Thermal Conductivity of Nanocomposites in the FDM Printing Process

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Abstract

Fused deposition modeling (FDM) is a common method of 3D printing in which filament is melted and extruded onto a build platform. Currently, bond strength between deposited threads suffers as a result of the poor thermal properties of available printing materials, which also limit the usefulness of FDM printing in applications involving heat transfer. Polylactic acid (PLA) and polypropylene (PP) are two thermoplastic polymers used in FDM printing, but both exhibit low thermal conductivities. Graphene nanoplatelets (GNP) and boron nitride (BN) nanoparticles are thermally conductive particles that can be dispersed into polymer matrices to improve the material's thermal properties. In this study, we fabricated nanocomposites from the aforementioned thermoplastics and nanoparticle additives and extruded filaments for thermal conductivity testing. We also tested printed samples and found that the additives increased the thermal conductivities of both filaments and printed products. Additionally, scanning electron microscopy (SEM) revealed that the additives improved fusion between extruded threads in the printing process and that the nanoparticles in printed samples were oriented in the direction of heat flow, which may explain the observed improvements to the thermal properties of tested materials.

1 Introduction

1.1 3D printing and fused deposition modeling

3D printing is an emerging technology with applications for rapid prototyping, industrial manufacturing, and hobbyist use. A common and affordable method of 3D printing is fused deposition modeling (FDM), in which filament is melted and layered on a build platform via extrusion through a small diameter nozzle (Wendel et al., 2008). It is important for the materials used in FDM printing to have high thermal conductivities, so heat can be transferred from the heating block to the filament as quickly as possible. If the polymer being printed does not heat to a temperature above its glass transition while in contact with the heating block, it will be unable to flow correctly which will clog the system and severely harm both the product and the printer. Additionally, during the FDM process, the nozzle of the 3D printer lays down single threads of material at a time, and it is crucial that these threads fuse well enough for the final product to maintain structural integrity upon completion. For the necessary fusion to occur, the two individual threads need to be as hot as possible to ensure that strong connection is formed when the material cools down. Thus, materials with improved thermal properties would also exhibit improved fusion during the printing process, and as a consequence would result in better printed products with improved mechanical properties (Sun et al., 2008).

Not only is the thermal conductivity of printing materials important during the actual printing process, but it also affects the properties of the final products that can be manufactured with FDM printing. The poor thermal properties exhibited by current 3D printing materials limit the method's usefulness, as many engineered systems, such as heat exchangers and thermal management devices, require materials with high thermal conductivities to effectively transfer heat from one medium to another. If printing materials supported a greater rate of heat transfer, the range of applications for FDM printing would be greatly expanded and printed products would be much more useful.

1.2 Significance of polymer nanocomposites

Polymer nanocomposites, materials defined by the dispersion of nanoparticles in a polymer matrix, can exhibit different physical and chemical properties than their pure polymer forms, inheriting qualities of the nanoparticle additives. Therefore, the introduction of thermally conductive nanoparticles into a polymer matrix can improve the thermal properties of the polymer, which, in this case, would improve the quality and usefulness of polymer-based 3D printed products (Ebadi-Dehaghani et al., 2012).

1.2.1 Polylactic acid and polypropylene

In this study, we investigated two thermoplastic polymers: polylactic acid (PLA) and polypropylene (PP). PLA, a biodegradable polymer fabricated from renewable resources like corn, is one of the most commonly used 3D-printing materials because of its ability to become moldable when heated and rigid when cooled (Jamshidian et al., 2010). PP is a common polymer in a wide range of applications largely because of its physical and chemical ruggedness and its use in manufacturing living hinges. (Lanzonby 2010). It is also gaining popularity in FDM printing due to its thermoplasticity, although it is not as readily available in filament form as PLA. With each of these polymers, we blended two different nanoparticle additives, discussed below, at different concentrations to create nanocomposite materials.

1.2.2 Graphene and boron nitride nanoparticles

Graphene is a two-dimensional, hexagonal lattice of carbon atoms with many unique and desirable properties. It is one of the strongest materials ever observed, is nearly transparent due to its atomically monolayer nature, and exhibits high thermal and electrical conductivity (Papageorgiou et al., 2017). Graphene nanoplatelets (GNP) are nanoparticles made from several small layers of graphene held together with van der Waals forces that inherit many properties of monolayer graphene, including high thermal conductivity. Graphitic boron nitride (BN) is, on the molecular scale, a hexagonal lattice comprised of boron and nitrogen in a structure similar to that of graphene. Within each sheet, boron atoms are bonded to nitrogen atoms with strong covalent bonds, while each sheet is attracted to each other with weak van der Waals forces, much like graphene (Lee et al., 2012). Boron nitride is of interest in this experiment because it has a large thermal conductivity and demonstrates a high thermal stability at temperatures below 1000°C (Kostoglou et al., 2015).

1.3 Importance of thermal conductivity testing of nanocomposites

Thermal conductivity testing reveals important information about a material's thermal properties, and thus its usefulness in various applications. The rate at which a material is able to transfer heat is crucial in engineered systems like heat exchangers, housings for electronics, or other applications in which the material is designed to dissipate heat quickly or is subjected to changes in temperature often. Currently, manufacturers turn to metals like copper, aluminum, or gold when looking for materials with high thermal conductivities, but these metals are relatively heavy and expensive when compared to most thermoplastic polymers. Polymer nanocomposites could potentially replace metals in specialized applications requiring high thermal conductivities, which would provide cheap, lightweight, and often environmentally friendly alternatives for manufacturers in the aerospace, automotive, or electronics industries.

Furthermore, despite having high thermal conductivities, metals are not ideal for FDM printing on account of their extremely high melting points. Polymers are far better suited to the FDM printing process in this regard, since thermoplastics like PLA and PP are able to flow easily when heated to temperatures above their glass transition points and harden upon cooling. For PLA and PP, the glass transition temperatures are roughly 60°C and -10°C, respectively (Mark, 1999). The drawback to using polymers in 3D printing is the relatively low thermal conductivity of pure polymers, which is why polymer nanocomposites may offer a viable option for FDM printing.

1.4 Importance of polymer chain and nanoparticle orientation for thermal conductivity

Since polymers are comprised of long chains of many molecules, they often become entangled with each other, which impedes the flow of heat through the material. For this reason, molded polymer samples have very low thermal conductivities; extruded filaments, on the other hand, are oriented in the direction of extrusion, which is known to improve directional heat conduction in polymers (Yoshihara et al., 2014).

Similarly, the orientation of nanoparticles in polymer nanocomposites has a significant effect on the thermal conductivity of the material. Materials in which nanoparticles are aligned parallel to the direction of heat transfer exhibit far higher thermal conductivities than those in which nanoparticles are oriented perpendicular to the direction of heat flow (Shahadat et al., 2017). Thus, in this study, we concerned ourselves not only with the extrusion of oriented polymer chains but also with the directional alignment of nanoparticles in the polymer matrix.

1.5 Importance of SEM analysis of 3D printed samples

Scanning electron microscopy (SEM) uses a focused beam of electrons to probe the surface of a sample and can produce topographical images with resolutions on the nanometer scale. SEM was used in our experiments to study the fusion of printed filaments and to analyze the orientation of nanoparticles in these printed samples.

1.6 Experimental overview

The present experiment was focused on the thermal properties of nanocomposite materials formed from PLA and PP with GNP and BN nanoparticles and the effect of these additives on the behavior of polymers in the FDM printing process. We used an infrared imaging camera to analyze the rate of heat flow in different materials and to study the conditions of fusion between printed threads for pure and composite materials during the printing process. To visualize the effects of these conditions on the quality of the final printed product, we used SEM to take high resolution photos of the morphology of various printed samples. We also used SEM to analyze the orientation of nanoparticles in the composite materials. Finally, we tested the thermal conductivity of the various printed samples using a thermal conductivity meter and studied the relationship between additive concentration and the thermal conductivity of the final printed product.

2 Materials and Methods

2.1 Preparation of composite materials

Commercial PLA 4042D and PP of molecular weight 250,000g/mol were used in the production of the nanocomposite materials as well as graphene H-5 nanoplatelets and commercial boron nitride. Composite materials were prepared from pure polymer and additive at concentrations of 5, 10 and 20wt% for a total of 50g of each material. Both the pure polymer and the additive were mixed in a C.W. Brabender Mixer on an Intelli-Torque Plasti-Corder for 20 minutes and left to cool in the Brabender for 10 minutes to allow the polymer composite temperature to fall below the glass transition.

2.2 Preparation of composite filaments

The composite materials were then pelletized in a three phase C.W. Brabender Granu-Grinder Granulator. The pelletizer was cleaned after each use so as to ensure that no samples were contaminated with foreign particles. Commercial PLA and PP pellets as well as the pelletized composite materials were extruded through a single phase Filabot EX2 Extruder at 160°C for PLA and at 165°C for PP samples. The extruder was cleaned in between each session with pure PLA or PP depending on the material that we planned to extrude next; PLA was used for cleaning before PLA-based materials were extruded and PP was used if we planned to extrude a PP-based material next. The extruded filament was collected on spools to be cooled and stored for later use.

2.3 Thermal conductivity testing of filaments

We performed initial thermal analysis on filaments by heating them from one end and recording this process via infrared camera. To attach each pure and composite filament to the 40W aluminum heating block used, we extruded a small amount of material through a nozzle in order to secure the sample in place. Since the nozzle diameter was far too narrow to allow for sufficient data collection, we oriented the nozzle upside-down so that the wider portion faced downwards and the diameter of the opening was roughly 3mm. Once the filaments were safely in full contact with the heating block, we began to introduce heat into the system by setting the heating block to temperatures of 80°C, 100°C, and 120°C. A FLIR A300C thermal camera, with 4X lens magnification and a working distance of 7.9cm, was used to image the changing thermal profiles of filaments at each temperature (Figure 1). For each temperature, the samples were filmed while the heating block was increasing in temperature and then again once the heating block was at a steady temperature. In between each set of tests, we allowed both the heating block and the filament to cool to room temperature before setting the heating block to the next temperature.

2.4 Preparation of 3D printed samples

The G-code files for the 3D printed samples were made for an Ultimaker 2 Extended+ using a semi-automated Java program, written in Eclipse Neon. This code was written to ensure that the printed filaments were oriented in the desired directions for thermal imaging, since traditional methods for generating G-code files were determined to be too imprecise and were observed to code for seemingly random filament placement, which is impractical for this application. Custom G-code files were written for two single-layer prints, one 10mm by 40mm and the other 150mm by 40mm (Competition Entrant, 2017). This difference in sample dimensions is directly related to the temperature at which fusion between adjacent filaments begins: in the larger sample, each filament has more time to cool down before the adjacent filament is laid down while the opposite is true in the smaller sample. By examining the fusion between filaments in the two different types

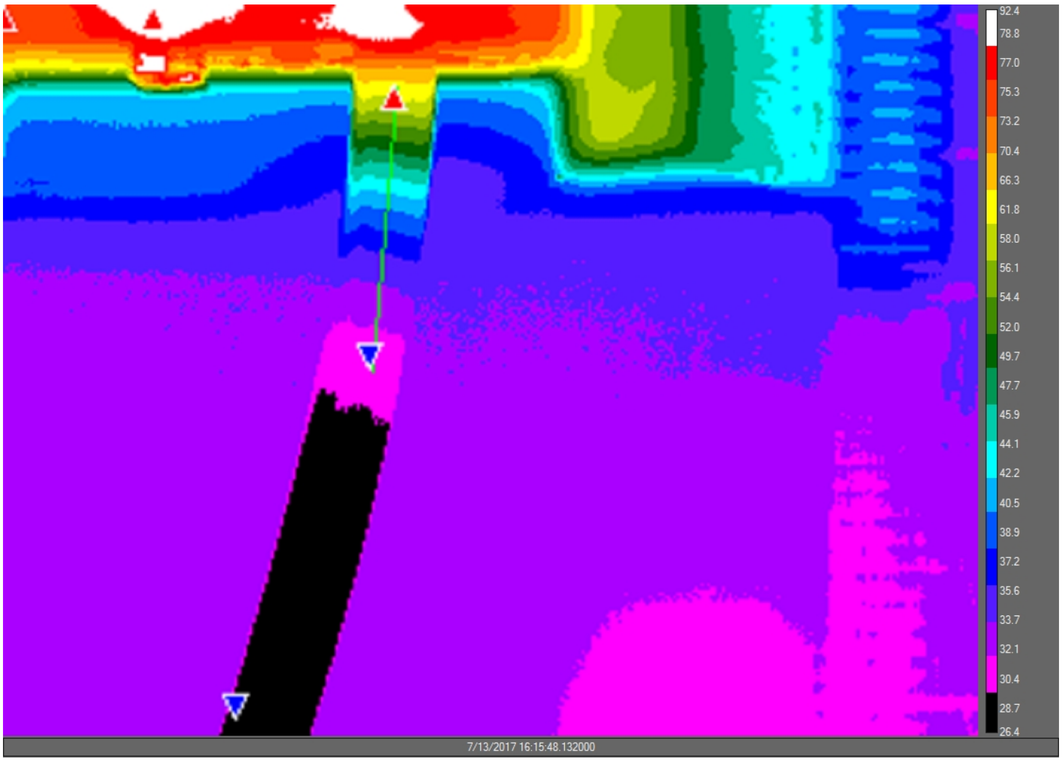

Figure 1: IR imaging of pure PP filament with heating block at 80°C, using ResearchIR software. The heating block is visible toward the top of the image, the filament in the middle (where the arrowed lines span), and a metal centimeter ruler for reference on the right. The temperature scale to the right of the image is in degrees Celsius.

of samples, we were able to study the effect of this temperature difference on the fusion within each layer of the prints.

2.4.1 Thermal analysis of the FDM printing process

Thermal analysis of the FDM printing process was performed with the same IR camera that was used to image the transfer of heat through pure and composite filaments in Section 2.3. These measurements were taken directly during the FDM printing process in order to provide data regarding the temperature of each filament during initial deposition and during fusion with the adjacent material. Thermal movies of the printing process were recorded both for the 10mm by 40mm samples and for the 150mm by 40mm samples, so the effect of filament temperature on fusion could be studied. Video footage from the thermal camera was later analyzed using FLIR ResearchIR software (Figure 1).

2.5 Preparation of printed samples for SEM

The printed samples were freeze-fractured in preparation for SEM in order to reduce the size of each sample without significantly altering the polymer matrices or surface morphologies. The samples were first submerged in liquid nitrogen for about 10 minutes to cool them down. The frozen samples were then fractured at the desired locations though blunt force from a straight edge perpendicular to the printed threads that make up the print. The desired size for each sample was roughly 1cm by 1cm. After the samples were cut to the correct size, their surfaces were sputter coated in gold to improve electrical conductivity for SEM.

2.5.1 SEM analysis of printed samples

Once the samples were prepared for SEM, they were organized and labeled on a glass plate. Using the SEM microscope, we took high resolution pictures of the surface morphology and cross-sections of each sample. The magnifications of the images ranged from 150X to 50kX and revealed topographical features of the printed samples on the micro- and nanometer scales.

2.6 Thermal conductivity testing of oriented samples

Using the process outlined in 2.4, we wrote custom G-code for oriented 3D printed samples for thermal conductivity testing (Competition Entrant, 2017) with a TA Instruments thermal conductivity meter (DTC-300). As the thermal conductivity meter uses two circular plates on either side of the sample to measure the rate of heat transfer through the sample, the print needed to be circular in shape. During this test, heat moves from one plate through the sample to the other plate, the extruded threads were to be oriented perpendicular to the face of the circle. We found that when the circle was printed in one piece, the force of gravity deformed the sample while it was still hot and moldable; this problem was averted by a new set of G-code that instructed the printer to lay down two upside-down semicircles on the build platform. These two halves were then joined together using the ink from a Circuitworks conductive circuit pen as a thermally conductive adhesive. The circular samples were sent to an affiliated research facility to be tested in the thermal conductivity meter.

3 Results and Discussion

3.1 Thermal analysis of extruded composite materials

FLIR ResearchIR software was used to extract temperature values from the thermal movies recorded of filament heat transfer and from the thermal imaging of the FDM printing process. In our analysis of the thermal conductivities of pure and composite filament, we used the aforementioned software to quantify the changing temperature of the material at different points along the filament. For the thermal movies of the printing process, we simply used the software to observe the temperatures of different locations on the printed sample at different times in order to aid in our study of fusion in FDM printing. This information was taken into account when performing SEM analysis on the printed samples, as described in Section 3.2.

3.1.1 Relative thermal conductivities at steady state

One-dimensional thermal conductivity along the length of a filament can be calculated by the following relationship derived from Fourier's expression for heat flux:

$$k = \frac{QL}{A\Delta T} \tag{1}$$

where k is thermal conductivity in W/m K, Q is the rate of heat transfer in W, L is the distance from the top of the filament in m, A is the cross-sectional area of the filament in m^2, and Δ T is temperature difference between a given point and the top of the filament in K or °C.

While the heating block has a known power output of 40W, we determined that this value was not accurate enough for our purposes, as the thermal conductivities calculated using this power output were improbable and unrealistic. Hence, without the actual value for

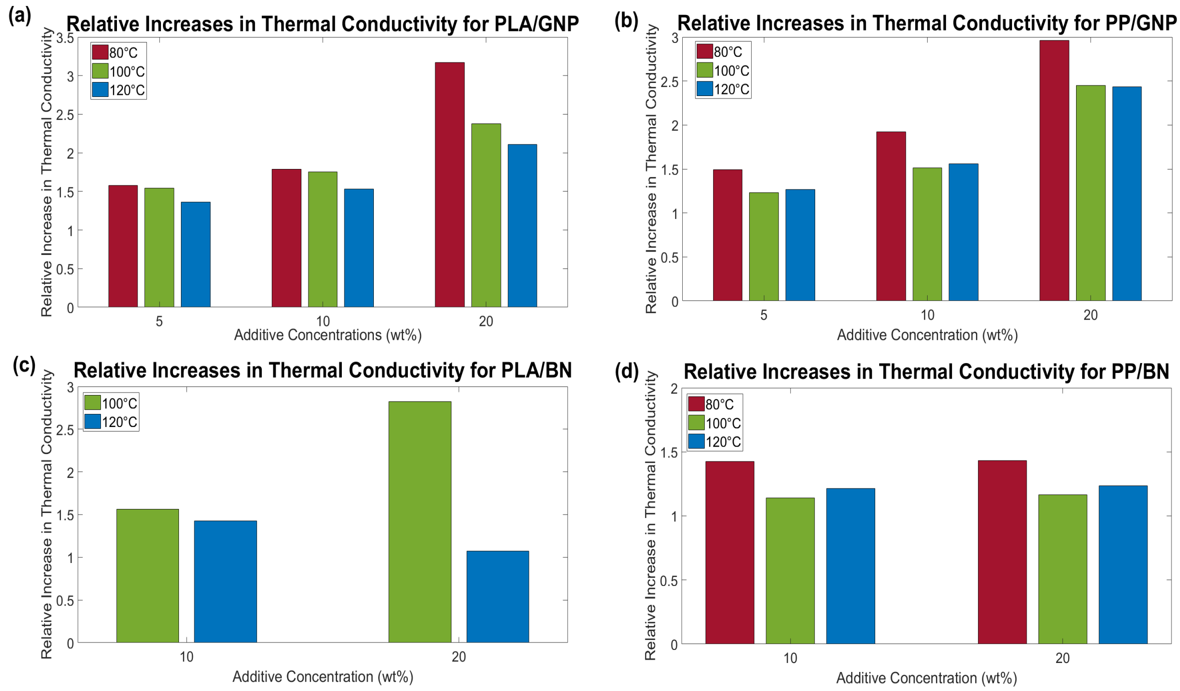

Figure 2: Relative increases in thermal conductivity of nanocomposite filaments from pure polymer filaments, at concentrations of 5, 10, and 20wt% and with the heating block set to 80°C, 100°C, and 120°C, for (a) PLA/GNP composite, (b) PP/GNP composite, (c) PLA/BN composite, and (d) PP/BN composite. Due to experimental error, the values at 5wt% BN concentration and 80°C heating block for PLA/BN composite could not be shown. The changes in thermal conductivity at different temperatures are to be expected; polymers are known to have changing thermal properties at different temperatures, particularly around their glass transition temperatures (dos Santos et al., 2013).

the rate of heat transfer, we could only calculate the relative thermal conductivities of each material.

Overall, the values (Figure 2) showed an increase in thermal conductivity with the addition of both GNP and BN. For instance, at 80°C, adding 10wt% GNP to PLA increased thermal conductivity by 79%, while adding 10wt% BN to PLA led to a 64% increase in thermal conductivity. Higher loadings of additive led to even higher thermal conductivities, with some composite materials reaching conductivities around three times that of the pure polymer.

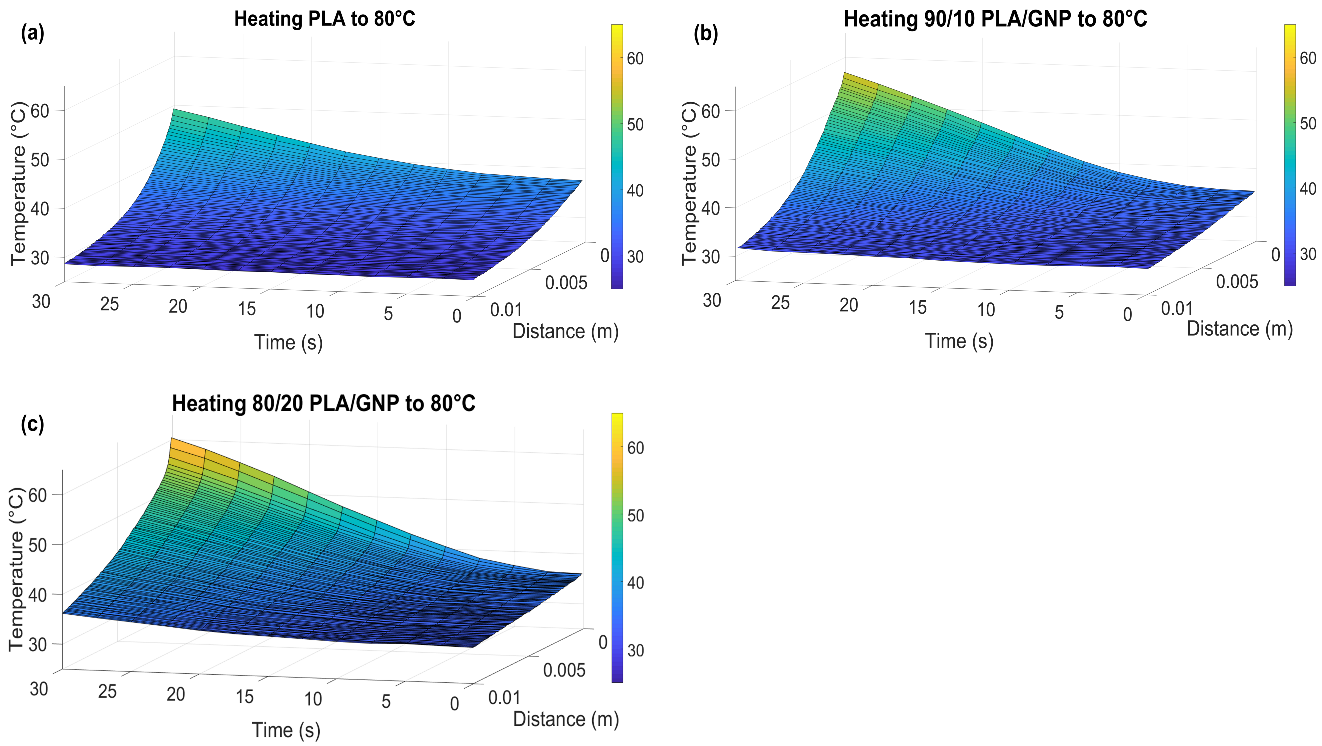

Figure 3: Surface plots depicting the relationship between a point's distance in meters from the heating block, the amount of time in seconds elapsed since the heating block reached 80°C, and the temperature of that point on the filament in °C. This relationship is visualized for (a) pure PLA, (b) PLA with 10wt% GNP, and for (c) PLA with 20wt% GNP.

3.1.2 Temperature change during heating

The thermal movies of pure and composite filaments that were recorded while the heating block was increasing in temperature visually demonstrate the far superior thermal conductivity of composite materials compared to pure polymers. By analyzing the thermal profile of each filament at regular time intervals of about 5s, we observed a positive correlation between the percent of additive and the rate at which each point on the filament increased in temperature. This relationship is also depicted in Figure 3 where the temperature of the material is represented as a function of time and distance from the heating block. The temperature of the composite materials increased far quicker than that of the pure polymers and heat travelled much further down the filament in the same amount of time.

A more comprehensive, 2-dimensional representation of the same data is shown in Figure 4, where the temperatures of comparable points on each filament are represented as func-

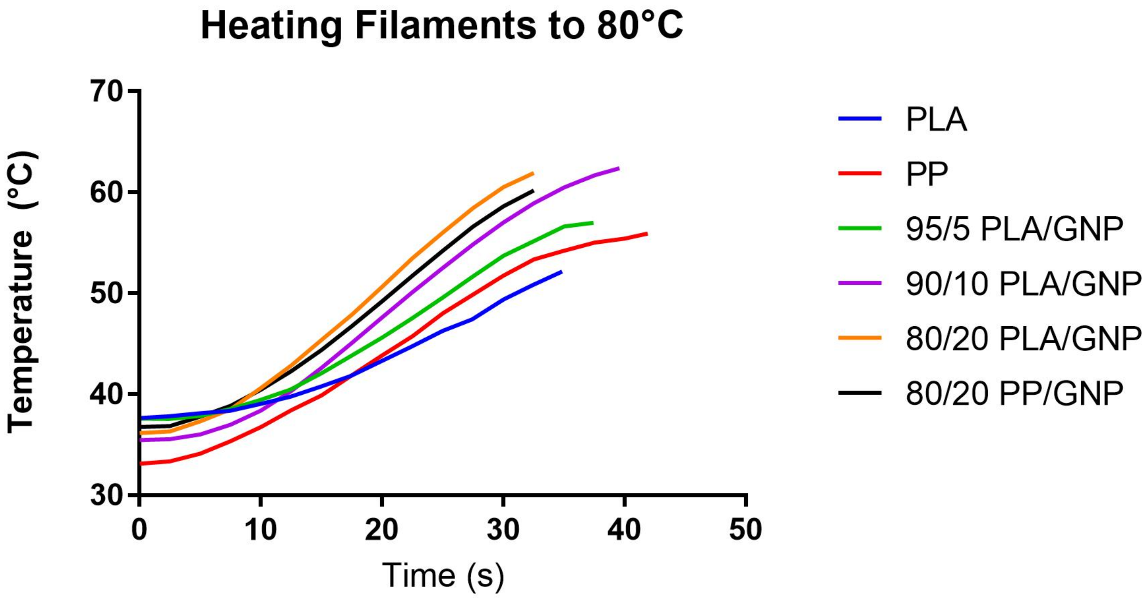

Figure 4: A two-dimensional representation of temperatures of comparable points on each filament as functions of time, showing the heating of each filament as the heating block increases temperature to 80°C.

tions of time. These graphs further elucidate the trend depicted by the surface plots: the rate at which composite materials heat up is much greater than that for pure polymers.

3.2 Analysis of morphology of 3D printed samples

The SEM pictures taken of the freeze fractured samples show that the samples that contained graphene and boron nitride had less surface defects and better fusion between threads. As seen in Figure 5a, the surface of the pure PLA has ridges between each pass of the nozzle, and overall the surface is very rough. Our analysis of the thermal movie of the printing process for a pure PLA sample using ResearchIR software revealed that the fusion temperature was around 45°C. In PLA with 10wt% graphene, as shown in Figure 5b, there are no major ridges present between each pass of the nozzle, and the surface is noticeably smoother and free from defects. This is likely because the composite material conducted heat quicker, so the fusion occurred at a higher temperature. For the sample depicted in the figure, the fusion temperature was recorded as 75°C. The same can be said about the boron nitride samples, where the ridge between filaments is not present, and the surface is very smooth (Figure 5c). Fusion in this sample occurred at approximately 60°C, so the fusion is not as strong as it is in the graphene samples, but is still stronger than that in the pure PLA samples. The trend observed in these images is clear; nanoparticle additives to pure polymers contribute to stronger adhesion between layers and superior surface quality of FDM printed samples.

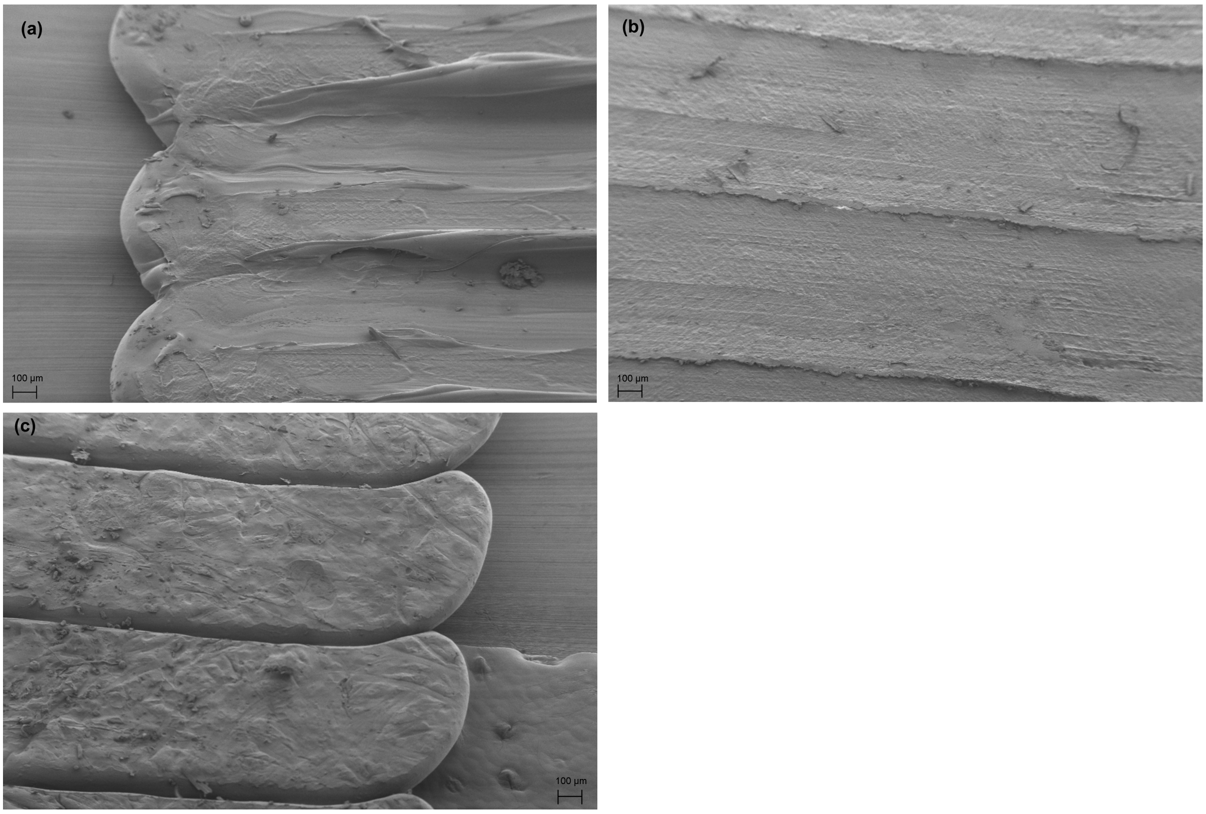

Figure 5: SEM images at 150X magnification of (a) printed pure PLA, (b) printed PLA with 10wt% GNP loading, and (c) printed PLA with 10wt% BN.

Furthermore, SEM images taken of the cross-sections of printed composite filaments reveal that both the graphene and boron nitride nanoparticles are aligned parallel to the direction of the filaments, which is also the direction of heat transfer in this study (Figure 6). This may explain the high increases in relative thermal conductivities that correlate to the introduction of nanoparticles into the polymer matrix, since oriented nanoparticles are known to drastically improve the rate of heat flow in solid materials.

Additionally, we found that the fusion between threads was much better in the smaller printed samples than in their larger counterparts (Figure 7). This difference in bond strength can be attributed to the temperature of the material at the time of fusion; in the smaller samples, the material had less time to cool down before adjacent threads

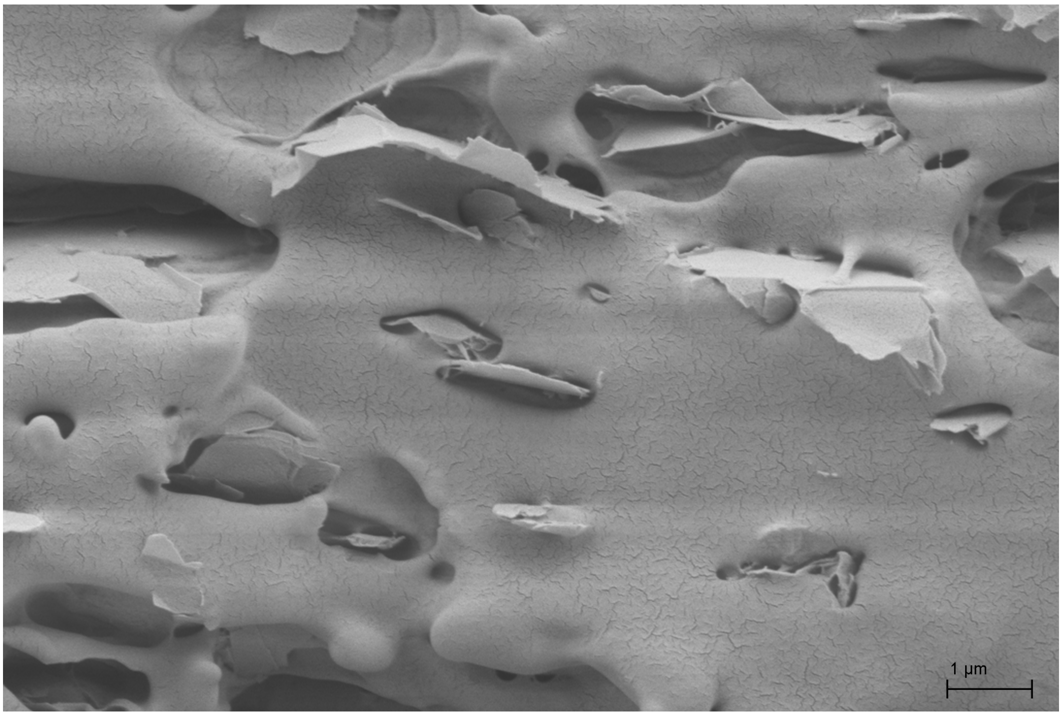

Figure 6: SEM cross-sectional image of printed PP with 10wt% GNP loading, at 30.00kX magnification, with the nanoparticles visibly embedded in and parallel to the filaments.

came into contact with each other. This finding illustrates the importance of heating during the printing process, and thus highlights the necessity of using materials with good thermal properties in FDM printing.

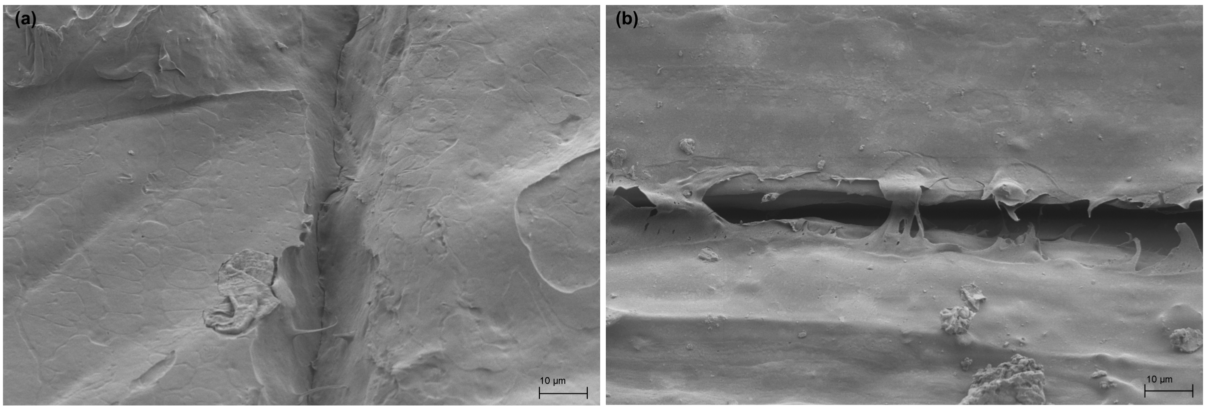

Figure 7: SEM images of the fusion between filaments in printed PP with 5% GNP loading of dimensions (a) 10x40mm and (b) 150x40mm, both at 3.00kX magnification, showing weaker fusion in the latter sample.

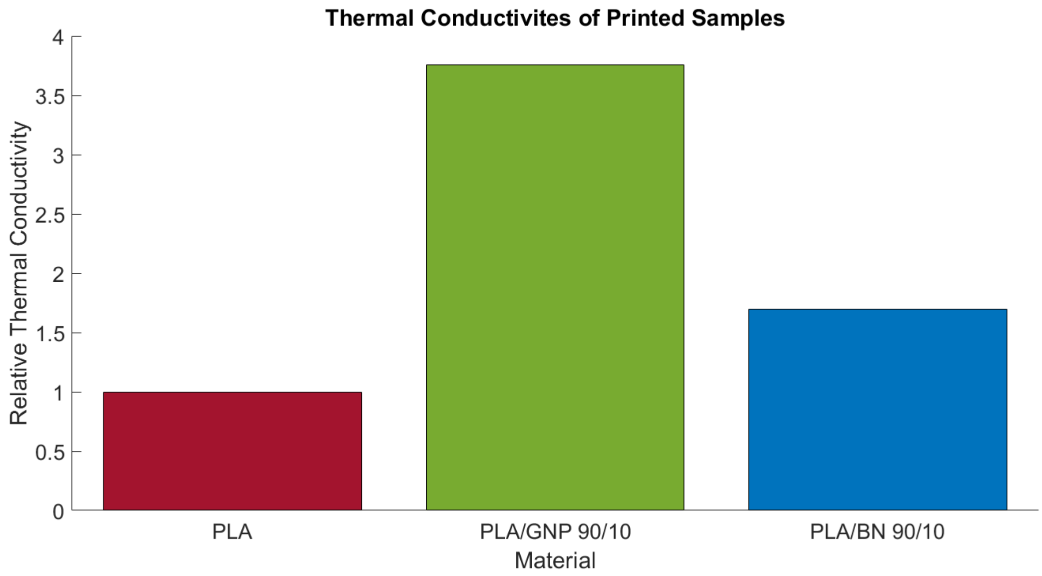

Figure 8: Thermal conductivities of select printed samples, as measured by the TA Instruments DTC-300.

3.3 Thermal conductivities of printed samples

The increases in relative thermal conductivities with the addition of GNP and BN nanoparticles observed in the printed samples were comparable to those observed with the filaments. As depicted in Figure 7, the thermal conductivity of PLA increased by 276% with the addition of 10wt% GNP and by 70% with the addition of 10wt% BN nanoparticles. These findings bolster the reliability of our results concerning the benefits of nanoparticle additives to the thermal properties of thermoplastics and demonstrate that nanocomposite printing materials allow for printed products with improved thermal properties.

4 Conclusion and Future Work

4.1 Conclusion

In this study, polymer nanocomposites were prepared from GNP and BN nanoparticles dispersed in the polymer matrices of PLA and PP at concentrations of 5, 10, and 20wt% and the thermal conductivity of each composite material was compared to that of the pure polymer from which it was made. Our findings reflect that the introduction of nanoparticles is very beneficial to the thermal properties of the polymer, improving thermal conductivity by as much as 176%. The importance of thermal conductivity to the fusion of threads extruded in the FDM printing process was demonstrated through SEM analysis of printed samples of different dimensions; we found that the quality of the final printed product is very much dependent on the temperature of the material while it is being printed. Furthermore, our results suggest that materials with greater thermal conductivities maintain higher temperatures during fusion, which, in our experiments, translated to stronger fusion and a better printed product. Not only did the improved thermal conductivity of the composite materials benefit the printability of the polymers used, but it also increased the thermal conductivity of the printed samples. Thus, the nanoparticle additives expanded the range of applications for these polymers in FDM printing, since printed products with high thermal conductivities can be used in far more engineered systems than those with poor thermal properties. In conclusion, the introduction of GNP and BN nanoparticles to the polymer matrices of PLA and PP improved thermal conductivity, heightened bond strength between extruded threads in printed samples, and enhanced the thermal properties of the final printed products.

4.2 Future Work

While the SEM images of 3D printed samples made clear that the quality of fusion was significantly improved by the nanoparticle additives, further research could be devoted to studying the resulting changes in the mechanical properties of printed samples. Although stronger bonds certainly contribute to improved mechanical strength, the dispersed nanoparticles may interfere with the integrity of the polymer matrices they inhabit, which could counteract some of the benefits of the improved fusion (Yang et al., 2015). More experiments need to be conducted to determine exactly this balance between the positive effects of stronger adhesion and the negative effects of nanoparticles on the structural integrity of the printed products.

Furthermore, the oriented FDM-printed samples should be more closely compared with those constructed by other means, such as by compression molding. While we conjecture, based on prior research, that the orientation of the filaments and alignment of the nanoparticles should help improve thermal conductivity, we have not yet verified those results experimentally. As such, the properties of both printed and molded samples should be compared in order to observe the effect of the orientation of the filaments on the material.

Additional research should also be conducted with different thermoplastics and thermally conductive nanoparticle additives. More thermal conductivity tests should be done at more extreme temperatures to establish the boundaries of effectiveness of different nanocomposites and to evaluate the thermal stability of various composite materials. Other thermal characteristics should also be studied to provide a more complete set of data with which to analyze nanocomposite materials in the FDM printing process. For example, material properties like thermal diffusivity, heat capacity, and thermal expansion in polymers are likely influenced by the addition of nanoparticles and can be very important in applications discussed earlier, such as heat exchangers and electronics housing.

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