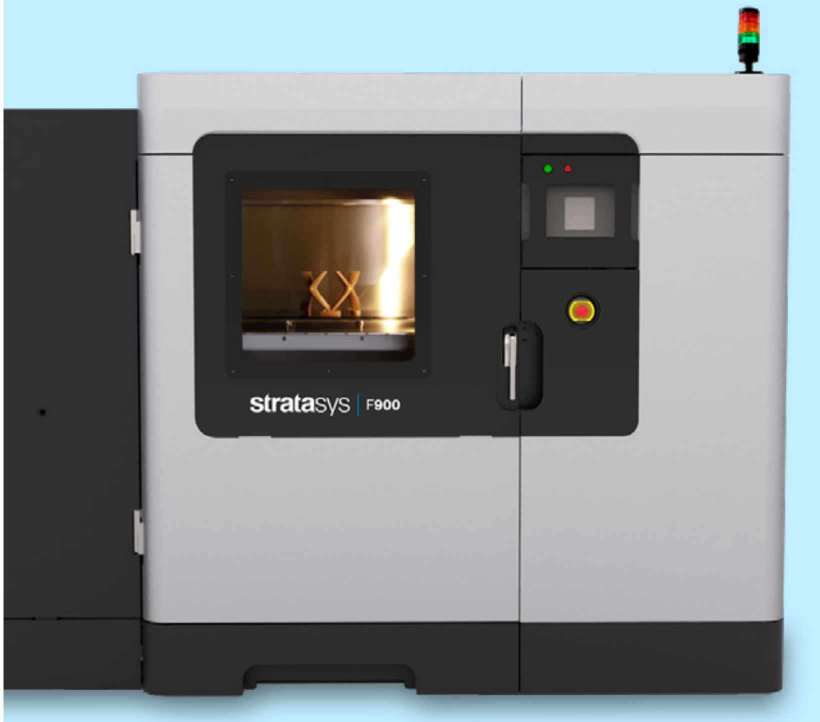
WHITE PAPER
FDM

VICTREX AM™ 200

Impact of Post-Print Annealing on Crystallinity



In the world of FDM® additive manufacturing, there are many post-processing techniques to help improve the performance of the printed part. One popular method involves annealing.

This paper will focus on determining the optimal annealing conditions for FDM 3D printed VICTREX AM™ 200. Additionally, it will discuss how those annealing conditions affect the material's dimensional stability and overall crystallinity.

VICTREX AM 200

The material selected for this study was VICTREX AM™ 200. This unique material is in the PolyArylEtherKetone (PAEK) polymer family making it very similar to PEEK and PEKK. Victrex PLC, the maker of VICTREX AM™ 200, optimized the crystallization rate of this material to promote gradual crystallization while printing with FDM. Traditional PEEK materials do not perform effectively when 3D printed due to the fast crystallization rate. This means that when a bead of material is being extruded by the print head, it starts to crystallize very shortly after being deposited. Once crystallized, any additional beads of extruded material will have difficulty adhering to the previous layer, resulting in a very weak part or unbonded layers. With VICTREX AM™ 200, this gradual rate of recrystallization allows for the new beads of material to better bond with previous layers, which enables it to easily be used in FDM 3D printing.

Another unique characteristic of this material is the ability to be printed in either a more amorphous state or a more semicrystalline state. When using SR-100™ soluble support material, it prints more semicrystalline. However, when using SUP8000B™ breakaway support material, it prints more amorphously. Furthermore, this material can be post-crystallized by annealing the part after printing has finished. This paper will dive further into annealing VICTREX AM™ 200 and present a few options for ways that it can be used.

Annealing

Annealing is a common post-processing technique used in various industries to increase the performance of a manufactured part. Often seen with metals, this process also works with most polymers. Annealing is the process of heating a material to a specific temperature and maintaining that temperature for a set amount of time. During this period, the molecular structure of the material, particularly the polymer chains, will relax and realign into a lower energy state. In polymers, this can lead to a more crystalline structure as the polymer chains rearrange.

The process of annealing is relatively straightforward, with two key variables: the temperature at which the material is annealed and the duration for which it is held at that temperature. In the case of semicrystalline polymers, the annealing temperature is typically chosen based on the glass transition temperature (Tg) and the melting temperature (Tm), selecting a value that falls between these two. Choosing a temperature too close to Tg may not allow sufficient crystallization for effective annealing, while selecting a temperature near Tm can result in excessive crystallinity, potentially leading to warping or deformation of the material.

This study focused on three different temperatures for two different annealing times. These temperatures were provided by Victrex and were 170 °C (338 °F), 185 °C (365 °F), and 200 °C (392 °F). The selected temperatures fall between Tg and Tm with those temperatures respectively being 151 °C (303.8 °F) and 303 °C (577.4 °F). The two annealing times that were used were two hours and four hours.

File Preparation and Printing

To ensure a consistent and accurate study, the file preparation and printing was done using the same method for all parts. This includes consistency in how the printing files were generated, the machine was set up, and how the parts were post-processed after printing.

File Preparation

The files used in this study include both dimensional-measurement parts and physical-property test parts.
The dimensional analysis parts are parts that Stratasys uses for material characterization on its FDM printers and include an X/Y dimensional accuracy part, a Z dimensional accuracy part, and an angled wall warp measurement part. An additional flat warp measurement part was also used to further characterize the deformation of the annealing process. The physical test samples were D648 deflection temperature bars which were used to test changes in crystallinity.

STL files were loaded into Insight™ 18.10 software that was configured for an F900® 3D printer using VICTREX AM™ 200, SR-100 support, a T20F model tip, a T12SR100 support tip, and a 0.254 mm (0.010 in.) layer height. All models were then processed using default settings. Once processed, they were loaded into Control Center™ 18.10. Packs of parts were generated within Control Center with dimensional parts being saved in packs of six and the physical-analysis parts being saved in packs of ten as seen in Figure 1. These packs were then individually sent to the printer for printing.

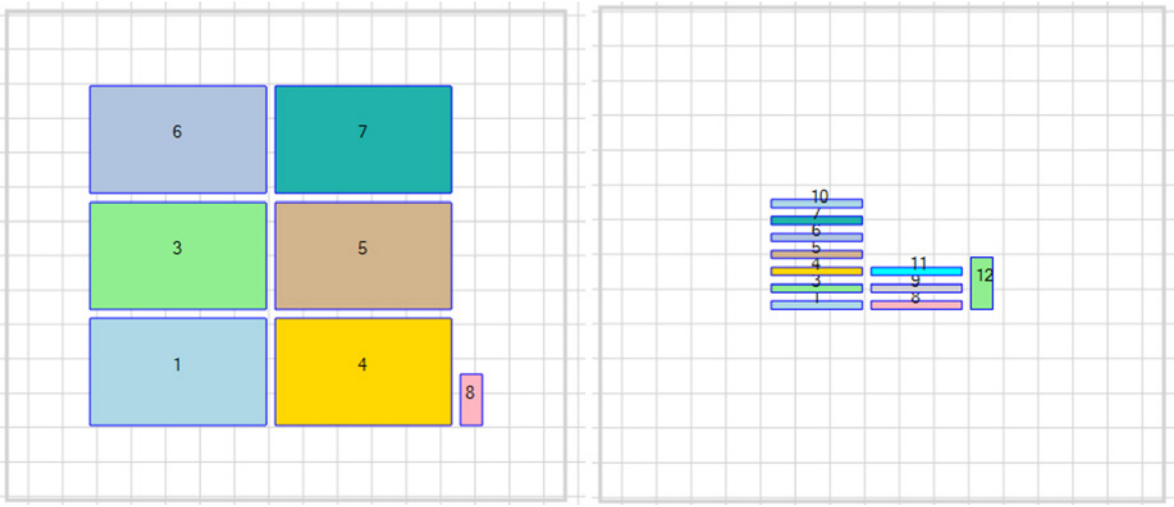


Figure 1: Print layout for dimensional parts (left) and physical-analysis (right).

Printing

Printing was performed on a Stratasys F900 3D printer using a T20F tip, SR-100 soluble support, and a high temperature build sheet.

After printing, the parts were tanked for four hours in an Oryx water bath at 70 °C (158 °F) using a Waterworks™ solution to remove any support material. Upon removal from the tank, the parts were rinsed in clean tap water and then dried on a drying rack using fans to circulate room temperature air.

Pre-Annealing Analysis

Before annealing any of the parts, a pre-analysis was done to determine a dimensional and physical baseline.

Each dimensional part was randomly assigned a number 1 through 6 and then the as printed dimensions were measured using each part's own specific internal measurement method. These initial measurements were used as the reference point for all future measurements on the parts. Additionally, this was done for all X/Y/Z dimensional parts and angled wall warp measurement parts.

The flat warp measurement parts were printed as a single build of six plaques. As with the dimensional parts, these parts were randomly assigned a number of 1 through 6. Then, on the top surface, eight points were labeled where measurements were to take place as seen in Figure 2. These flatness measurements were recorded and used as the reference point for future measurements.

Figure 2: Flat plaque numbering.

The physical baseline was used to find the starting crystallinity of the material. This was done by testing a piece of unannealed printed material using Differential Scanning Calorimetry (DSC), as well as testing a Heat Deflection Temperature (HDT) coupon in a Dynamic Mechanical Analyzer (DMA).

Annealing

After concluding the pre-annealing analysis on all parts, the annealing process could begin. This was done in a standard laboratory oven using sand as a medium to help reduce the thermal strain on the parts while annealing.

Three temperatures and two annealing times were chosen for this study. The temperatures were 170 °C (338 °F), 185 °C (365 °F), and 200 °C (392 °F), with the two times being two hours and four hours. Table 1 shows which sample set number is associated with the corresponding annealing temperature and time.

Table 1: Annealing Set Temperature and Time

Set Number	Temperature (°C)	Time (hr.)
1	170	2
2	170	4
3	185	2
4	185	4
5	200	2
6	200	4

The first parts to be annealed were the dimensional parts. This was done by separating each type of dimensional part into sets that correlated with their number. For example, all dimensional parts with a 1 were placed together, all parts with a 2 were placed together, etc. Along with the dimensional parts, HDT parts were numbered 1 through 6 and annealed with the respective sample set.

To prepare the parts to go into the oven, a base layer of 12 mm (0.5 in.) of sand was evenly spread at the bottom of a 229 x 229 mm (9x9 in.) metal pan to create a solid bottom layer of sand for the parts to sit on. Next, a single set of parts was placed on top of the sand, with each part spaced so that none of them were touching. Sand was then poured on top of the parts to cover them completely until there was 12 mm (0.5 in.) of sand covering them all. The pan was then placed in a room temperature oven that was turned on and set to the desired temperature. When the oven warmed up completely, an external timer was started to track the duration of the annealing process. After the allotted time elapsed, the oven was turned off allowing it to slowly cool the parts in the oven. Finally, when the oven cooled to room temperature, the pan was removed and all parts were taken out of the sand and placed in a labeled bag. This procedure was repeated for all sample sets.

Post-Annealing

When all annealing trials were completed, the parts for each trial were cleaned of any loose sand using soft brushes and compressed air. After cleaning, they were remeasured to gather data on potential dimensional changes.

Along with dimensional measurements, the change in crystallinity was captured using both DSC and DMA. DSC was used to show the overall change in crystallinity of the samples and to determine which samples crystallized the most. The DMA was used to look at the change in HDT and compare that to the original values. When assessing the HDT, it can be reasonably assumed that a higher HDT indicates a higher percentage of crystallinity in the part. For this study, HDT was captured and analyzed, but the results from the DSC were used to ultimately determine which annealing method had the most crystalline parts, as it provides a more direct and quantitative measure of crystallinity.

Due to the increase in crystallinity of the annealed parts, there was an observable color change in the parts. Non-annealed parts have a darker tan or caramel color. After annealing, this color lightens up to a light tan color as seen in Figure 3.

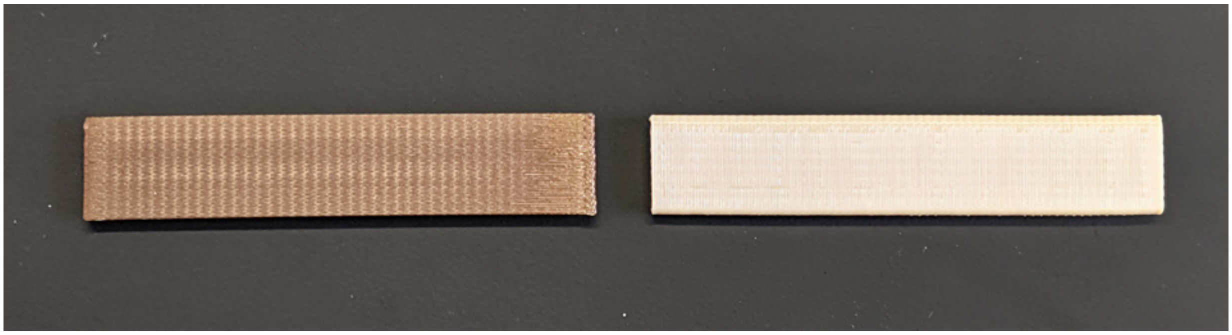


Figure 3: Non-annealed part (left), annealed part (right).

Results

After observing any dimensional changes and changes in crystallinity, the results were compared with one another to determine the optimal annealing processes based on the users needs. If parts were very dimensionally accurate but there wasn't a significant change in crystallinity or if parts were highly crystalline but not dimensionally stable, then those processes may not be recommended. Ultimately, three annealing processes were chosen, which include the most dimensionally accurate, the most crystalline, and an in-between of increased crystallinity and dimensional accuracy.

Dimensional Stability

The dimensional stability was found by measuring two distinct categories – shrink/expansion and warp/deformation. These measurements were found using several different geometries that each highlight the specific categories. To gather the dimensional data, dimensional-accuracy parts were remeasured after annealing and all the values were compared to the original baseline measurement. For warp, two parts were selected. One was an angled wall, and the other was a flat plaque. As with the dimensional parts, these parts were measured pre- and post-annealing and compared to the baseline measurements.

Shrink / Expansion

This first category discusses how much the part shrinks or expands due to changes in crystallinity from annealing. From the post annealing dimensional data that was gathered, most parts shrank in the X and Y directions and grew in the Z direction while annealing. Tables 2 and 3 show the degree of shrink/growth that each annealing trial produced.

Table 2 goes over the change in dimension in both the X and Y axis. Areas highlighted in red show the greatest amount of shrinkage while areas in green show the least amount of shrinkage.

Table 2: X and Y Axis Dimensional Changes in Inches

Measurement	Set 1 170 °C, 2 hrs	Set 2 170 °C, 4 hrs	Set 3 185 °C, 2 hrs	Set 4 185 °C, 4 hrs	Set 5 200 °C, 2 hrs	Set 6 200 °C, 4 hrs
1A	-0.0023	-0.0046	-0.0048	-0.0046	-0.0043	-0.0046
1B	-0.0011	-0.0052	-0.0047	-0.0049	-0.0046	-0.0050
2A	-0.0154	-0.0322	-0.0325	-0.0245	-0.0318	-0.0050
2B	-0.0178	-0.0378	-0.0394	-0.0341	-0.0391	-0.0485
3A	-0.0399	-0.0487	-0.0615	-0.0504	-0.0659	-0.0420
3B	-0.0308	-0.0491	-0.0555	-0.0512	-0.0562	-0.0567
3C	-0.0295	-0.0592	-0.0476	-0.0607	-0.0422	-0.0655
4	-0.0021	-0.0021	-0.0030	-0.0023	-0.0042	-0.0027
5	-0.0079	-0.0126	-0.0119	-0.0078	-0.0131	-0.0103
6	-0.0024	-0.0035	-0.0037	-0.0022	-0.0044	-0.0036
7	-0.0019	-0.0038	-0.0008	-0.0034	-0.0014	-0.0012
8	-0.0089	-0.0153	-0.0149	-0.0088	-0.0137	-0.0081
9	-0.0132	-0.0195	-0.0164	-0.0109	-0.0201	-0.0162
10	-0.0035	-0.0051	-0.0012	-0.0042	-0.0034	-0.0082
11A	-0.0334	-0.0349	-0.0301	-0.0232	-0.0333	-0.0301
11B	-0.0246	-0.0326	-0.0282	-0.0227	-0.0304	-0.0307
12	-0.0120	-0.0174	-0.0176	-0.0121	-0.0190	-0.0143
13	-0.0074	-0.0203	-0.0151	-0.0104	-0.0113	-0.0125
14	-0.0045	-0.0069	-0.0062	-0.0063	-0.0074	-0.0077
15	-0.0082	-0.0189	-0.0158	-0.0149	-0.0160	-0.0164
16	-0.0093	-0.0195	-0.0147	-0.0152	-0.0179	-0.0182
17	-0.0065	-0.0129	-0.0115	-0.0094	-0.0113	-0.0134
18A	-0.0004	-0.0013	-0.0028	-0.0053	-0.0019	-0.0042
18B	-0.0006	-0.0039	-0.0027	-0.0045	-0.0033	-0.0053
19A	-0.0398	-0.0480	-0.0393	-0.0303	-0.0474	-0.0448
19B	-0.0285	-0.0400	-0.0401	-0.0389	-0.0428	-0.0414

From this table, it shows that each measured dimension shrank during the annealing process in both the X and Y directions as demonstrated in Figure 4. Set 1 and set 4 show the least amount of overall shrink while sets 2, 5, and 6 appear to show the greatest amount of shrink.

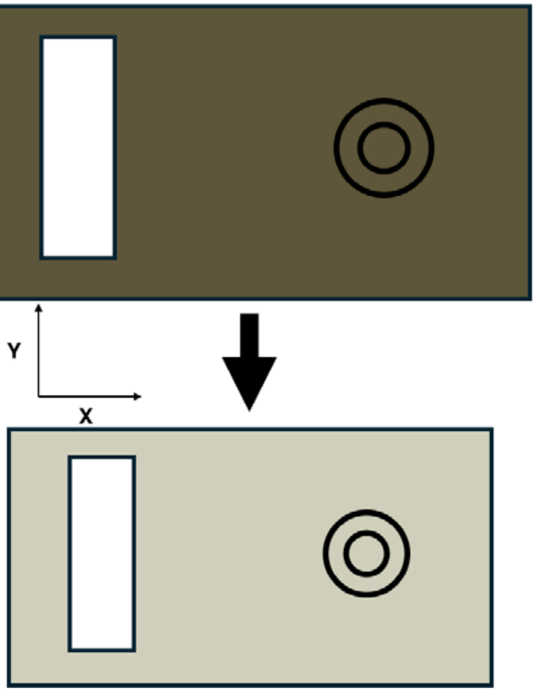


Figure 4: X and Y dimensional part shrink.

In addition to measuring the X and Y dimensions of these parts, measurements in the Z direction were also collected using calipers and are shown in Table 3 below. Similar to the X and Y measurements, set 1 and set 4 show low Z dimensional changes. Additionally, set 2 did not have significant changes to the Z height. Overall, all sets increased in Z height after annealing.

Table 3: Z Axis Dimensional Measurement Changes in Inches

Measurement	Set 1 170 °C, 2 hrs	Set 2 170 °C, 4 hrs	Set 3 185 °C, 2 hrs	Set 4 185 °C, 4 hrs	Set 5 200 °C, 2 hrs	Set 6 200 °C, 4 hrs
1	0.0045	0.0045	0.0105	0.0035	0.0085	0.0065
2	0.0055	0.0035	0.0045	0.0055	0.0105	0.007
3	0	-0.001	0.001	0	0.002	0.0005
4	0.0015	0	0.002	0	0.0045	0.003
5	0.003	0.0025	0.0075	0.003	0.003	0.005
6	0.004	0.002	0.0025	0.005	0.003	0.0045
7	0.0025	0.003	0.004	0.005	0.005	0.0065
8	0.002	0.002	0.0025	0.0005	0.001	0.002
9	0.0005	0.001	0.002	-0.0005	0.0025	0.0025
10	0	0.002	0.0055	0.0005	0.0035	0.0005

To simplify the data a little further, the average of each dimension type was taken and compared to each other. To do this, all dimensions for outside X, inside X, outside Y, inside Y, outside diameter, inside diameter, and Z height were averaged for each set. Figure 5 shows how the average of each set compares to one another.

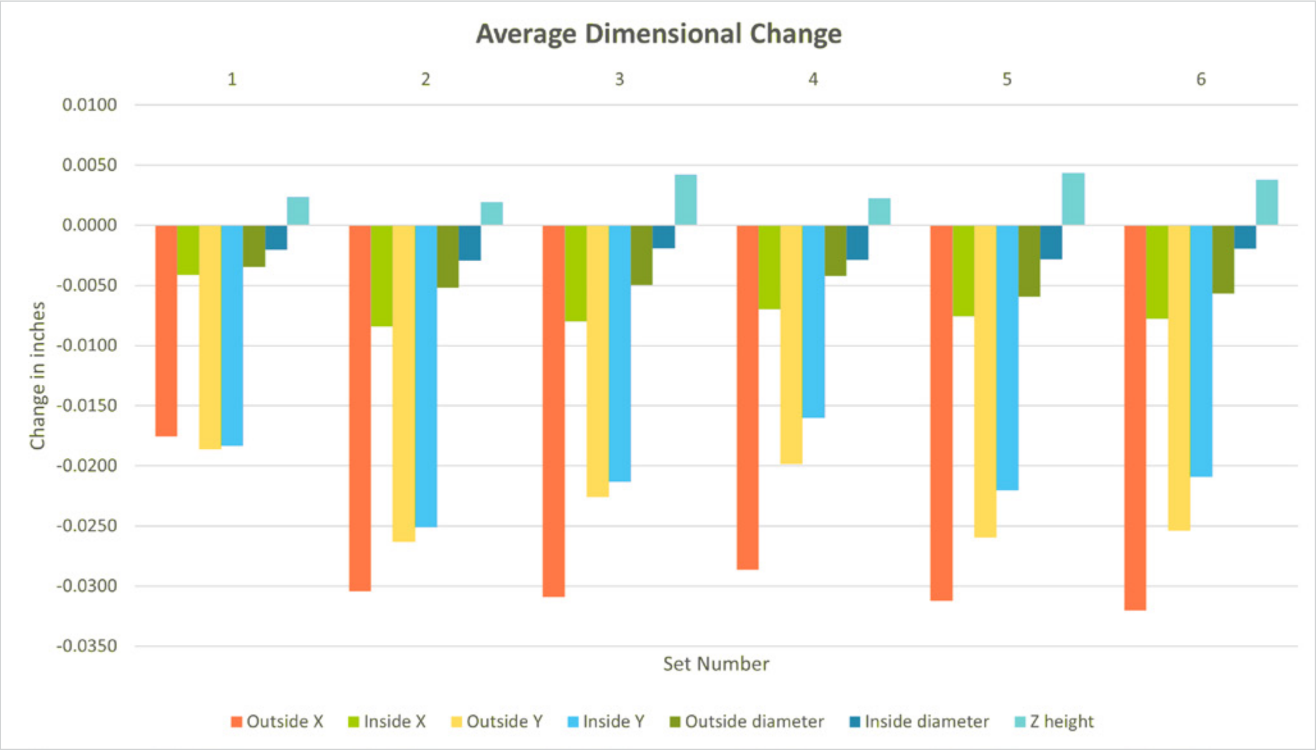


Figure 5: Average dimensional change in X, Y, and Z

The chart in Figure 5 can help clarify which parts had the best X/Y/Z dimensional stability overall. Set 1 still shows the least amount of shrink for all features with set 4 being just behind set 1. All sample sets show a similar change in Z with this particular model.

To further analyze the change in Z, a different and more appropriate model was printed and measured. This dimensional test part looks like a set of stairs with five steps, and each step one inch tall as shown in Figure 7. As with the other dimensional test parts, measurements were taken pre and post annealing and can be seen in Figure 6.

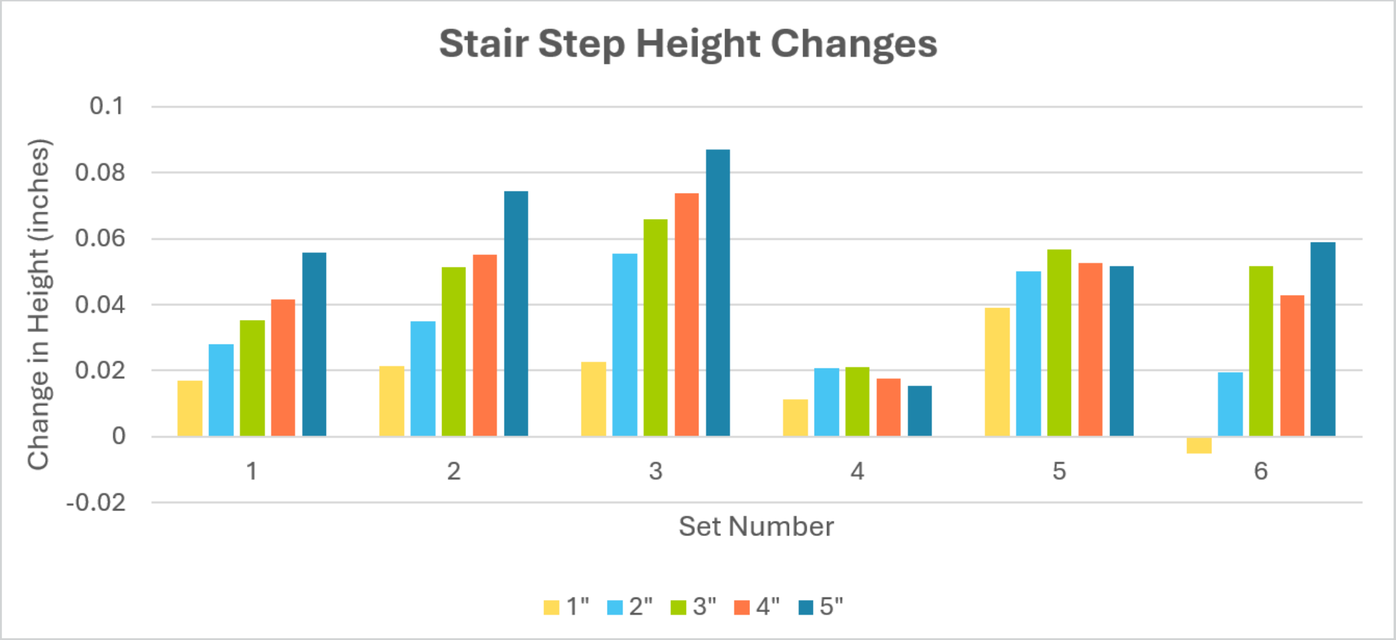


Figure 6: Z Height measurement with stair step part

This chart clearly shows that sample set 4 had the least amount of dimensional change in Z, while set 3 appears to have the most. Set 6 is the only set to show some shrink with the first step shrinking slightly, but the rest of the measurements indicate that all samples grew in Z height after annealing. From the given dimensional measurements, one can conclude that during the annealing process the parts tend to shrink in the X and Y directions and grow in the Z direction as demonstrated in Figure 7. One possible explanation is that during annealing, the polymer chains relax and pull the material inward, causing shrinkage in the X and Y directions. Since the material volume stays constant, this inward contraction can force the layers to compress and bunch up slightly in the Z direction. This cumulative effect across layers can result in a noticeable increase in the part's height.

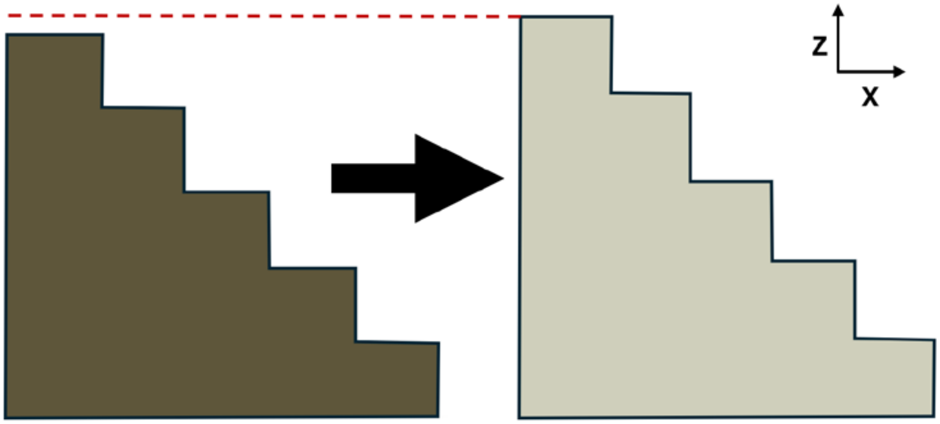


Figure 7: Z dimensional part growth.

Warp and Deformation

When the material heats up in the oven it is possible that it could start to deform in a way commonly called warp. This is where the part will move and distort away from the intended shape and is often due to inadequate cooling or unfavorable thermal conditions. This defect is typically seen on large unsupported overhangs or in smaller thin walls.

Ideally, there would be no warp or distortion when annealing a part, but due to the thermal nature of the annealing process and the rapid increase in crystallinity, this can be very difficult to minimize. Because this defect is hard to avoid, it was a key feature measured when annealing the parts in an effort to find a process that minimizes warp.

To find this data, two tests were performed. First, an angled wall part was printed and measured to determine the flatness. This was done using an internal measurement process used by Stratasys to characterize materials and printing profiles. The part consists of a thin vertical wall, a small flat base, and a thin angled wall – like a check mark, but with a flat base instead of a point. The vertical and angled walls are 76.2 mm (3 in.) tall, the base is 12.7 mm (0.5 in.) wide, and the total length is 152.4 mm (6 in.). Each wall on this part is about 3.175 mm (0.125 in.) thick.

The second test involved printing a single flat 127 x 127 x 6.35 mm (5 x 5 x 0.25 in.) plaque. The flatness of the plaque was measured at eight different points around the edges, as seen earlier in Figure 2, using a height gauge to see how much the part warped during the initial printing process. The parts were then annealed and measured again to record the amount of distortion from the original value. Analyzing both of these parts will show how much the annealing process affects small, thin-walled parts with the angled wall part, as well as thicker, bulkier parts with the flat plaque as demonstrated in Figures 8 and 9.

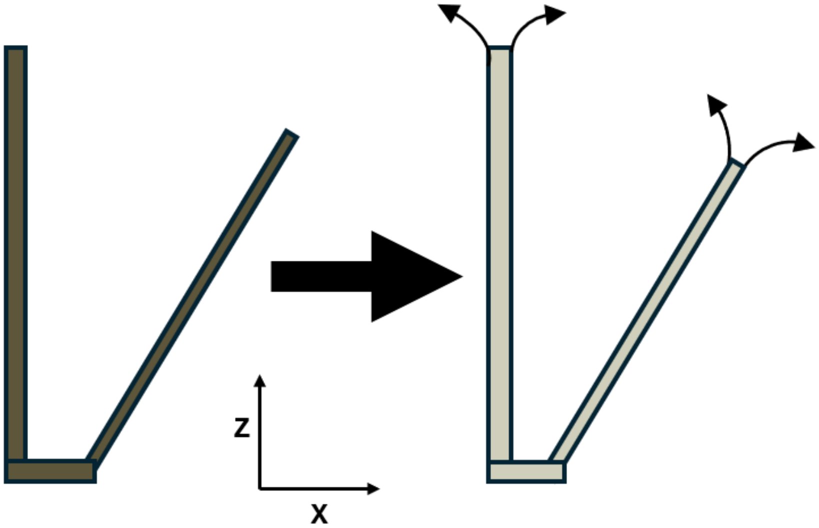


Figure 8: Angled wall warp/deformation movement

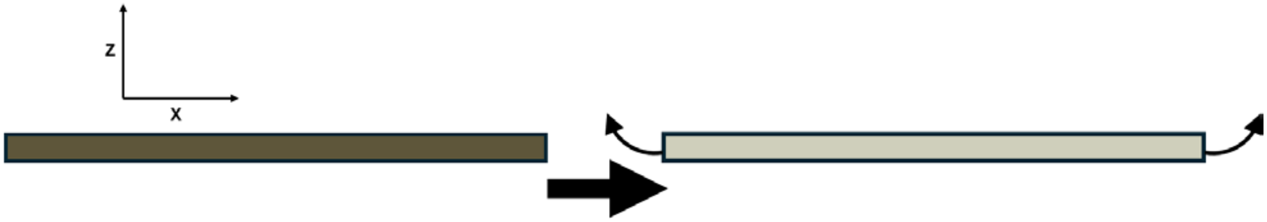


Figure 9: Flat plaque warp/deformation movement

Table 4: Angled Part Flatness

Set Number	Original Flatness (in.)	Annealed Flatness (in.)	Change in Flatness (In.)	Change in Flatness (%)
1 (170 °C, 2 hrs)	0.00386	0.0204	0.01654	81.08%
2 (170 °C, 4 hrs)	0.00396	0.0297	0.02574	86.67%
3 (185 °C, 2 hrs)	0.00455	0.01986	0.01531	77.09%
4 (185 °C, 4 hrs)	0.00398	0.00708	0.0031	43.79%
5 (200 °C, 2 hrs)	0.00404	0.08171	0.07767	95.06%
6 (200 °C, 4 hrs)	0.00423	0.01451	0.01028	70.85%

For the angled wall, Table 4 shows the degree of warp. The data shows a severe degree of warp when printing a very thin wall as seen on the angled part with all sets showing a change in flatness of about 44% – 95% of the original. Although the overall dimensional change for these thin walls was small, the percentage of warp based on the total size of the wall was significant. This shows that when printing a part with a post-annealing intent, the minimum wall thickness should be increased. Otherwise, some warp should be expected.

For the flat plaque (Figure 2, page 4), Table 5 and Figure 10 highlight how the annealing processes change the degree of warp. Table 5 averages the change in flatness of the eight measurement points of the flat plaque to simplify the data. The cells in the table are color coded in a way where the deeper green shows the lowest change while the deeper red shows the greatest change.

Table 5: Average Change in Flatness of the Flat Plaque

Set Number	Change in Flatness (in.)	Percentage
1 (170 °C, 2 hrs)	0.0358	11.92%
2 (170 °C, 4 hrs)	0.0219	7.58%
3 (185 °C, 2 hrs)	0.0168	5.93%
4 (185 °C, 4 hrs)	0.0224	7.73%
5 (200 °C, 2 hrs)	0.0159	5.59%
6 (200 °C, 4 hrs)	0.0266	9.02%

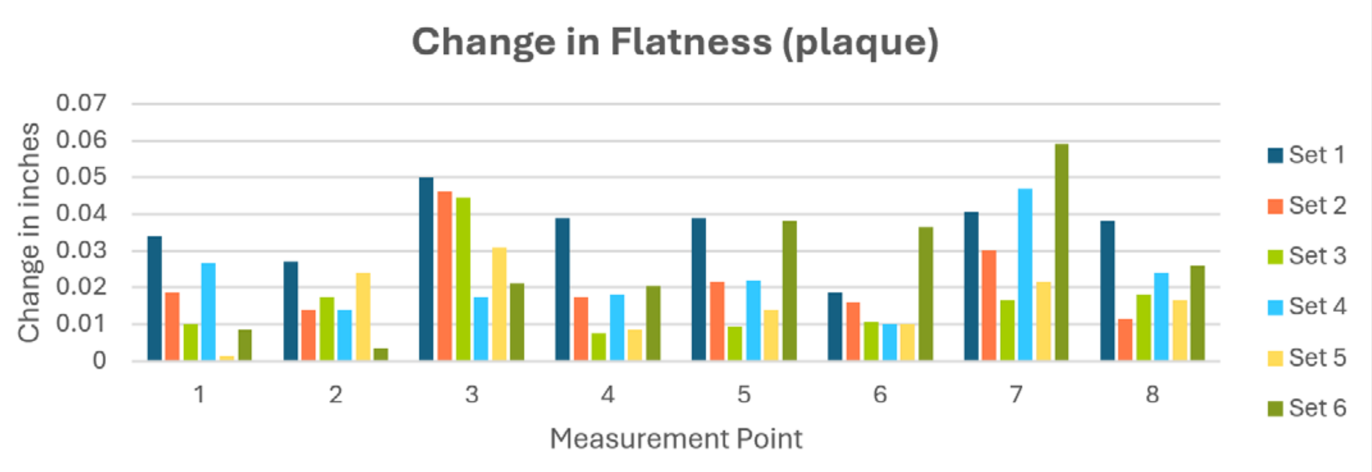


Figure 10: Change in flatness for flat plaque.

From the data in the table and chart, one can see that there is a clear difference between the thinner and thicker parts in terms of degree of warp or distortion. The thicker plaque showed a similar degree of distortion in terms of inches, but the overall percentage based on the thickness of the part was much smaller. This indicates that with a larger part there could still be the occurrence of warp or distortion, but that relative to the size of the part it is less severe compared to thin parts.

Change in Crystallinity

To see the change in crystallinity of the material after annealing, there were two instruments used: DSC and DMA. Through both instruments, one can analyze how the material changes by comparing physical and thermal properties before annealing to the properties after annealing.

DSC

When analyzing material properties using DSC, one can typically find values for the glass transition, cold crystallization, melt temperature, and recrystallization temperature. In this study, this instrument was used to find the melt temperature and the energy required to melt the material. The theory behind this is that the different degrees of crystallinity will require different amounts of energy to remelt the polymer. In this context, the amount of energy required to melt the unannealed material will be less than that of a material that has greater crystallinity from annealing.

The change in crystallinity was analyzed using data from a heat–cool–heat cycle on a DSC instrument. During this process, several thermal properties were recorded, including the cold crystallization onset and enthalpy, melting temperature and enthalpy, and recrystallization onset and enthalpy. Graphs generated during DSC can be seen in the Appendix. From this data, the change in crystallinity can be calculated using the formula below.

$$\text{Crystallinity (\%)} = \frac{\text{Melt Enthalpy-Cold Crystallization Enthalpy}}{\text{Theoretical Melt Enthalpy}}$$

Additionally, for some of the annealed samples there was no cold crystallization. In this occurrence, the equation simplifies to:

$$\text{Crystallinity (\%)} = \frac{\text{Melt Enthalpy}}{\text{Theoretical Melt Enthalpy}}$$

These calculations were performed while assuming a theoretical melt enthalpy of 130 J/g for PAEK. Figure 11 illustrates the degree change in crystallinity for the annealed samples.

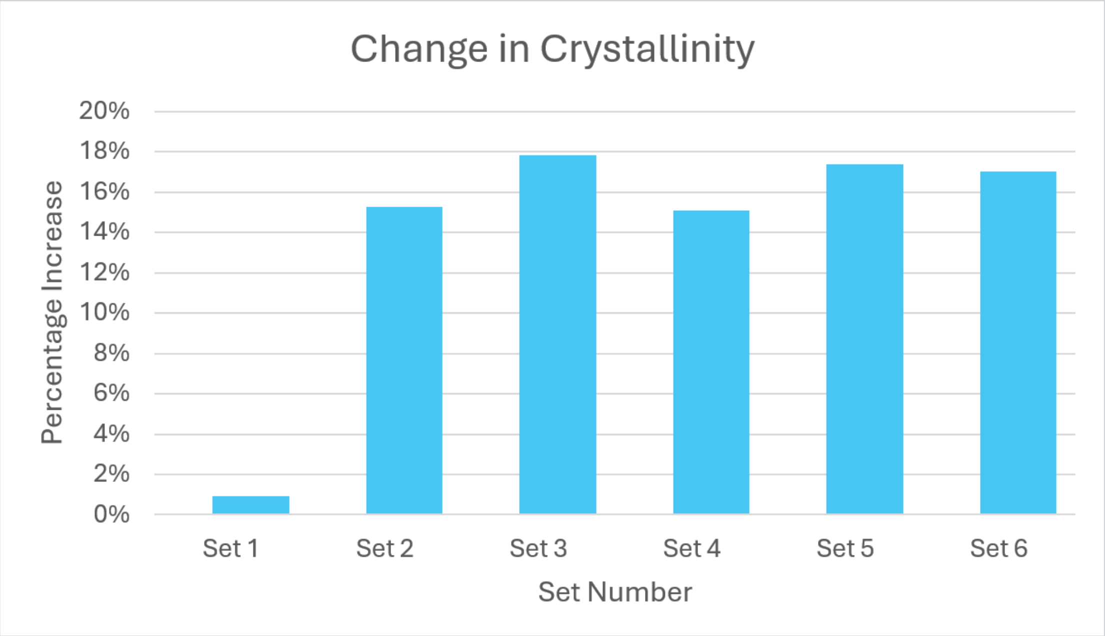


Figure 11: Change in crystallinity per set.

From the chart, it is clear that there is a drastic increase in total crystallinity of the material after annealing for four hours at 170 °C (338 °F), as seen with set 2 through set 6. After annealing at those conditions, the overall change was not significant, with all the other annealing conditions producing results within a few percentage points of one another. This indicates that the material will become as crystalline as it can when choosing any of the annealing settings from sets 2 through 6.

DMA

Analysis using DMA is less straightforward than that of DSC but can still be used as a way to observe the change in crystallinity. With this instrument, the heat deflection temperature (HDT) was captured for the material unannealed and after each round of annealing. The goal of collecting this data was to see how the HDT changed with annealing. In theory, the HDT should increase as the degree of the material's crystallinity increases. Although this method is less technical, it can still be valuable when paired with the data from DSC.

Figure 12 displays how the HDT of the annealed samples changed in response to their given annealing temperature and time. Similar to the chart showcasing the change in crystallinity, this graph shows that there was not a significant increase in HDT when annealing with the settings for set 1. Unlike the change in crystallinity, the change in HDT only increases with increased temperature and times of the higher set numbers. This is valuable information because even a few degree increase in HDT could potentially open up more applications for the material. For reference, the HDT of the unannealed sample was 144.6 °C (292.3 °F) and increased to 169.8 °C (337.6 °F) with set 6. The DMA charts for each set can be viewed in the Appendix.

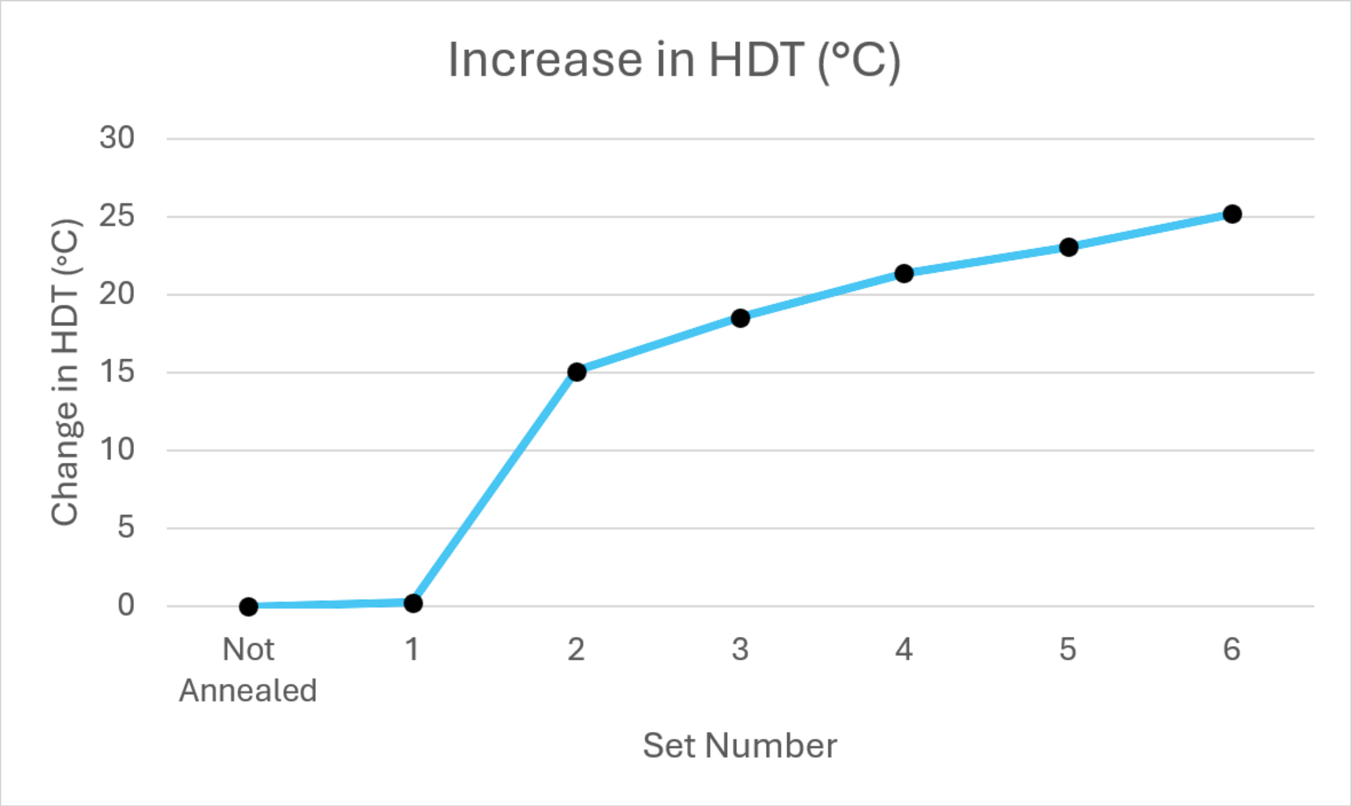


Figure 12: Increase in HDT per set

Optimal Process

When considering the optimal annealing process, one has to think of what is most important for their final part or design. Is dimensional stability critical for the part, or does the total crystallinity have higher priority? Is there a happy medium between dimensional stability vs. crystallinity that will be best? Will the part be machined to the final form so the amount of crystallinity is most important? These types of questions must be thought about when considering the annealing process, and for this reason, this study will highlight three categories: best dimensional, mixed dimensional and crystallinity, and most crystallinity.

Best Dimensional

If dimensional stability is the most important factor for the annealed parts, then there are a few options that can be chosen. If the parts are thicker, which could make them less prone to warping during annealing, then using a higher temperature and time could work well. If they are thinner, then staying on the lower end of temperature or time would be the better option. When looking at the data collected for dimensional stability, it shows that selecting a temperature of 170 °C (338 °F) and annealing for two hours as seen in set 1 will keep the dimensions relatively stable, but it will only increase the crystallinity minimally. For a slightly higher degree of crystallinity, one can increase the annealing time to four hours, as seen in set 2, to get a better increase in crystallinity of about 15% while still being mostly dimensionally stable.

Mixed Dimensional and Crystallinity

This category would need focus on how dimensionally stable the parts are along with considering how much the part crystallized while annealing. This could be applicable for parts that have a lower fit tolerance, or for parts that might have some degree of post processing to help them fit to the final application.

Sets 3 and 5 seemed to fit this fairly well with both having some change in dimensions while showing an increase in crystallinity. Both showed an increase in crystallinity by about 17% - 18% and both show similar results in the change in X and Y dimensions. Sample set 5 appears to have a more dimensionally stable Z measurement which could indicate that this would be the best choice as a middle ground for crystallinity and dimensional stability.

Best Crystallinity

For this category, the overall increase in crystallinity is the priority. This could be used for parts that have a very loose tolerance, or for parts that were designed in a way to allow for the change in dimensions. Additionally, parts that may be machined or cut down to final shape could also be annealed using this process.

Based on the overall change in crystallinity, sample sets 3, 5, and 6 all showed the greatest increase at 17% – 17.8%. Of these, sets 5 and 6 also show the greatest rise in HDT with an increase of 23.05 °C (73.49 °F) and 25.18 °C (77.32 °F) respectively. Because of the increase in HDT of sample sets 5 and 6, annealing at 200 °C (392 °F) for two to four hours would be the best options for the greatest change in crystallinity and increase in physical property performance.

Cause of Error and Future Learnings

With any type of study, there is always a chance that there was some error when collecting the data. For this study in particular, there are a few areas that could be improved upon if this was to be repeated.

First, the lab oven used for this study was not designed to anneal parts. It was a simple lab oven that allowed the user to set a temperature that would hold a consistent temperature until turned off. Ideally, an oven that allowed for a slow temperature ramp-up followed by a slow cooldown would be optimal. This would allow the tester to increase the temperature very slowly, which would minimize any distortion by slowing down the crystallization rate of the part.

Another area that may have introduced some errors is the conditioning state of the parts. For example, if the parts were sitting out in an environment that was higher in humidity, then parts that were not tested until a later date may have had more moisture in them which could have caused some error in testing.

One last area could be sample preparation. Although each set of parts was annealed the same way, variations in the amount of sand used to cover the parts may have introduced some error. Additionally, part placement in the oven, whether they were closer to the front, back, left, or right could also play a role if the oven had areas of temperature variability.

Conclusion

Annealing significantly influences the dimensional stability, crystallinity, and thermal performance of FDM parts printed with VICTREX AM™ 200. By selecting appropriate annealing temperatures and durations, users can tailor part properties to meet specific application requirements. For dimensional accuracy, lower annealing temperatures with gradual cooling are recommended. For enhanced crystallinity and HDT, higher annealing temperatures and times yield optimal results. This study provides a framework to optimize post-processing strategies based on performance goals.

Appendix

Dimensional changes:

Average Dimensional Changes in Inches

Set Number	Outside X	Inside X	Outside Y	Inside Y	Outside diameter	Inside diameter	Z height
1	-0.0176	-0.0041	-0.0186	-0.0183	-0.0034	-0.0020	0.00235
2	-0.0304	-0.0084	-0.0263	-0.0251	-0.0052	-0.0029	0.00195
3	-0.0309	-0.0080	-0.0226	-0.0213	-0.0050	-0.0019	0.0042
4	-0.0286	-0.0070	-0.0198	-0.0160	-0.0042	-0.0028	0.00225
5	-0.0312	-0.0076	-0.0259	-0.0220	-0.0059	-0.0028	0.00435
6	-0.0320	-0.0077	-0.0254	-0.0209	-0.0057	-0.0019	0.0038

Change in Z Height in Inches

	Set 1 170 °C, 2 hrs	Set 2 170 °C, 4 hrs	Set 3 185 °C, 2 hrs	Set 4 185 °C, 4 hrs	Set 5 200 °C, 2 hrs	Set 6 200 °C, 4 hrs
Left side top	0.0045	0.0045	0.0105	0.0035	0.0085	0.0065
Left side bottom	0.0055	0.0035	0.0045	0.0055	0.0105	0.007
Bottom left	0	-0.001	0.001	0	0.002	0.0005
Bottom middle	0.0015	0	0.002	0	0.0045	0.003
Bottom right	0.003	0.0025	0.0075	0.003	0.003	0.005
Right side bottom	0.004	0.002	0.0025	0.005	0.003	0.0045
Right side top	0.0025	0.003	0.004	0.005	0.005	0.0065
Top right	0.002	0.002	0.0025	0.0005	0.001	0.002
Top middle	0.0005	0.001	0.002	-0.0005	0.0025	0.0025
Top left	0	0.002	0.0055	0.0005	0.0035	0.0005

Change in Height (in.) Stair Step

Height	Set 1 170 °C, 2 hrs	Set 2 170 °C, 4 hrs	Set 3 185 °C, 2 hrs	Set 4 185 °C, 4 hrs	Set 5 200 °C, 2 hrs	Set 6 200 °C, 4 hrs
5"	0.05575	0.07422	0.08709	0.01552	0.05159	0.05902
4"	0.04143	0.05507	0.07361	0.0177	0.0527	0.04272
3"	0.03541	0.05145	0.06583	0.0212	0.05669	0.05157
2"	0.02805	0.03481	0.05537	0.02068	0.05017	0.01955
1"	0.01714	0.02146	0.02264	0.01123	0.03911	-0.0052

Change in Flatness (Flat Plaque)

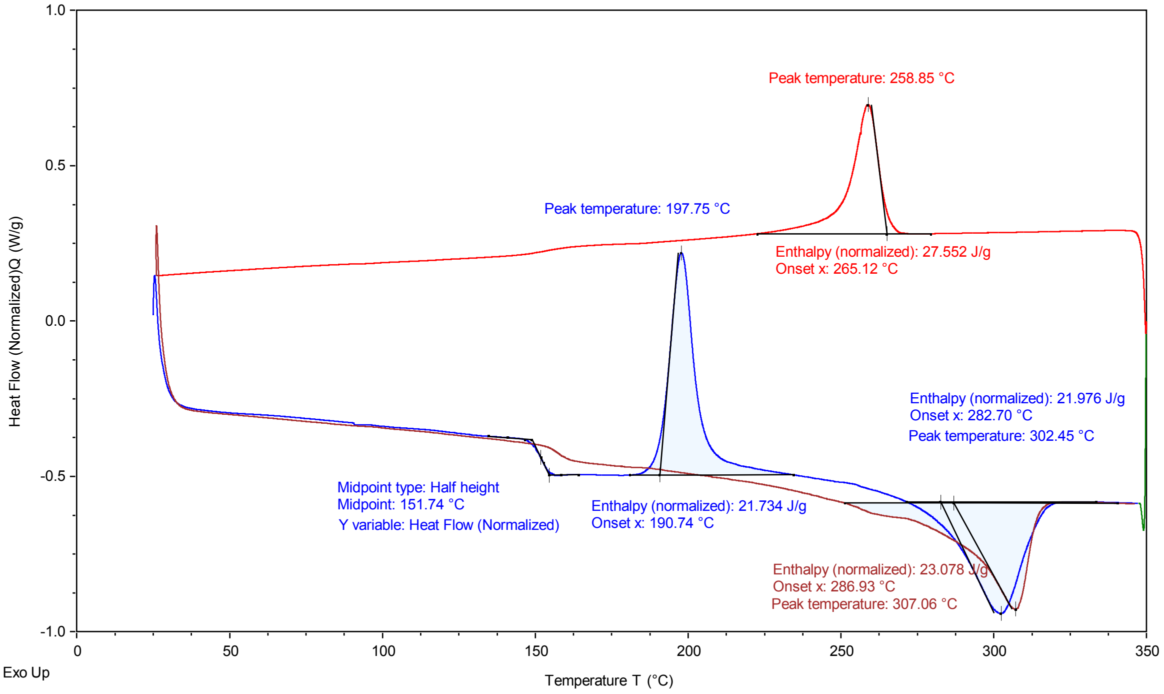
Set	Measurement point							
	1	2	3	4	5	6	7	8
1 (170 °C, 2 hrs)	0.034	0.027	0.05	0.039	0.039	0.0185	0.0405	0.038
2 (170 °C, 4 hrs)	0.0185	0.014	0.046	0.0175	0.0215	0.016	0.03	0.0115
3 (185 °C, 2 hrs)	0.01	0.0175	0.0445	0.0075	0.0095	0.0105	0.0165	0.018
4 (185 °C, 4 hrs)	0.0265	0.014	0.0175	0.018	0.022	0.01	0.047	0.024
5 (200 °C, 2 hrs)	0.0015	0.024	0.031	0.0085	0.014	0.01	0.0215	0.0165
6 (200 °C, 4 hrs)	0.0085	0.0035	0.021	0.0205	0.038	0.0365	0.059	0.026

Change in crystallinity table and graphs:

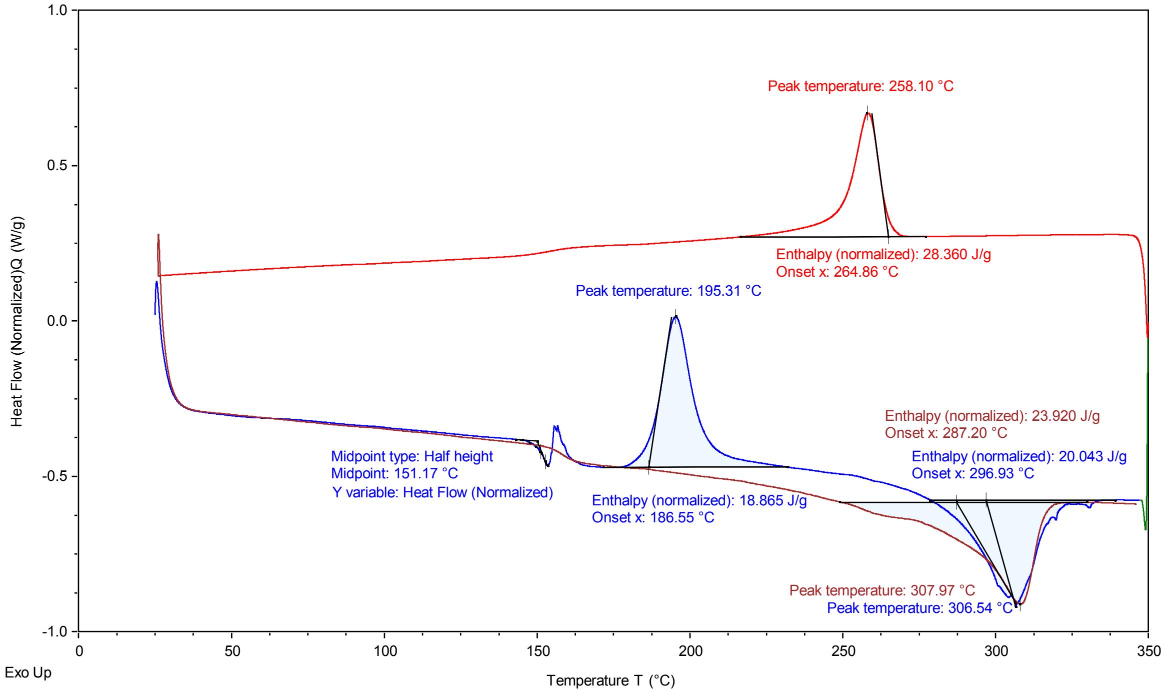
Change in Crystallinity

Sample	Cold Crystallization Enthalpy (J/g)	Melting Enthalpy (J/g)	Theoretical Melt Enthalpy at 100% crystallized (J/g)	Crystallization Percentage
No Annealing	21.734	21.976	130	0.186%
1 (170 °C, 2 hrs)	18.865	20.043	130	0.906%
2 (170 °C, 4 hrs)	2.7401	22.595	130	15.273%
3 (185 °C, 2 hrs)	0	23.171	130	17.824%
4 (185 °C, 4 hrs)	0	19.606	130	15.082%
5 (200 °C, 2 hrs)	0	22.59	130	17.377%
6 (200 °C, 4 hrs)	0	22.137	130	17.028%

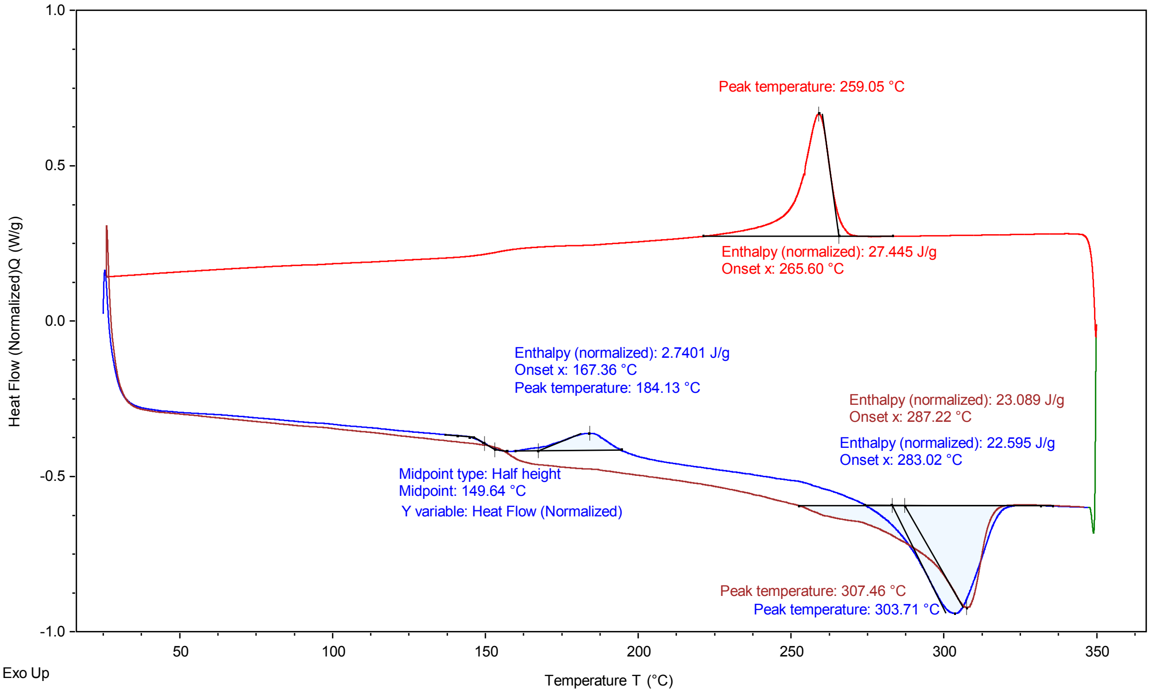
AM200 non annealed



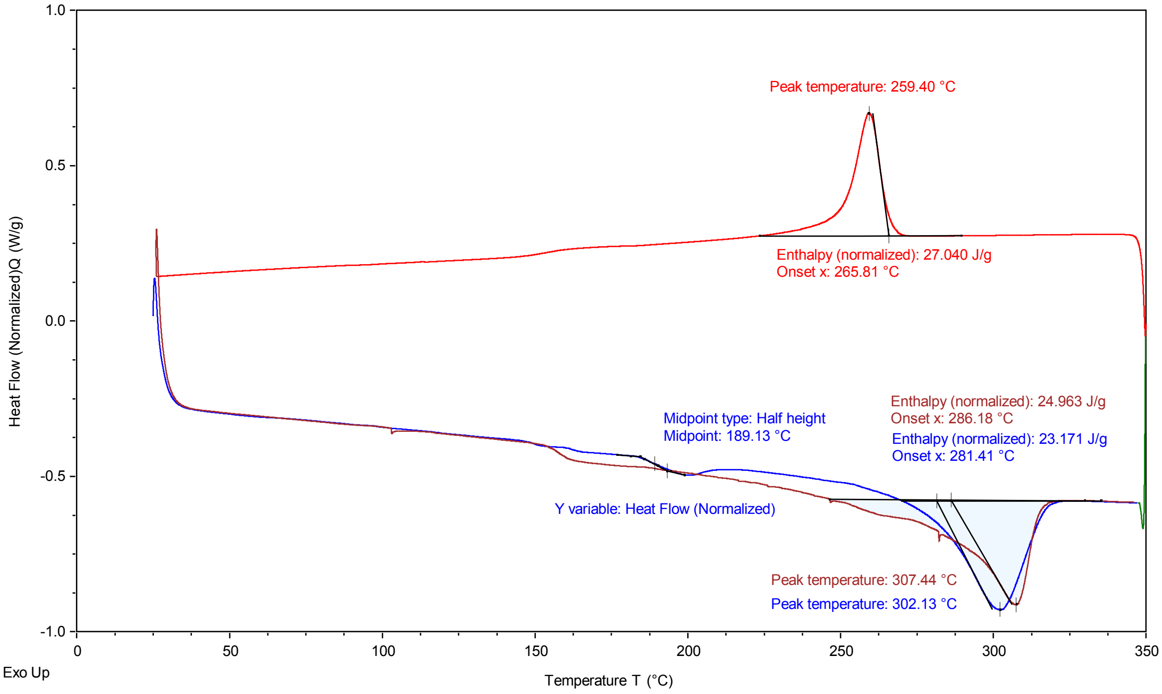
AM200 Set 1



AM200 Set 2



AM200 Set 3_2



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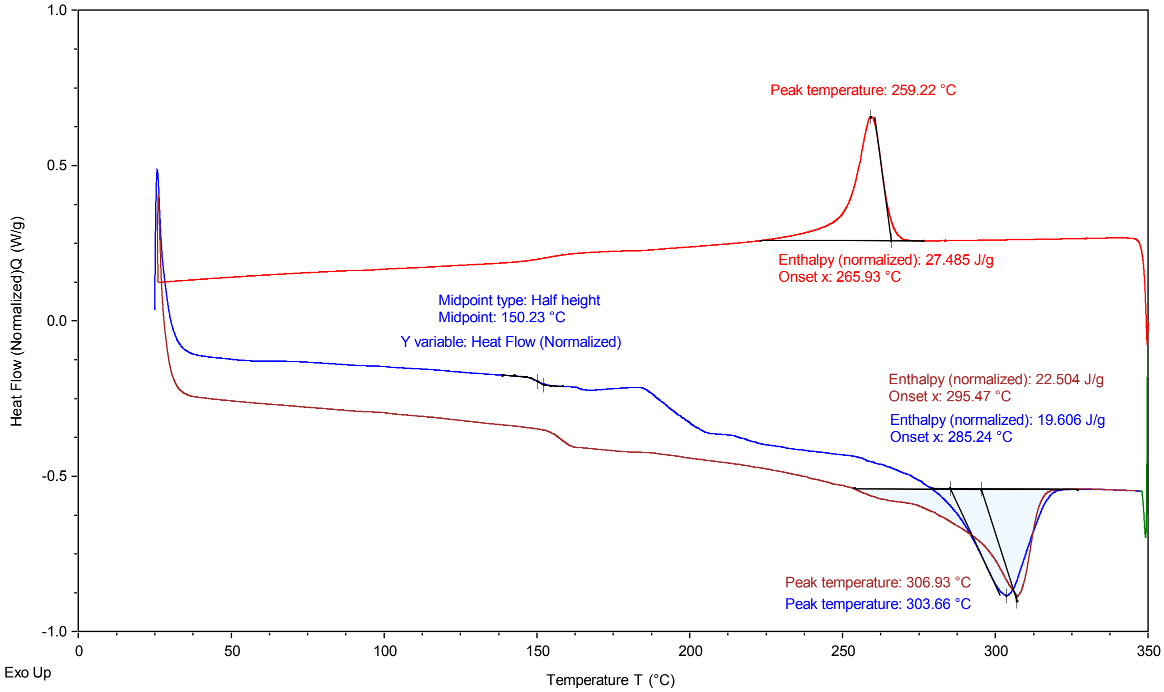
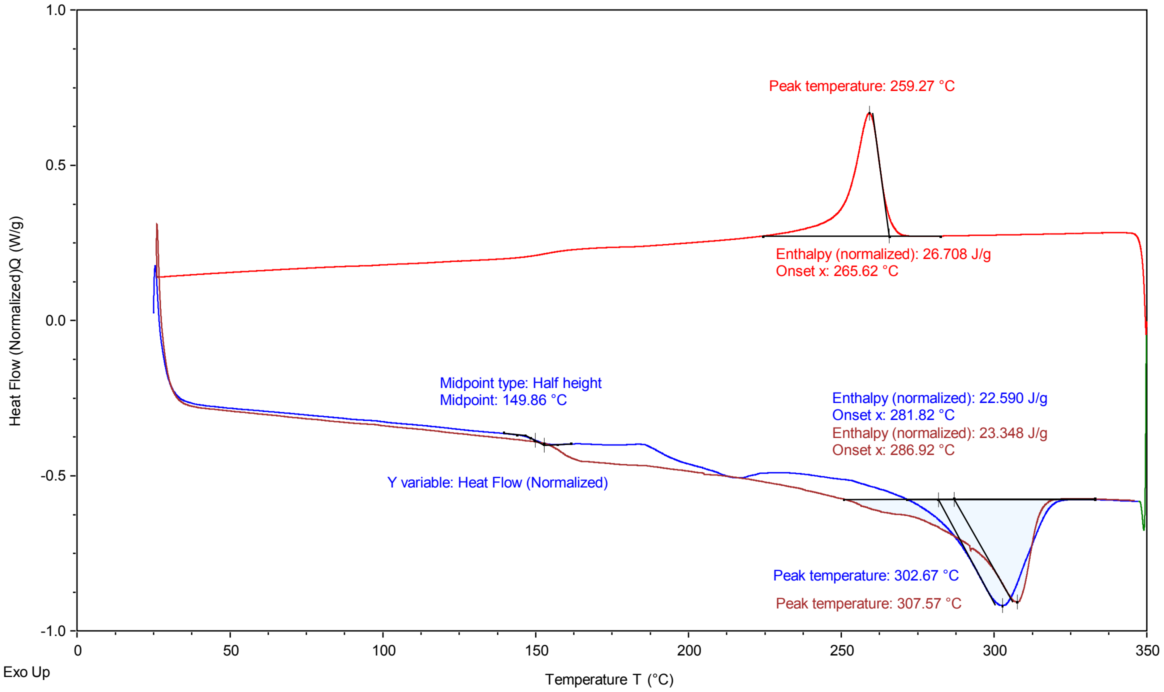
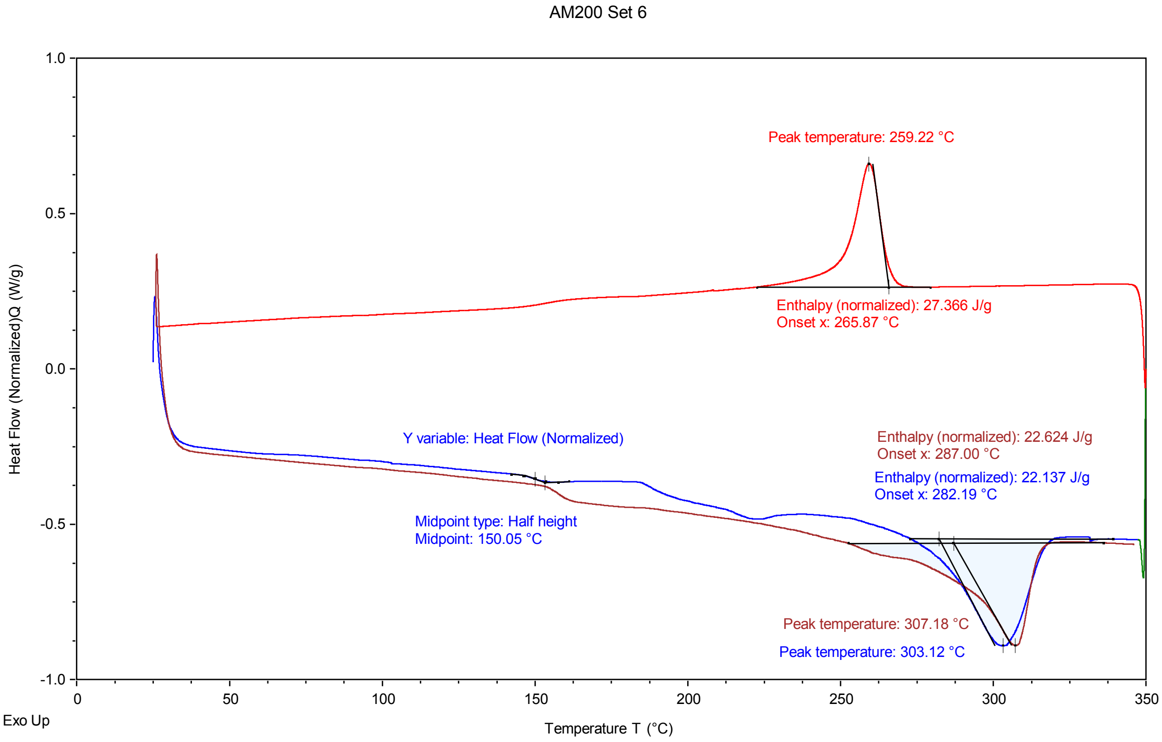


AM200 Set 5_2

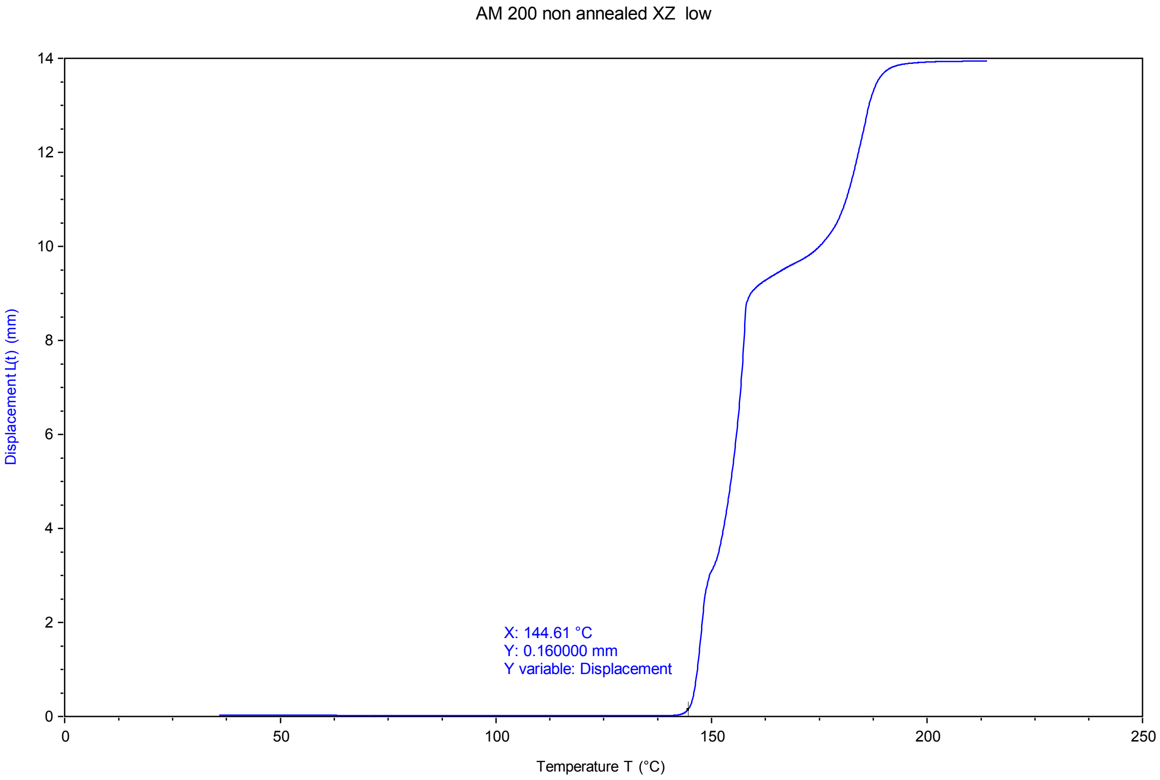


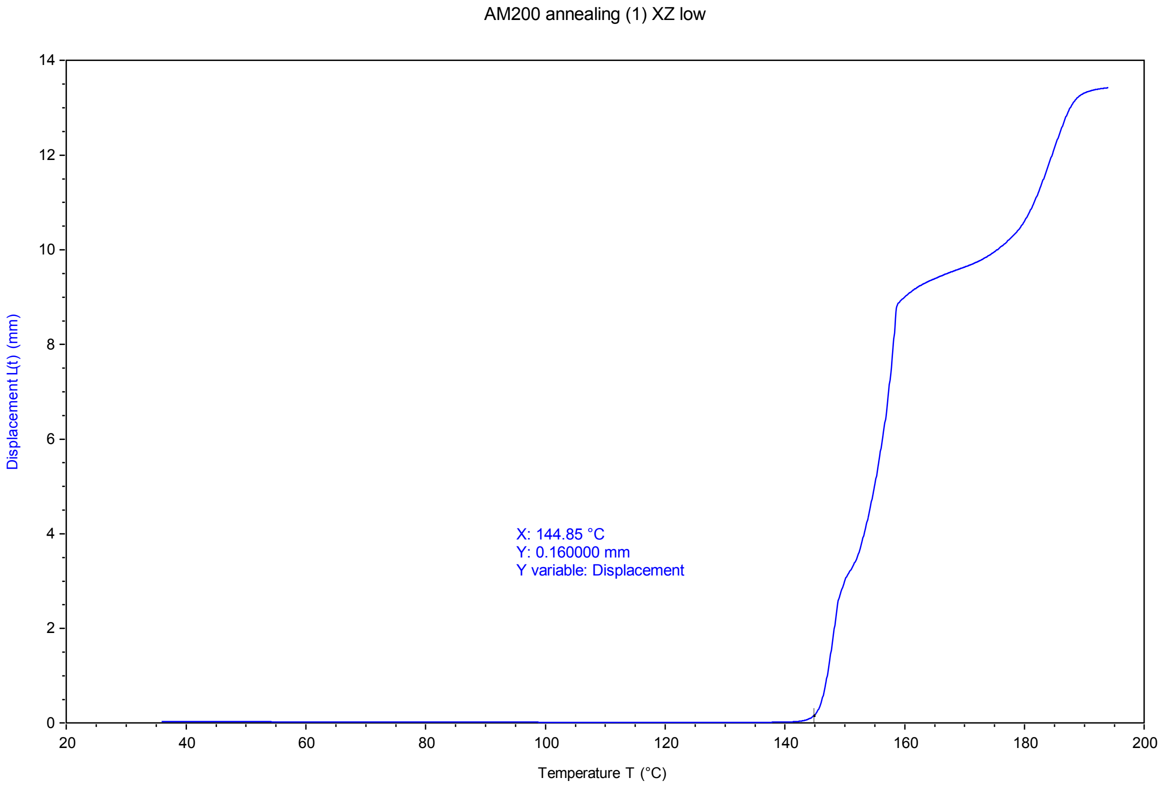


Heat Deflection Temperature Data:

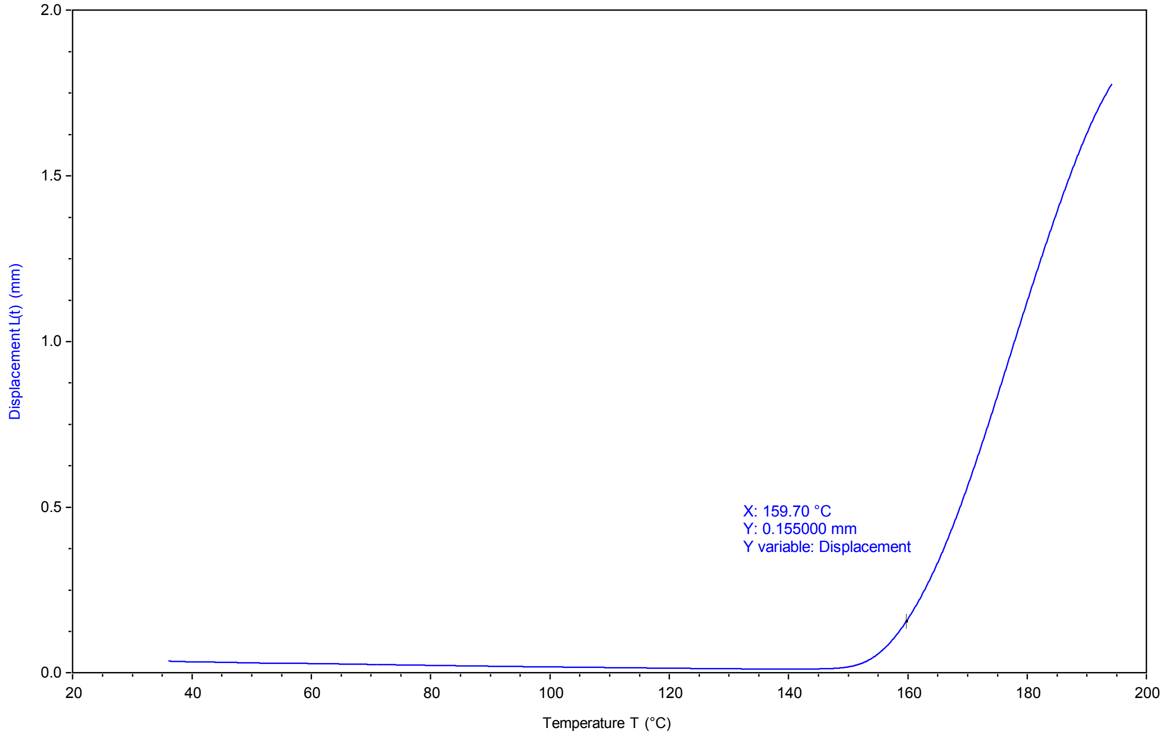
Change in HDT

Sample Set	HDT (°C)	Increase in HDT (°C)
Not Annealed	144.61	-
1	144.85	+ 0.24
2	159.7	+ 15.09
3	163.13	+ 18.52
4	165.96	+ 21.35
5	167.66	+ 23.05
6	169.79	+ 25.18

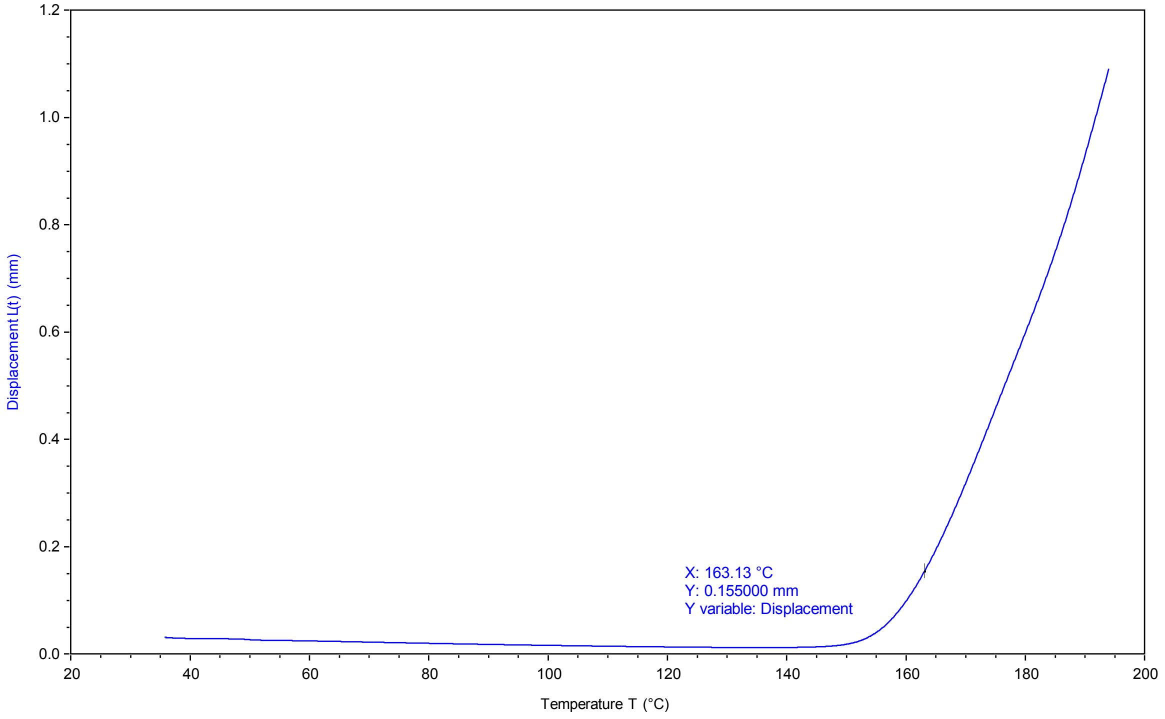

AM 200 non annealed XZ low

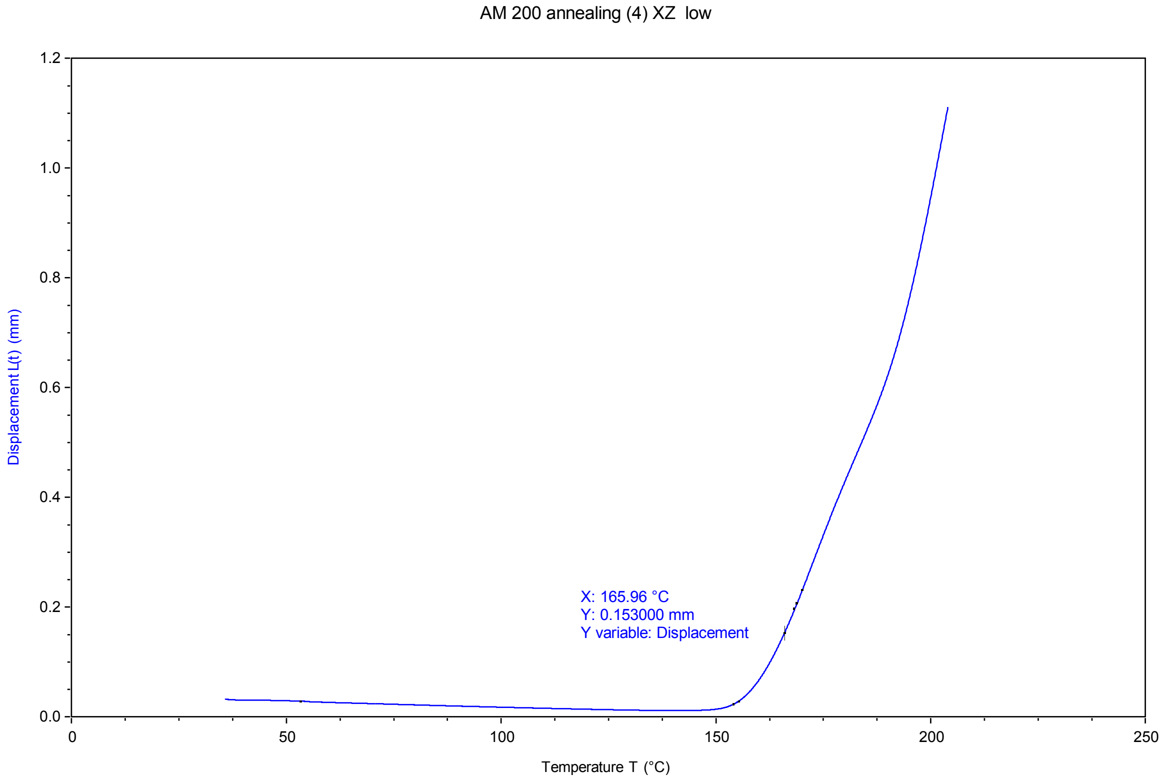

AM200 annealing (1) XZ low

AM200 annealing (2) XZ low

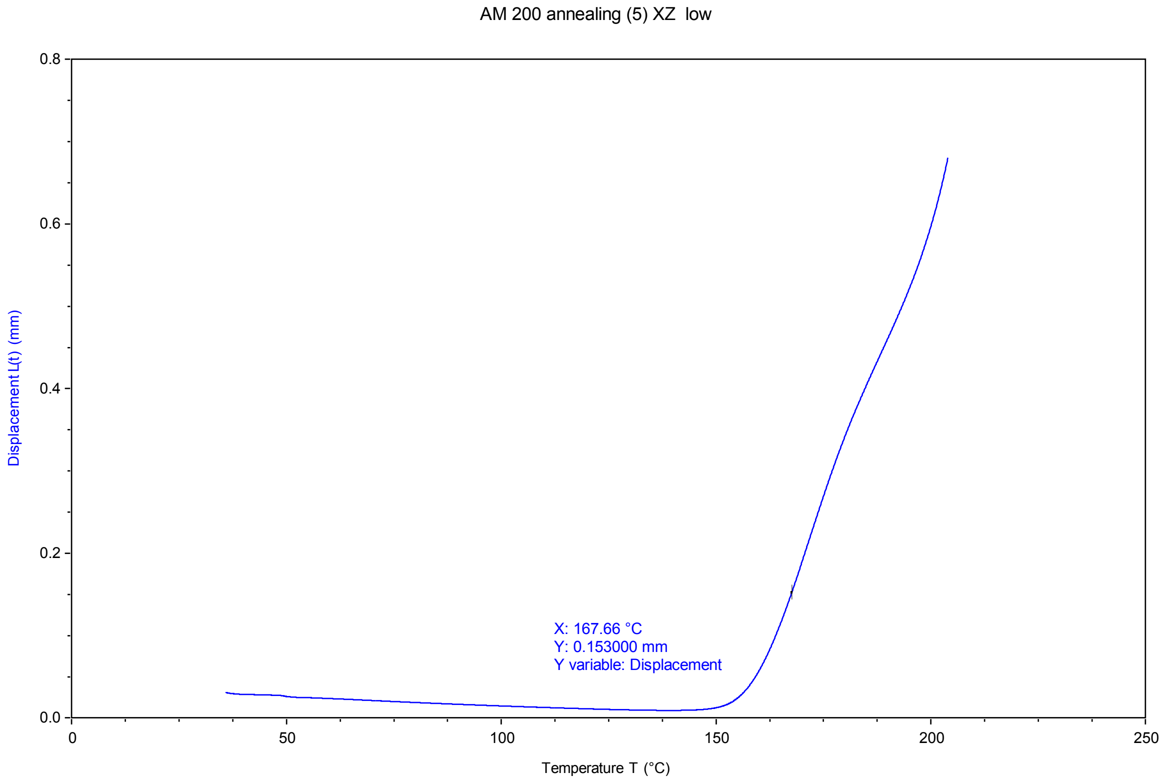


AM 200 annealing (3) XZ low

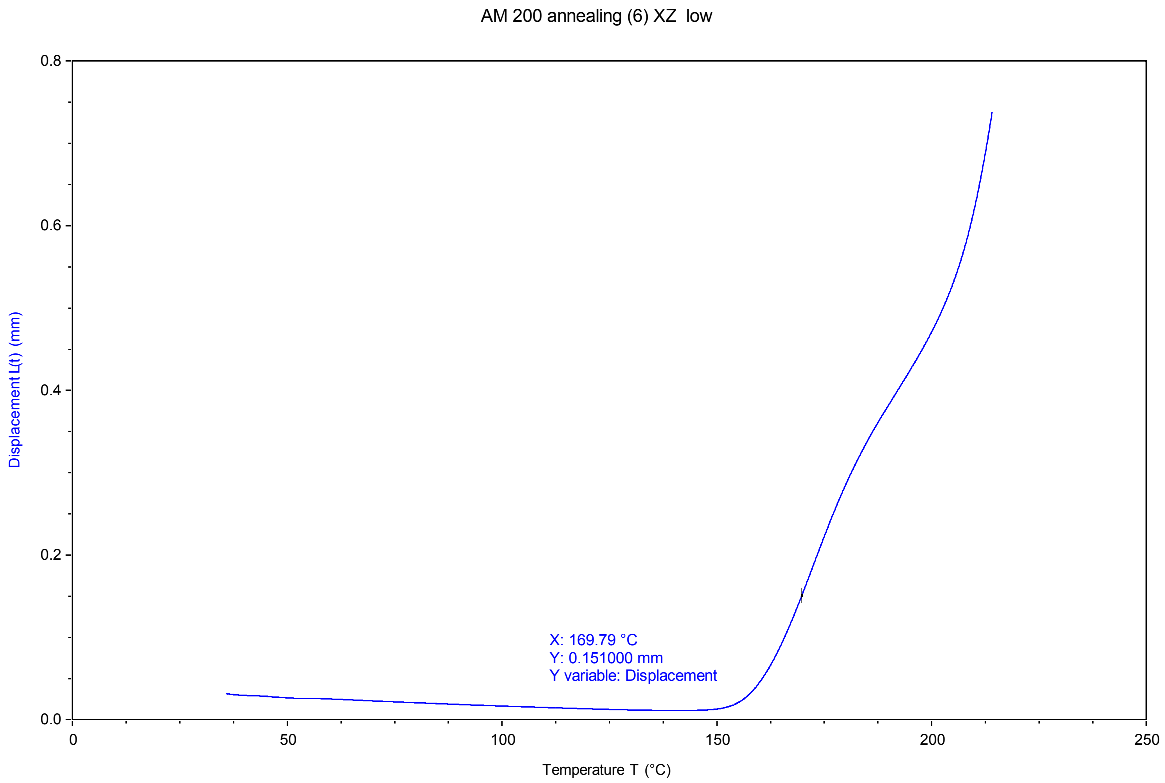




X: 165.96 °C
Y: 0.153000 mm
Y variable: Displacement



X: 167.66 °C
Y: 0.153000 mm
Y variable: Displacement



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