, S. C. Corbett, and H. Nayeb-Hashemi, *Mater. Sci. Eng. C*, 35(1), pp. 335–340, 2014, doi: 10.1016/j.msec.2013.11.015.
3. Crago, Matthew Lee, Aeryne Farajikhah, Syamak Oveissi, Farshad Fletcher, David Frederick Dehghani, Fariba Winlaw, David Scott Naficy, Sina., *Mater. Today Commun.*, 33, October, p. 104916, 2022, doi: 10.1016/j.mtcomm.2022.104916.
4. Li, Richard L. Russ, Jonathan Paschalides, Costas Ferrari, Giovanni Waisman, Haim Kysar, Jeffrey W. Kalfa, David., *Biomaterials*, 225, September, p. 119493, 2019, doi: 10.1016/j.biomaterials.2019.119493.
5. B. Rahmani, S. Tzamtzis, H. Ghanbari, G. Burriesci, and A. M. Seifalian, *J. Biomech.*, 45(7), pp. 1205–1211, 2012, doi: 10.1016/j.jbiomech.2012.01.046.
6. D. Bezuidenhout, D. F. Williams, and P. Zilla, *Biomaterials*, 36, pp. 6–25, 2015, doi: 10.1016/j.biomaterials.2014.09.013.
7. A. G. Kidane, G. Burriesci, M. Edirisinghe, H. Ghanbari, P. Bonhoeffer, and A. M. Seifalian, *Acta Biomater.*, 5(7), pp. 2409–2417, 2009, doi: 10.1016/j.actbio.2009.02.025.
8. H. Ghanbari, H. Viatge, A. G. Kidane, G. Burriesci, M. Tavakoli, and A. M. Seifalian, *Trends Biotechnol.*, 27(6), pp. 359–367, 2009, doi: 10.1016/j.tibtech.2009.03.002.
9. T. G. Mackay, D. J. Wheatley, G. M. Bernacca, A. C. Fisher, and C. S. Hindle, *Biomaterials*, 17(19), pp. 1857–1863, 1996, doi: 10.1016/0142-9612(95)00242-1.
10. L Masheane, WB du Preez, J Combrinck, "Assessment of manufacturability and performance of polyurethane heart valves produced through a locally developed dip moulding process," in *Proceedings of 17th RAPDASA Annual International Conference*, 2016, 4(2), pp. 5–37.
11. H. Jiang, G. Campbell, D. Boughner, W. K. Wan, and M. Quantz, *Med. Eng. Phys.*, 26(4), pp. 269–277, 2004, doi: 10.1016/j.medengphy.2003.10.007.
12. F. Oveissi, S. Naficy, A. Lee, D. S. Winlaw, and F. Dehghani, *Mater. Today Bio*, 5, p. 100038, 2020, doi: 10.1016/j.mtbio.2019.100038.
13. A. Karakoç, O. Aksoy, and E. Taciroğlu, *J. Biomech.*, 156, May, p. 111663, 2023, doi: 10.1016/j.jbiomech.2023.111663.
14. G. M. Bernacca, B. O'Connor, D. F. Williams, and D. J. Wheatley, *Biomaterials*, 23(1), pp. 45–50, 2002, doi: 10.1016/S0142-9612(01)00077-1.
15. L. Reginald Masheane, *Development of a repeatable dip moulding process for manufacturing polymer artificial heart valves master of engineering in mechanical engineering in the*, no. October. 2017.