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| RESEARCH ARTICLE

A Feasible Study on the Application of Cyclic Annealing Treatment for Functionality Improvement of 3D Printed PLA Parts

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| ABSTRACT
In the 3D printing of polymeric parts, the annealing cycle is a post-process method that allows different polymeric materials to enhance their mechanical and thermal properties and also eliminate their residual stresses. However, annealing has some drawbacks, such as producing induced shrinkage to the treated parts. In this study, a new technique named "cyclic annealing" is proposed to reduce shrinkage due to annealing treatment. By subsequent increasing and reduction of the temperature over and below the glass transition temperature instead of suddenly increasing the temperature to the target annealing temperature, a significant reduction of shrinkage was observed for the printed PLA parts. Various cylindrical shape samples with different infill densities (30%, 50%, 70%, and 100%) were examined at 70°C, 90°C, and 110°C annealing temperatures. Considerable reduction of shrinkage by up to 50% and improvement of the material strength were observed, which confirms the applicability of the proposed cyclic annealing method as an alternative to traditional polymer annealing for 3D printed materials.

| KEYWORDS

Fused Deposition Modeling; Poly-Lactic Acid, Shrinkage; Residual Stress, Cyclic Annealing

| ARTICLE INFORMATION

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1. Introduction

Nowadays, 3D printing technology is opening the way for applying in the manufacturing of various products as well as rapid prototyping, which includes the applications of 3D printing in medicine such as regenerative medicine, cardiovascular implants, dental niches, drug carriers, orthopedic interventions, cancer therapy, skin and tendon healing, medical tools and applications in industry, such as manufacturing of fixtures. [Mohammed, 2021 ; Park, 2022 ; Thompson, 2022 ; Raheem, 2021]

Due to the need for manufacturing 3D printed parts with higher strength and acceptable dimensional accuracy, the use of methods that can improve the strength of parts has received much attention from many researchers [Uribe-Gomez et al. 2021]. Among the proposed processes, the annealing treatment has been studied by some researchers, and its efficiency in increasing strength and durability has been proven [Szust, 2022; Bhandari, 2019; Pazhamannil, 2022]. However, annealing of polymeric parts faces some challenges, such as induced shrinkage occurring after the treatment.

In the annealing method, the polymer parts are kept above their glass transition temperature for a certain period of time and gradually cooled. During the gradual cooling process, crystalline structures are formed in the polymer (Fig .1). The higher the crystallinity, the stronger the piece. It has been reported that when the parts are annealed, the higher the annealing temperature, the stronger the crystal structure is, but in contrast, more deformation and shrinkage are observed in part [Gupta et al. 2021]. On the other hand, the thermal resistance of the part increases. In other words, softening and unwanted deformation of the polymeric part occur at higher temperatures [Naveed, 2021]. According to what was mentioned above, in order to prevent unwanted deformations and shrinkages due to increasing temperature and entering the glass transition temperature zone, the effect of cyclic annealing on shrinkage of the part after the process is examined.

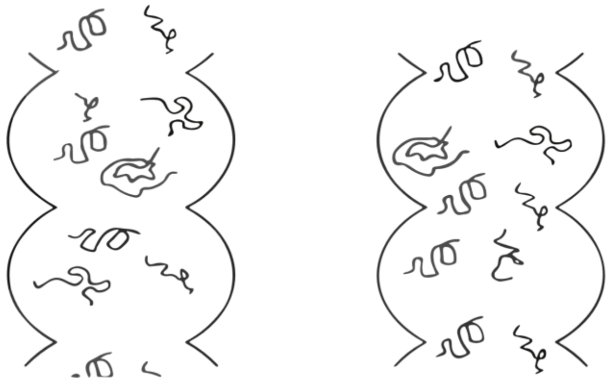

Fig. 1: Annealing treatment for improvement of crystallinity of polymeric parts (Left: before annealing, right: after annealing)

2. Research objectives

The higher the crystallinity due to the annealing process in PLA parts, the greater the expectation of improved mechanical properties. The greater the difference between the heating and cooling temperatures of the part, the greater the probability of shrinkage due to the annealing operation. In general, the higher the temperature of the polymer material, the higher the glass temperature due to the formation of crystalline structures, and consequently, the higher the thermal resistance of the annealed material compared to the untreated one. Changing the heat treatment method of the material from continuous to forward and backward tempering, it is expected that due to the reduction of the heating range compared to the point of entry into the glass area, less shrinkage will be observed in the final piece.

3. Methodology

To investigate the effect of cyclic annealing strategy on the induced shrinkage and improvement of the mechanical properties, test samples were prepared based on the ASTM D695-15 standard test for compression of rigid plastics. As shown in Fig .2, the GEEETECH Prusa i3 FDM printer has been used for printing the test samples using a 1.75 mm PLA filament. As shown in Fig .3, a triangle pattern has been chosen for the infill pattern in 4 densities using Ultimaker Cura V.4.13 slicer software. The printing parameters are summarized in Table 1. 16 samples with infill densities of 30%, 50%, 70%, and 100% were printed and used for annealing treatment. An F11L-1320 Azar furnace has been used for annealing the printed specimens, as shown in Fig .4. The samples were exposed to temperatures above the glass transient point for 30 minutes and then gradually cooled in the furnace after the heating time elapsed. Annealing temperatures were set to 70°C, 90°C, and 110°C. For the cyclic annealing treatment, four samples in different densities were heated to 110 in 3 steps. In the first step, they heated up to 70°C and cooled down to 50°C after 10 minutes. In the second step, they heated up to 90°C and cooled down to 50°C after 10 minutes, and in the third step, they heated up to 110°C and cooled down to 50°C after 10 minutes.

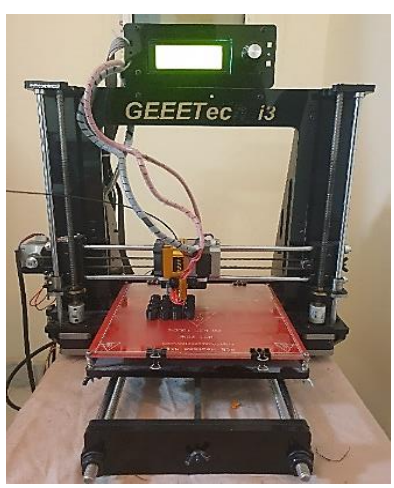

Fig. 2: The process of 3D printing for preparation of the test samples.

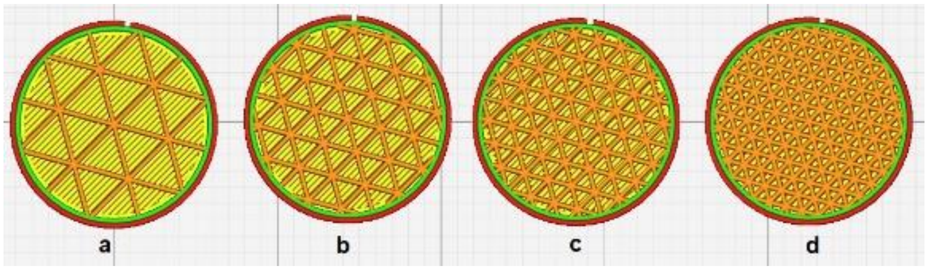

Fig. 3: Triangle infill pattern used for printing specimens with various infill densities; a:30% b=50% c=70% d=100%.

Table 1: The printing parameters

Filament	*Nozzle size*	*Nozzle Temp.*	*Heatbed Temp.*	*Layer height*	*Print speed*
PLA 1.75 mm	0.3 mm	200	60	0.25 mm	60 mm/s

Fig. 4: F11L-1320 Azar furnace used for the annealing process.

The designed experiments for various annealing temperatures and infill densities are depicted in Table 2.

Table 2: The designed experiments for annealing treatment

Number of Experiments	Annealing Temperature	Infill density
16	70 90 110 cyclic110	30 50 70 100

To investigate the influence of annealing treatment on the mechanical properties of the PLA test samples, compression tests have been done using Santam STM-250 universal test machine based on ASTM D695-15. (Fig. 5)

Fig. 5: Compression test apparatus.

Sixteen 3D-printed annealed cylindrical samples were scanned, and their diameters were measured using Solidworks 2021. tested based on ASTM D695-15.

4. Results and Discussion

The obtained results for shrinkage due to the annealing process have been depicted in Table 3. To investigate the effect of annealing temperature and efficiency of the proposed cyclic strategy, the induced shrinkage due to traditional and cyclic annealing is illustrated by bar charts in Figs. 6 to 9.

Table.3: Compression test results for PLA printed samples

infill density	*Annealing temperature*	*Final diameter*	*Shrinkage with respect to the fully filled sample*	*Shrinkage with respect to its non-tempered sample*
30	A: No	12.63	0.5512	0
	B:70	12.58	0.9449	0.3959
	C:90	12.51	1.4961	0.9501
	D:110	12.41	2.2835	1.7419
	E: cyclic	12.58	0.9449	0.3959
50	A:No	12.61	0.7087	0
	B:70	12.56	1.1024	0.3965
	C:90	12.54	1.2598	0.5551
	D:110	12.39	2.4409	1.7446
	E:cyclic	12.57	1.0236	0.3172
70	A:No	12.64	0.4724	0
	B:70	12.58	0.9449	0.4747
	C:90	12.53	1.3386	0.8703
	D:110	12.48	1.7323	1.2658
	E:cyclic	12.56	1.1024	0.6329
100	A:No	12.7	0	0
	B:70	12.68	0.1575	0.1575
	C:90	12.58	0.9449	0.9449
	D:110	12.48	1.7323	1.7323
	E:cyclic	12.68	0.1575	0.1575

Fig. 6 shows the effect of traditional and cyclic annealing on induced shrinkage for 30% infilled PLA samples. For traditional annealing of 30% infilled samples, by increasing the annealing temperature, shrinkage is increased. More temperature deviation from the glass transition temperature provides more time for the material to increase its crystallinity while the cooling process. More crystallinity causes more shrinkage in the part. Among the 30% infilled parts, one with an annealing temperature of 70°C has the least shrinkage with about 0.5%. The highest shrinkage was observed at 110°C. The cyclic annealed part shows much lesser shrinkage than the traditional annealing at 110°C by about 1%. Its shrinkage is similar to the 70°C annealing.

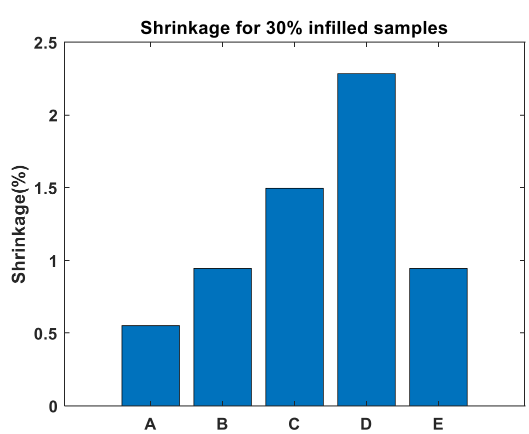

Fig. 6: The effect of traditional and cyclic annealing on induced shrinkage for 30% infilled PLA samples.

Fig. 7 illustrates the effect of traditional and cyclic annealing on induced shrinkage for 50% infilled PLA samples. For traditional annealing, similar to what was observed for 30% infilled parts, by increasing the annealing temperature, shrinkage is increased. Among the 50% infilled parts, one with an annealing temperature of 70°C has the least shrinkage with about 0.7%, which is more than the 30% infilled part with similar annealing conditions. The highest shrinkage was observed at 110°C. The cyclic annealed part shows much lesser shrinkage than the traditional annealing at 110°C by about 1%. Its shrinkage is lower than 70°C and 90°C annealing.

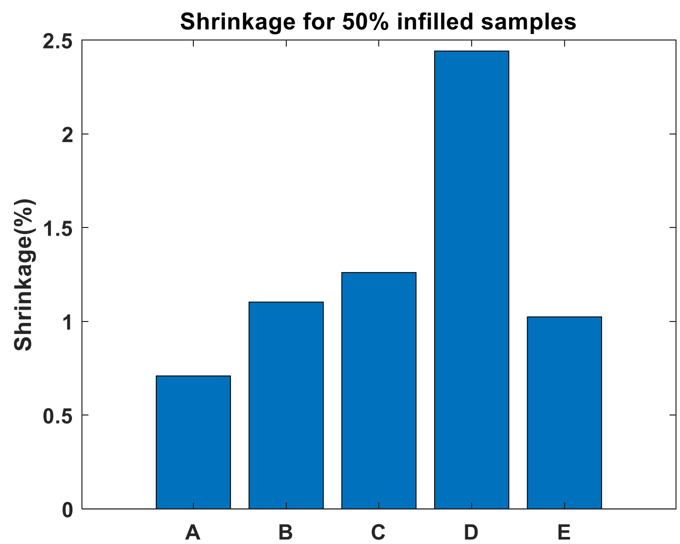

Fig. 7: The effect of traditional and cyclic annealing on induced shrinkage for 50% infilled PLA samples.

The effect of traditional and cyclic annealing on induced shrinkage for 50% infilled PLA samples is depicted in Fig. 8. For traditional annealing, similar to what was observed for 30% and 50% infilled parts, an increase of the annealing temperature leads to more shrinkage. Among the 70% infilled parts, one with an annealing temperature of 70°C has the least shrinkage with about 0.45%, which is less than the 30% and 50% infilled parts with similar annealing conditions. The highest shrinkage was observed at 110°C with 1.7%. The cyclic annealed part shows again lesser shrinkage than the traditional annealing at 110°C with about 1.1%. Its shrinkage is lower than 90°C and 110°C.

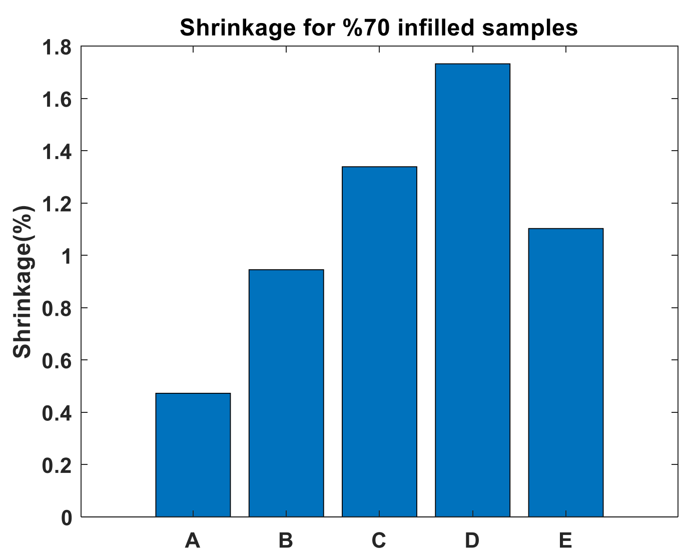

Fig. 8: The effect of traditional and cyclic annealing on induced shrinkage for 70% infilled PLA samples.

Observation of the effect of traditional and cyclic annealing on induced shrinkage for fully infilled PLA samples shows a magnificent shrinkage reduction for the cyclic annealed sample with respect to the traditional annealing technique in all temperatures. It can be observed that for fully infilled parts, cyclic annealing is significantly more effective in reducing induced shrinkage.

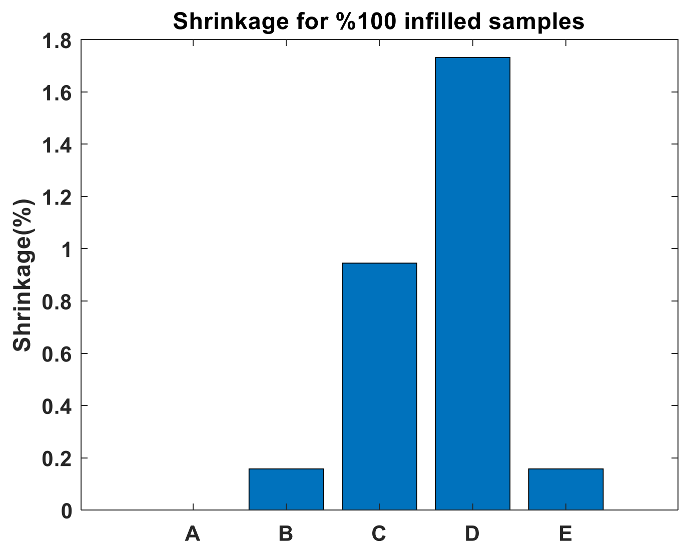

Fig. 9: The effect of traditional and cyclic annealing on induced shrinkage for fully infilled PLA samples.

As mentioned before, annealing can affect the strength, toughness, and heat resistance of polymers. In addition, the proposed cyclic annealing method shows its effectiveness in reducing the shrinkage of the 3D printed PLAs dramatically with respect to traditional annealing. To see the success of the proposed technique for strengthening 3D printed parts, some compression tests were performed on the annealed parts. The force-displacement curves for 70% infilled density in conditions of not annealed, regular annealing at 70°C, 90°C, and 110°C, and cyclic annealed parts have been shown in Figure 10. The force-displacement curve for the cyclic annealed sample shows that the proposed strategy works very well and has the ability to improve the material's strength and toughness. Cyclic annealing shows a high potential to be applied as a post-process for strengthening a wide range of 3D printed materials while controlling the challenges of shrinkage.

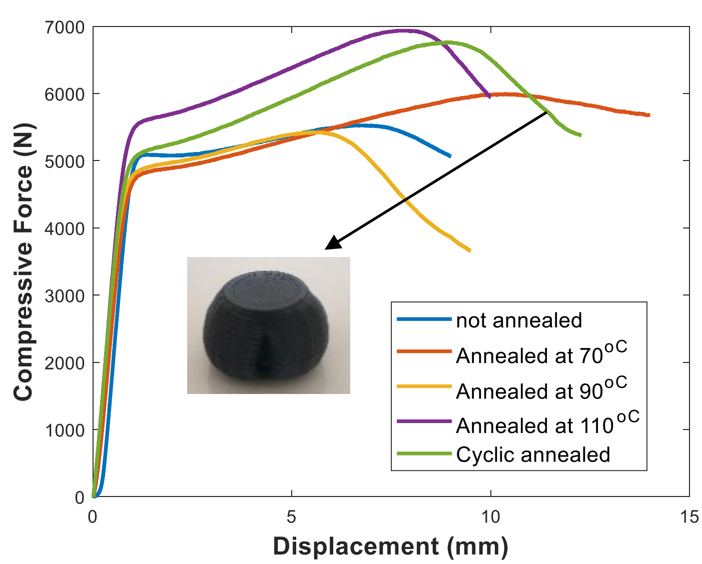

Fig. 10: Compressive force-displacement curves for 70% infilled PLA samples in various annealing temperatures.

5. Conclusion

A feasible study on the application of cyclic annealing for reducing shrinkage and strengthening 3D-printed PLA parts was done. The main important findings are summarized as follows:

- Cyclic annealing can reduce induced shrinkage for PLA printed parts with various infill densities during the annealing process.
- Cyclic annealing can be applied to increase compressive strength and toughness.
- Cyclic annealing helps the improvement of the functionality of polymeric 3D printed products.

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