International Journal of Scientific Research in Engineering and Management (IJSREM)
Volume: 09 Issue: 04 | April - 2025 SJIF Rating: 8.586 ISSN: 2582-3930

Effects of Heat Treatment on the Mechanical Properties of 3D Printed Polylactic Acid (Pla+)

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ABSTRACT

This present study investigated how heat treatment affects the mechanical properties of 3D-printed PLA+ by manipulating two parameters: heating temperature and holding time. The mechanical properties of 3D-printed PLA+ components are crucial for assessing their structural integrity and performance.

Annealing can improve the internal structure and mechanical characteristics of 3D-printed objects made from PLA+. Heat treatment is an appropriate method to remove the porosity defect because the air gaps in the material are filled with itself when it melts at high temperatures during this process. The research explores changes in flexural strength, creep resistance, tensile and compressive strength before and after subjecting PLA+ samples to controlled annealing conditions. The findings aim to enhance the understanding of heat treatment as a method to improve the performance of 3D-printed PLA+ components.

KEYWORDS

3D-Printing; Polylactic acid (PLA+); Heat treatment; Heating temperature; Holding time; Flexural strength; Tensile strength; Hardness; Impact resistance; Compressive strength.

1. INTRODUCTION

Additive Manufacturing (AM) is more popular than ever because of its unique features, such as flexibility, precision, reduced material wastage, and the ability to produce parts in a short timeframe. This is achieved by the unique way in which AM parts are being made. In particular, parts are manufactured layer-by-layer, producing complex designs. In material extrusion additive manufacturing, a thermoplastic polymer is fed into an extruder which is melted and deposited on the substrate in a controlled manner . Fused deposition modeling (FDM) uses a thermoplastic filament with a fixed diameter, and this technique is considered among popular AM methods. Also, it is an energy-effective and environmentally friendly approach. Currently, a wide range of materials including polylactic acid (PLA+), acrylonitrile butadiene styrene (ABS), polyether ether ketone (PEEK), polyamide (PA), polycarbonate (PC), and polypropylene (PP), have been used. PLA+ and ABS are among the most common filaments used in FDM additive manufacturing. PLA+, however, demonstrates superior environmental and sustainable characteristics. More specifically PLA+ is biocompatible, biodegradable, has low melting temperature, low shrinkage, good wettability, and printability.

Polylactic acid (PLA+) is a widely used Thermoplastic polymer in Additive Manufacturing due to its biodegradability, ease of printing, and cost-effectiveness. However, PLA+ exhibits relatively low thermal stability and mechanical strength compared to other engineering polymers. Heat treatment, also known as Annealing, is a method used to improve these properties by enhancing the polymer's crystalline structure. This study aims to analyze how different heat treatment conditions affect the mechanical properties of 3D-printed PLA+ parts.

2. MATERIALS AND METHODS

Experimental Set-Up

This study was conducted to analyze the surface roughness and dimensional accuracy of parts printed using FDM. Setting up the 3D printer and selecting the proper material for the intended use are both essential steps in the preparation of samples for 3D printing. In this case, the PLA+ filament was printed using an Ender- 3 printer. The infill density is chosen to be 50%, which balances the printed pieces weight and strength. The control parameters for the printer are shown in **Table 1**.

Table 1. Recommended printing parameters by manufacturer PLA+ filament.

Printing speed (mm/s)	60
Layer height (mm)	0.2
Wall Thickness (mm)	1.0
Infill Density (%)	50
Infill Pattern	Gyroid
Printing Temperature (°C)	210

Sample Preparation

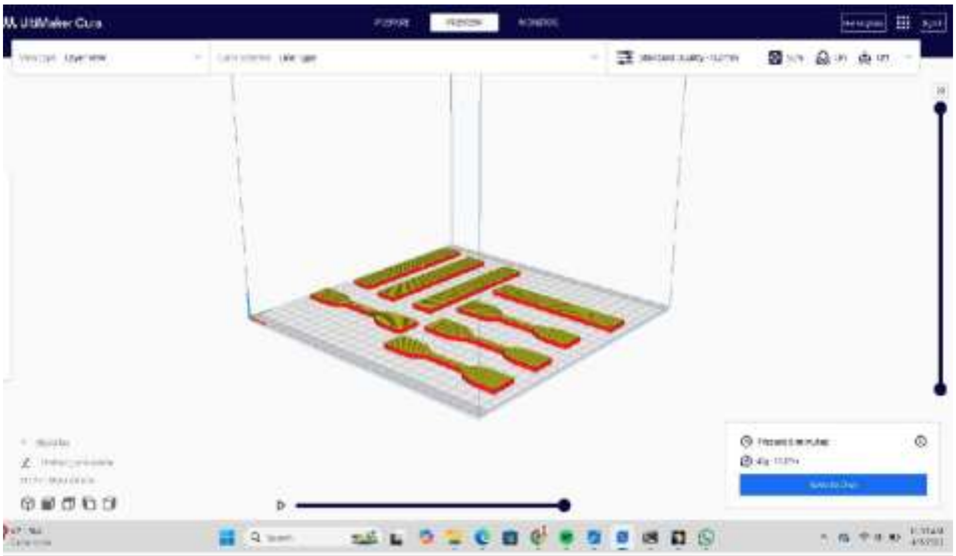


FIG 1: Ultimaker Cura

The samples are prepared using this Ultimaker Cura software, after adjusting all the required parameters then slice it. We have to save the file into memory card then it has to insert into Ender3 printer and proceed to further steps.

- Select the "Print TF Card" and then
- Select "Print" option.

FIG 2: Before and after printing the samples

PLA+ samples were 3D-printed using a Fused Deposition Modeling (FDM) Ender-3 printer with standardized dimensions for tensile, flexural, and creep resistance tests. Printing parameters, including layer height, infill density, and print speed, were kept constant to ensure consistency.

Mechanical tests were conducted using a Universal Testing machine. The tensile tests followed the guidelines outlined in ASTM D638 Type IV, the compressive tests adhered to ASTM D695, and the flexural tests were in accordance with ASTM D790.

The ASTM D638 Type IV is a dog-bone-shaped specimen ideal for testing very soft polymers and comparing materials of different stiffness. It features a 25-mm gage length with a narrow parallel section with width of 6 mm, wider shoulders of 19 mm, and an overall length of 115 mm. Thickness can vary depending on the material, but typically falls within a range of 3–14 mm. For this study, 3 mm thickness was used. This specific geometry provides a good balance between gripping strength and stress concentration in the gage length, making it suitable for accurate tensile testing of soft materials.

For Tensile and Brinell Hardness tests, the following specimen FIG.1. is used to determine the Tensile strength and Hardness accordingly. Tensile test involves applying a controlled tensile load to a material specimen to measure its response to pulling forces, ultimately determining properties like tensile strength and elongation. Brinell Hardness test determines the indentation hardness of materials by measuring the diameter of the indentation left by a hardened steel or tungsten carbide ball under a specified load.

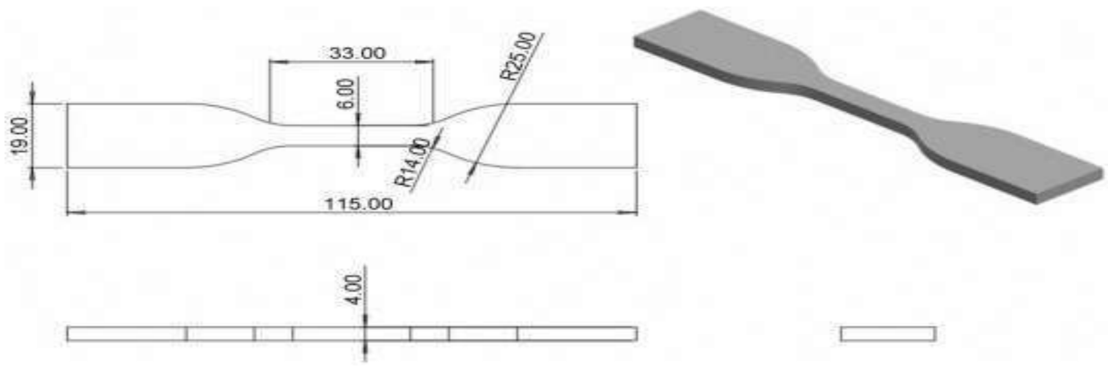


FIG.1. Isometric view of Dog-bone-shaped specimen

For compressive tests, ASTM D695 is used as the standard design for the mechanical properties tests. ASTM D695 uses two common cylindrical specimen sizes: common shape and larger specimen, which is highly versatile. The chosen design had common dimensions of 12.7 mm diameter and 25.4 mm length, making it compact and ideal for measuring various compressive properties such as strength and modulus. The following specimen FIG.2. is used to determine the compressive strength. Compression test determines a material's behaviour under compressive loads, measuring its resistance to crushing forces and deformation.

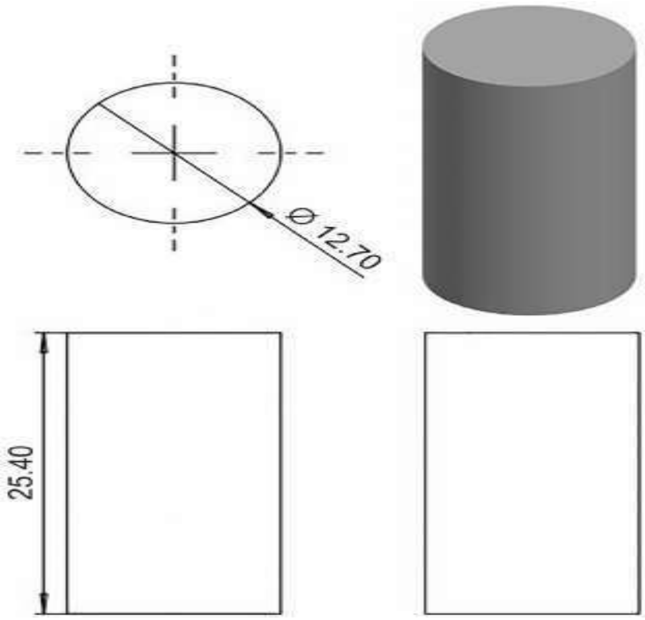


FIG.2.Isometric view of Cylindrical specimen

For flexural tests, ASTM D790-10 is employed as the standard 3-point flexural test. A cuboid shape with a thickness of 4mm was used to study the bending properties of the materials. The chosen design featured a 19- mm width and 115-mm length. The following specimen FIG.3. is used to determine the Flexural strength.

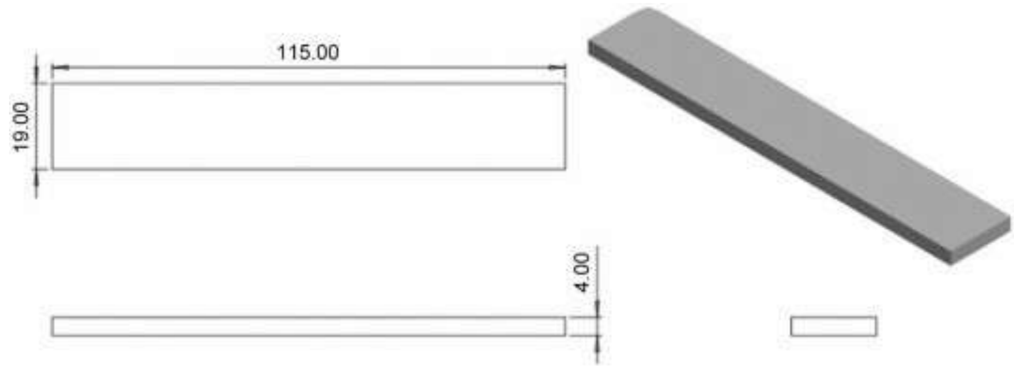


FIG.3.Isometric view of cuboid shape for Flexural test

For Izod Impact test, the test follows the ASTM D256 standard, which specifies the dimensions and shape of the test specimen. The standard specimen is typically 75 x 10 x 10 mm (length x width x thickness). The following specimen FIG.4. is used to determine the Impact resistance of the material.

FIG.4.Isometric view of specimen for Izod impact test

Heat Treatment Process

The total experiment is done on printed specimens of 4 samples for each test, one sample is treated without heat treatment and remaining 3 samples undergo heat treatment process at various temperatures (55°C,65°C,75°C) with some constant durations (30min) which is mentioned below in the **Table.2.** The samples were allowed to cool slowly to minimize warping.

- **Printing Parameters:** The specimens were printed using an FDM Ender-3 printer with a 0.4 mm nozzle, 50% infill density, and layer height of 0.2 mm.
- **Material:** PLA+ filament (1.75 mm diameter).
- **Flexural Testing:** It involves evaluating their resistance to bending forces, often using methods like three-point or four-point bending tests, to determine properties like flexural strength and modulus.
- The whole Heat treatment process is done in a Precision Furnace. These furnaces are designed to control temperature accurately and uniformly, which is crucial for achieving consistent results during the Heat Treatment process.

FIG.5.Furnace for Heat Treatment

FIG.6. 3D-Printed Specimens

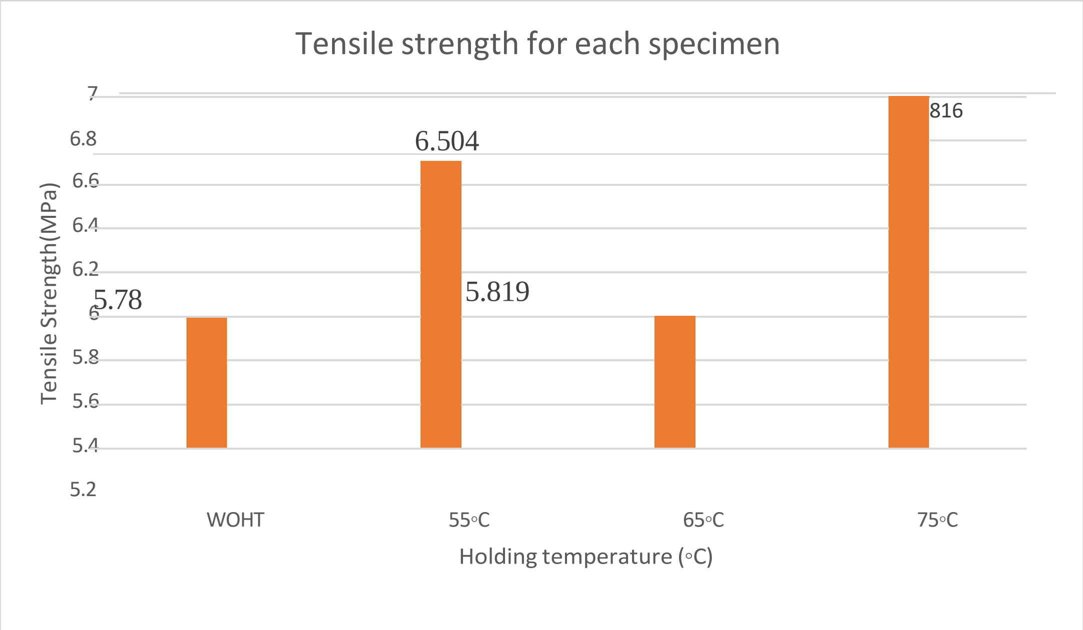
Specimen	Heating Temperature(°C)	Holding Time(min)
1	Without Heat Treatment	NA
2	55	30
3	65	30
4	75	30

3. RESULTS AND DISCUSSION

- **Influence of temperature conditions on the mechanical properties towards 3d printed PLA+ with 50% Infill density and Infill pattern is "GYROID"**

3.1. TENSILE TESTING

As the work of R.A. Wach et al. suggests, there must be considerable amount of improvement in the mechanical properties of the FDM printed parts after annealing. To measure the changes in the properties of the FDM printed part, a tensile test was conducted. The unannealed PLA+ specimen shows a tensile strength of 5.78MPa and for annealing specimen at a temperature of 55°C, holding time 30minutes, the maximum tensile strength is 6.504MPa. For annealing specimen at a temperature of 65°C, holding time 30minutes, the maximum tensile strength is 5.819MPa. For annealing specimen at a temperature of 75°C, holding time 30minutes, the maximum tensile strength is 6.816MPa.



The image shows a bar chart comparing the tensile strength of 3D printed specimens at various heat treatment temperatures (55°C, 65°C, 75°C) and a control without heat treatment (WOHT). Here's a breakdown of why tensile strength varies with different holding temperatures:

How PLA+ Responds to Heat Treatment:

1. **WOHT (Without Heat Treatment) – 5.78 MPa**

PLA+ parts printed without post-processing often suffer from:

- Layer adhesion weaknesses
- Internal stresses due to rapid cooling
- Uneven crystallinity
- These factors reduce the mechanical integrity and result in lower tensile strength.

2. 55°C – 6.504 MPa

- At 55°C, the temperature is just below the glass transition temperature (Tg) of PLA+ (~60– 65°C).
- This mild heating allows some molecular relaxation and slight improvement in interlayer diffusion, improving bonding strength without deforming the part.
- Hence, tensile strength increases noticeably.

3. 65°C – 5.819 MPa

- Around Tg, PLA+ becomes more rubbery and can start to deform internally if not well supported.

- This can cause:
- Partial softening
- Loss of dimensional stability
- Reduction in load-bearing capacity
- So although some relaxation happens, the structure might get slightly weakened, explaining the dip in strength.

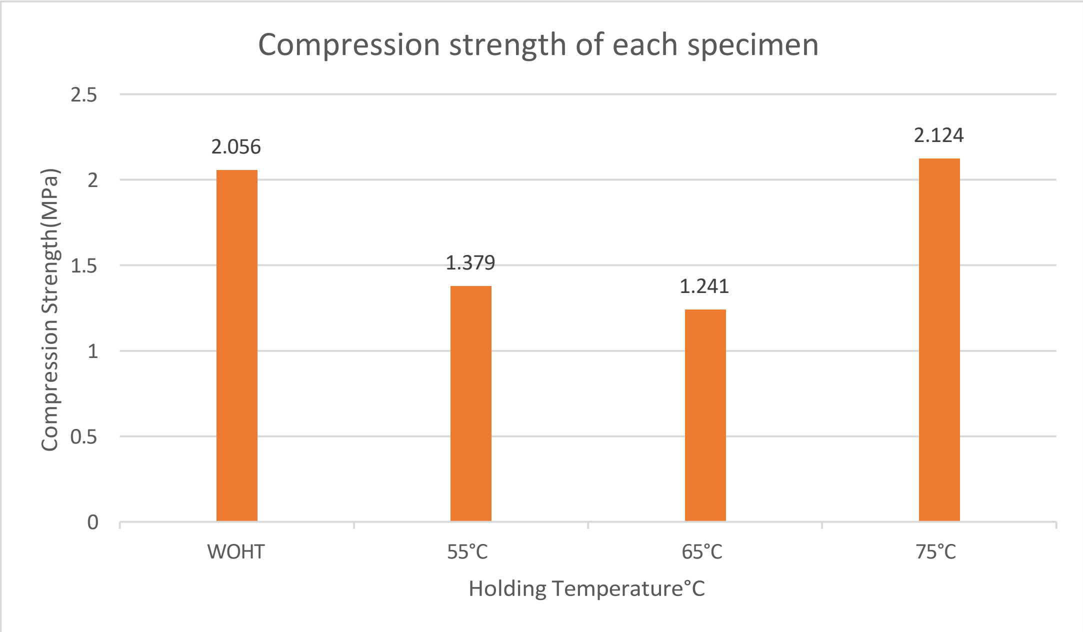
4. 75°C – 6.816 MPa (Maximum)

- At this temperature, PLA+ reaches ideal conditions for chain mobility without reaching the melting point.
- This:
- Maximizes interlayer bonding
- Improves crystallinity
- Eliminates residual stresses
- The structure is reinforced and stabilized, giving the highest tensile strength.

TEMPERATURE	EFFECT ON PLA+	RESULT
WOHT	No stress relief, weak bonding	Lowest strength
55(°C)	Mild relaxation and bonding improvement	Higher strength
65(°C)	At Tg, may cause softening and slight weakening	Slight dip
75(°C)	Optimal stress relief and interlayer fusion	Maximum strength

3.2 COMPRESSION TESTING

The unannealed PLA+ specimen shows a compressive strength of 2.056MPa and for annealing specimen at a temperature of 55°C, holding time 30minutes, the maximum compressive strength is 1.379MPa. For annealing specimen at a temperature of 65°C, holding time 30minutes, the maximum compressive strength is 1.241MPa. For annealing specimen at a temperature of 75°C, holding time 30minutes, the maximum compressive strength is 2.124MPa.



The behaviour of compressive strength in PLA+ under different heat treatment temperatures, based on the chart.

<u>How Heat Treatment Affects Compressive Strength of PLA+</u>

1. WOHT (Without Heat Treatment) – 2.056 MPa

- PLA+ without heat treatment retains internal stress from 3D printing, but:

- The rigid structure from fast cooling can help resist compression to an extent.

- However, it’s also brittle and less stable under long-term or higher loads.

- Still, initial compressive strength remains relatively high, possibly due to material stiffness.

2. 55°C – 1.379 MPa

- At this mild temperature:

- Some internal stress is relieved, but not enough molecular rearrangement occurs to improve structure under compression.

- May even soften slightly, reducing its ability to resist compressive loads.

- This leads to a noticeable drop in compressive strength.

3. 65°C – 1.241 MPa (Lowest)

- Around PLA+’s glass transition temperature (~60–65°C):

- The material becomes rubberier and more deformable.

- This can weaken structural integrity under compression.
- As a result, the specimen shows the lowest resistance to compressive forces.

4. 75°C – 2.124 MPa (Highest)

- At this temperature:
- PLA+ chains gain enough mobility for optimal reorganization and recrystallization.
- Internal voids may collapse and interlayer bonds tighten, increasing density and compressive strength.
- The structure becomes more uniform and stronger under compressive loading.
- Hence, we see the maximum compressive strength here.

TEMPERATURE	EFFECT ON PLA+	RESULT
WOHT	No treatment; stiff but brittle	Higher strength
55(°C)	Mild softening, slight weakening	Lower strength
65(°C)	Near Tg, most rubbery state, weakest	Slight dip under Lower strength
75(°C)	Optimal annealing, strong and dense	Maximum strength

3.3 FLEXURAL STRENGTH

The unannealed PLA+ specimen shows a flexural strength of 18.947MPa and for annealing specimen at a temperature of 55°C, holding time 30minutes, the maximum flexural strength is 22.105MPa. For annealing specimen at a temperature of 65°C, holding time 30minutes, the maximum flexural strength is 28.421MPa. For annealing specimen at a temperature of 75°C, holding time 30minutes, the maximum flexural strength is 22.105MPa.

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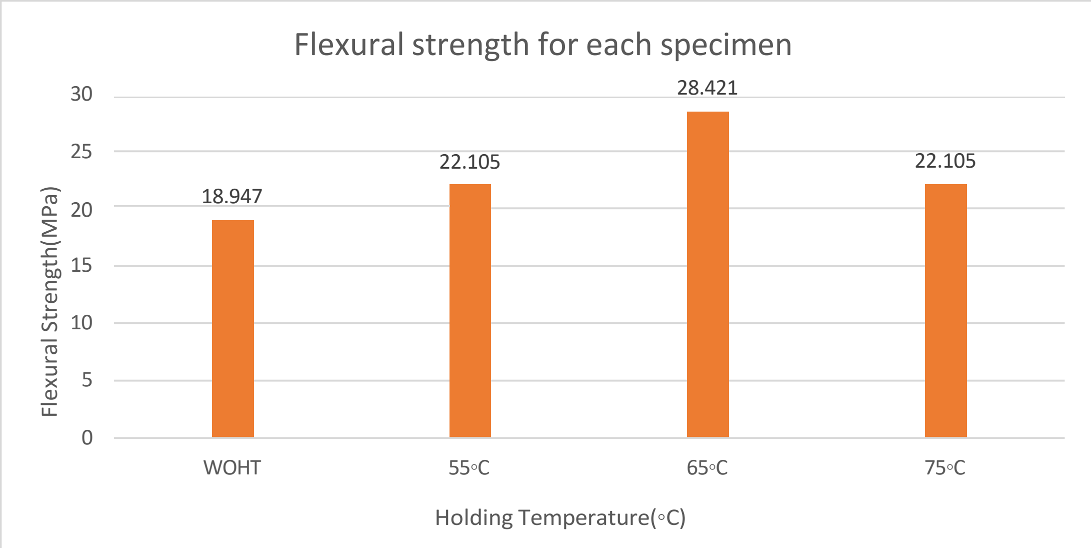


TEMPERATURE(°C)	**EXPLANATION**	**RESULT**
WOHT	As-printed PLA+ has micro voids and weak interlayer bonding, resulting in low resistance to bending stress	Lower strength
55°C	Improved molecular relaxation and slight interfacial strengthening enhance bending resistance.	Higher strength
65°C	Near the glass transition temperature: optimal chain mobility and alignment under bending, maximizing flexural strength	Maximum strength
75°C	Possible over-softening or internal warping reduces strength; loss of optimal stiffness under flexing.	Higher strength

3.4 HARDNESS TESTING

VALUES OF BRINELL HARDNESS TEST:

S.NO	FORCE APPLIED (KG)	DIAMETER OF BALL - D(mm)	DIAMETER OF INDENTATION – d(mm)
1	250	5	2.6
2	250	5	2.4
3	250	5	2.2

4	250	5	2

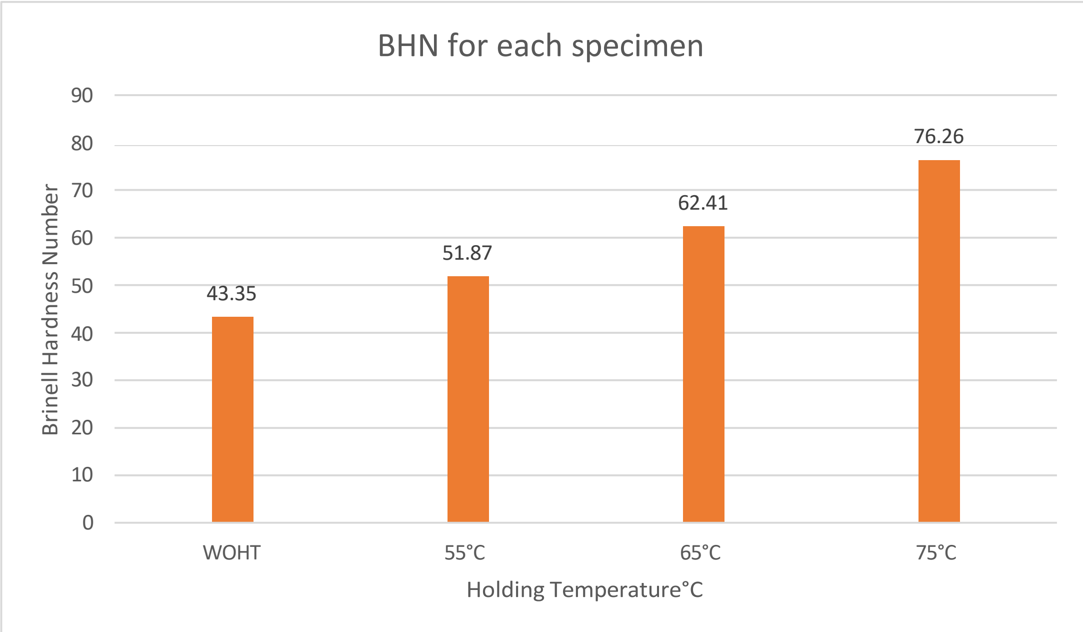
FORMULA

$$\text{BHN} = \frac{2P}{\pi D\left(D - \sqrt{D^2 - d^2}\right)}$$

BHN = Brinell hardness number

P = Maximum applied load (250 kg) D = Diameter of ball (5mm)
d = diameter of indentation (mm)

The unannealed PLA+ specimen shows a Brinell hardness number of 43.35 and for annealing specimen at a temperature of 55°C, holding time 30minutes, the Brinell hardness number is 51.87. For annealing specimen at a temperature of 65°C, holding time 30minutes, the Brinell hardness number is 62.41. For annealing specimen at a temperature of 75°C, holding time 30minutes, the Brinell hardness number is 76.26.



TEMPERATURE(°C)	EXPLANATION	RESULT
WOHT	As-printed PLA+ has limited crystallinity and interlayer fusion, resulting in lower resistance to indentation	Lower Number
55°C	Initial annealing starts relieving stress and increases bonding	Higher Number
	density, slightly improving surface	

	hardness.	
65°C	Close to glass transition temperature (Tg); promotes molecular rearrangement and crystalline growth, improving hardness.	Higher Number
75°C	Optimal annealing. Maximizes molecular packing and interlayer diffusion, leading to highest resistance against deformation.	Maximum Number

3.5 IMPACT TESTING

IZOD TEST RESULT

S.NO	HOLDING TEMPERATUTRE (◦C)	ENERGY OBSERVED (J)	IMPACT STRENGTH (KJ/M^2)
1	25	2	25
2	55	2	25
3	65	1.5	18.75
4	75	1.5	18.75

The unannealed PLA+ specimen shows an maximum Impact Strength of 25 KJ/M^2 and for annealing specimen at a temperature of 55°C, holding time 30minutes, the maximum Impact Strength is 25 KJ/M^2. For annealing specimen at a temperature of 65°C, holding time 30minutes, the maximum Impact Strength is 18.75 KJ/M^2. For annealing specimen at a temperature of 75°C, holding time 30minutes, the maximum Impact Strength is 18.75 KJ/M^2.

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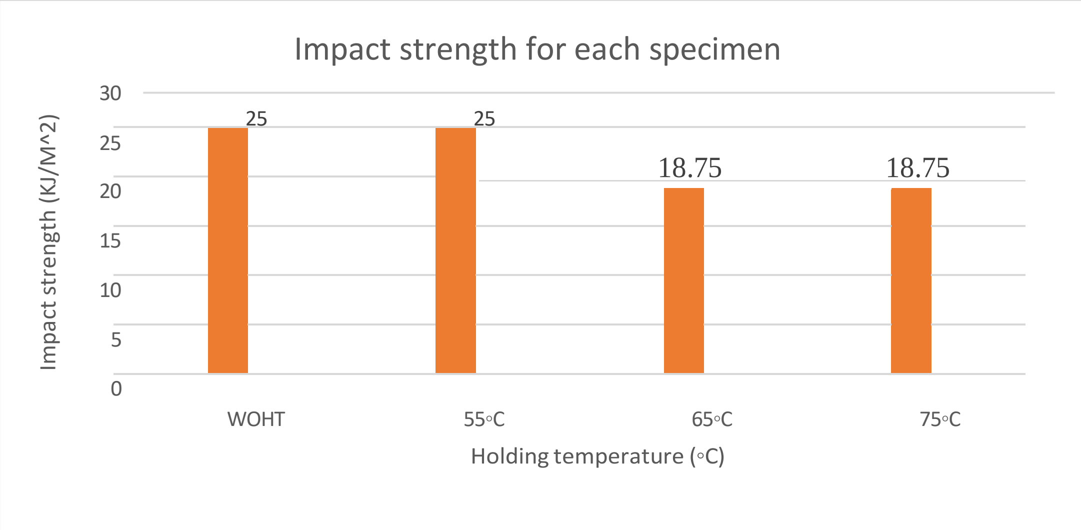

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At lower temperature (55°C), the polymer chains may experience minor rearrangement without a loss in ductility, preserving the material's energy absorption capacity.

At 65°C and 75°C, heat treatment may cause increased crystallinity. While this improves strength and hardness, it tends to reduce toughness, making the material more brittle and lowering its impact strength.

The Izod test measures the material's ability to resist sudden shock loading. More crystalline and brittle structures absorb less energy before fracture.

4. CONCLUSION

TEST TYPE	BEST TEMPERATURE	REASON
Tensile Strength	75°C	Highest tensile strength (6.816 MPa) – better interlayer bonding.
Compressive Strength	75°C	Highest compressive strength (2.124 MPa) – structural rigidity improved.
Flexural Strength	65°C	Highest flexural strength (28.421 MPa) – best resistance to bending.
Brinell Hardness	75°C	Highest surface hardness (76.26 BHN) – enhanced durability.
Impact Strength	WOHT/55°C	Highest impact resistance (25 kJ/m²) – retains toughness, less brittle.

It provides superior tensile, compressive, and hardness values.

While impact strength drops slightly, this is a typical trade-off for increased crystallinity and hardness in PLA+. If your application prioritizes strength, durability, and surface hardness, 75°C is optimal.
If impact resistance (toughness) is critical (e.g., for dynamic or impact-prone parts), 55°C may be better.

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