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Zayden Chambers

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The Effect of Glass Fiber Reinforcement on the Thermal Properties of PETG

Zayden Chambers

Abstract: In the additive manufacturing industry, many polymer composites have properties - thermal and mechanical - that are unknown. The purpose of this study was to determine the key thermal properties of Polyethylene Terephthalate Glycol (PETG) with and without 15% glass fiber to understand the extent to which the glass fiber (GF) reinforcement affected the thermal properties of the thermoplastic. The PETG and PETG GF samples were produced via Fused Deposition Modeling (FDM). The samples were then weighed and tested using Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), and a Dynamic Mechanical Analysis (DMA). The change in thermal properties given by these instruments suggests that the glass fiber has a minimal effect on the key thermal properties when reinforcing PETG. These findings imply that using a material like PETG GF is not recommended for applications that require high temperatures.

Keywords: Additive Manufacturing, Fused Deposition Modeling, Polymer Composites, Glass Fiber Reinforcement, Poly Ethylene Terephthalate Glycol, Thermal Properties.

1.1 Introduction

Over the years, many industries have utilized additive manufacturing (AM) to effectively produce desired products. Additive manufacturing is used in many ways to create these products, each having its own advantages and disadvantages. Common additive manufacturing processes include material jetting, where the machine produces droplets of a material layer by layer. Selecting laser sintering (SLS) is a process in which a laser combines power-based layers into three dimensional objects. Fused deposition modeling (FDM) uses a nozzle - fed with thermoplastics - to eject the plastic in a controlled manner with increasing layers. Among the plethora of AM methods, FDM is the most widely used due to its accessibility.

Numerous thermoplastics can be used as filament in FDM, all of which have their own properties. For instance, the most popular thermoplastic for FDM is Polylactic Acid (PLA), which is a completely biodegradable plastic made from plant starch. Acrylonitrile Butadiene Styrene (ABS), however, is a thermoplastic known for its stiffness and its production of an unpleasant odor when extruded. Polyethylene Terephthalate Glycol (PETG) is another popular thermoplastic that is known for its chemical resistance and mechanical properties. Hence, PETG is widely used in food packaging and medical applications. It is evident that each thermoplastic has a weakness, so manufactures use different types of reinforcements - an additive that is combined with a thermoplastic in hopes of strengthening performance of the plastic - to assist the material with performance.

Reinforcements are commonly used to create numerous types of polymer composites (a thermoplastic and a reinforcement). There are many different types of reinforcements; the most common ones include carbon fiber (CF), glass fiber (GF), and Kevlar fiber (KF). Similar to thermoplastics, each reinforcing fiber has its different benefits and characteristics. For instance, carbon fiber is known for its excellent thermal resistance, whereas glass fiber is popular for increasing strength in the composite. There are numerous thermoplastics, with numerous reinforcements; the combinations of composites are nearly endless. Researchers have been trying to optimize different properties by combining a thermoplastic and reinforcement, but many combinations have yet to be studied. For example, the use of PETG combined with GF has not been assessed thermally. In turn, the properties of this composite have not been tested, making it difficult to evaluate the composite's full potential.

Literature Review

2.1 Overview of FDM and Polymer Composites

As time progresses, the use of additive manufacturing - the act of creating an object one layer at a time - has become prominent in the production process [1]. There are numerous types of additive manufacturing, with fused deposition modeling (FDM) becoming one of the most utilized [2]. FDM works by extruding thermoplastic filament through a heated nozzle one layer at a time until the object is fully created. FDM is most commonly used because of its ease of use compared to other techniques of additive manufacturing, and material variability [2].

There are plenty of thermoplastics available to use for FDM, each with its own benefits. The three most common thermoplastics for FDM are Polylactic Acid (PLA), Acrylonitrile Butadiene Styrene (ABS), and Polyethylene Terephthalate Glycol (PETG) [3]. PLA is used for its biodegradability and its low risk of warping when produced [4]. However, PLA can be brittle and does not have optimal heat resistance [4]. ABS is a strong and heat-resistant thermoplastic, but has a higher chance of warping and requires ventilation when conducting FDM [5]. PETG is known for its ductility and chemical resistance, but needs specialized settings for FDM and has moderate heat resistance [6].

In an attempt to mitigate the disadvantages of thermoplastics, reinforcement fiber is added. The addition of reinforcement fibers with traditional thermoplastics results in a polymer composite. Reinforcement fibers are known to increase strength, thermal stability, and wear resistance, all while reducing weight. Common reinforcement fibers include Carbon Fiber (CF), Glass Fiber (GF), and natural fibers [7].

2.2 Thermal Role of Glass Fiber Reinforcement

The addition of glass fiber significantly changes the thermal properties of thermoplastic polymers including PLA, ABS, and Nylon [8]. When combined with PLA, the glass fiber commonly improves the thermal stability (to an extent) and tensile strength of the composite [8]. ABS acquires enhanced heat resistance and dimensional stability when glass fiber reinforcement is added. Nylon with added glass fiber reinforcement is known to increase thermal conductivity and the decomposition temperature of the composite [8].

Generally, the addition of appropriate fiber to a thermoplastic will most likely increase the glass transition temperature (Tg), decomposition temperature (Td), and thermal conductivity, whereas the specific heat capacity (Cp) will decrease [9]. The glass transition temperature increases due to the restraint on the polymer chain mobility. The decomposition temperature generally increases, as most fibers improve thermal stability, which has a direct effect on the temperature at which the composite starts to decompose. Generally, the fibers will store less heat than the thermoplastic that is being reinforced. This will cause a decrease in the specific heat capacity. The thermal conductivity of the composite [10] will increase because of the fibers allowing for improved heat transfer pathways [9].

It is important to note that the type of fiber reinforcement and morphology will have an effect on the properties of the composite. For instance, short fibers and continuous fibers have differing effects when reinforcing a composite [11]. Short fiber reinforcement is randomly orientated, which decreases the mechanical

strength of the composite, whereas continuous fibers drastically improve the mechanical properties. The fiber length, orientation, and volume fraction need to be considered when reinforcing a thermoplastic [12].

2.3 Thermal Testing in Polymer Composite Research

In the literature on thermal testing with polymer composites, many studies use thermal characterization instruments in order to gain credible thermal properties for the material [13]. One of the most commonly used instruments for thermal characterization is a differential scanning calorimetry (DSC). The DSC requires two crucibles - one where the sample is placed and a reference crucible. Both crucibles are heated in a controlled environment. While the heating occurs, the DSC records the difference in the heat flow between the sample and reference crucibles. The DSC records phase transitions of the sample, ultimately providing values such as melting point, heat capacity, and stability [14]. In the DSC, the specific heat capacity is calculated with the equation:

$$C_p = \frac{q}{m \cdot \Delta T}$$

where q is heat flow, m is mass, and ΔT is the temperature change.

Another common instrument used in thermal testing is a thermogravimetric analysis (TGA). The TGA requires a sample to be placed in a furnace. The furnace gradually heats up in a controlled environment, as the instrument measures the weight of the sample. The sample is exposed to varying gases, while the TGA records the temperature [13].

A dynamic mechanical analysis (DMA) is a testing instrument that can be used to obtain both mechanical and thermal data. The DMA gives data relating to the viscoelastic properties of the sample. A DMA requires a sample that will respond to a constant, oscillating force while simultaneously heating. The DMA records values such as strain, stress, storage modulus, and loss modulus. The damping factor (tan) is calculated by the equation:

$$tan\,\delta = \frac{E''}{E'}$$

where E'' is loss modulus and E' is storage modulus.

Most studies using these instruments widely accept the rules and guidelines set by the American Society for Testing Materials (ASTM). These rules give procedure protocols to guarantee safety while testing. Commonly used protocols include ASTM D3418 for DSC and ASTM E1131 for TGA [15].

2.4 Existing Gaps in PETG GF Literature

Although there has been a significant amount of research dedicated to polymer science, a multitude of gaps still need to be addressed. More specifically, there gaps remain regarding certain polymer composites, like PETG GF. Most of the research in this topic focuses on carbon fiber, or graphene reinforcement, which has been shown to enhance the mechanical and thermal properties when combined with most polymers [17,20]. However, many researchers do not consider the advantages of glass fiber, such as its lower cost, printability, and thermal insulation properties; glass fiber should be prioritized over its counterparts for specific applications where these factors are crucial. Despite these benefits, studies on glass fiber have not identified key thermal properties like the specific heat capacity and thermal conductivity, especially when combined with PETG for 3D printed applications.

Existing studies investigating glass fiber with other polymers like PLA or ABS suggest that the glass fibers help increase the thermal and mechanical properties of the material [18,25]. However, these findings cannot be assumed with PETG, as each polymer has its own molecular structure, as well as thermal behavior. Additionally, these studies specialize in observing the mechanical properties of the composites, leaving little to no focus on the thermal properties of the glass fiber composite. All of these factors leave a gap in the literature that will need to be addressed.

The proposed research question is, "To what extent does the addition of glass fiber reinforcement affect the thermal properties of Polyethylene Terephthalate Glycol (PETG) polymers in 3D-printed parts?" Based

on studies surrounding the topic and the mentioned trend of improved properties of polymer composites, the initial hypothesis is that the addition of glass fiber reinforcement will have a significantly positive effect on the thermal properties of PETG. This study proposes to discover the thermal properties of PETG when reinforced with glass fibers, in order to compare the results with PETG in its pure form, to determine how the glass fiber affected the polymer. There are many purposes for conducting this study. One is to determine if PETG, reinforced with glass fiber, can be a cost-effective counterpart to expensive thermoplastics such as polyetheretherketone (PEEK) or polyetherimide (PEI).

3.1 Materials and Methods

This study used an experimental method to investigate the thermal effect that glass fibers have when combined with PETG produced via FDM. This method was chosen in order to gain accurate numerical results of numerous thermal properties, which is important in understanding the materials performance, and determining fitting applications. A quantitative approach was chosen due to the need for numerical data and statistical analysis.

In order to assess the thermal properties of PETG, thermogravimetric analysis, differential scanning calorimetry, and a dynamic mechanical analysis were conducted using a discovery DSC, discovery TGA, Simultaneous TGA-DSC Q600 SDT, and a Q800 DMA. The TGA measured weight loss and thermal stability, while the DSC provided information regarding heat flow, crystallization temperature, glass transition temperature, and melting temperature. The DMA provided viscoelastic properties such as Storage Modulus (E'), Loss Modulus (E"), and tan delta (δ). These methods are standard in polymer research and have been utilized in similar studies for characterizing thermal behavior (Blanco, 2022).

The Glass Fiber Reinforced PETG Filament from TINMORRY (Guangdong, China) was chosen because of the popularity of PETG, and the potential performance enhancement of the glass fiber. The PETG-GF filament had polymer volume content, Vw, of 85%, and GF volume content, Vw, of 15%. The samples were produced via Fused Deposition Modeling (FDM). The FDM production method was chosen because it is the most popular method of producing thermoplastics, and is openly accessible for users.

Table 1
Printing Parameters of PETG and PETG

FDM Parameters	PETG	PETG GF
Extruder temp	250°C	260°C
Printing speed	40mm/s	40mm/s
Bed temp	60°C	85°C
Filament diameter	1.75mm	1.75mm
Fill density	100%	100%
First layer height	0.2mm	0.2mm
Layer height	0.2mm	0.2mm

The validity of the data collected was guaranteed during the data collection of PETG and PETG GF. Internal validity was maintained by standardizing the sample preparation process, including consistent drying conditions and uniform sample sizes for DSC and TGA tests. This technique minimized variation and guaranteed that the differences observed were due solely to the material properties rather than external factors. The content validity of measurement instruments was ensured by calibrating the Discovery DSC and Discovery TGA systems before testing. Furthermore, replicating measurements for each sample confirmed the construct validity and consistency of the data.

3.2 Testing Procedures

Both PETG and PETG GF were dried in a commercial filament dryer set at 70°C before being extruded via FDM. This was done to remove any moisture in the thermoplastic, which will assist with the printing quality. The two materials were printed with an Ender 3-S1 commercial 3D printer, with a hardened steel nozzle attached.

The 3D printed samples were carefully crushed to create small enough pieces weighing approximately 5 mg for the DSC and TGA tests. All samples were ac-

companied by a desiccator before testing to make sure that the samples remained dry.

The Discovery DSC was first calibrated in order to determine accurate results for the tests. The PETG and PETG GF were placed onto the aluminum crucibles, which were sealed and crimped to prevent leakage while testing. All tests in the DSC produced a heating and cooling cycle, with the temperature ranging from ambient to 300°C. The heating rate of 5°C/min was chosen to accurately capture all significant changes during the tests. The test was conducted under a nitrogen environment with a flow rate of 50 mL/min to prevent oxidation. The DSC recorded glass transition temperature (Tg), crystallization temperature (Tc), and melting temperature (Tm) during each thermal cycle.

The Discovery TGA was also calibrated in order to determine accurate results for the tests. The PETG and PETG GF were placed onto the aluminum crucibles, which were sealed and crimped to prevent leakage while testing. All tests in the TGA produced a heating and cooling cycle, with the temperature ranging from ambient to 700°C. The heating rate of 5°C/min was chosen to accurately capture all significant changes during the tests. The test was conducted under a nitrogen environment with a flow rate of 50 mL/min to prevent oxidation. The TGA recorded values such as the onset decomposition temperature (Td), the weight loss profile, and the residual mass percentage after thermal degradation.

The Q800 DMA was also calibrated in order to determine accurate results for the tests. PETG GF with dimensions of 30.0mm x 6.00mm x 2.00mm were attached to the machine with the clamps. All tests in the DMA produced a heating cycle, with the temperature ranging from ambient to 150°C. The heating rate of 2°C/min was chosen to accurately capture all significant changes during the tests. The test was conducted under a nitrogen environment with a flow rate of 50 mL/min to prevent oxidation. The DMA recorded values such as the Storage Modulus, Loss Modulus, Stress, and Tan Delta.

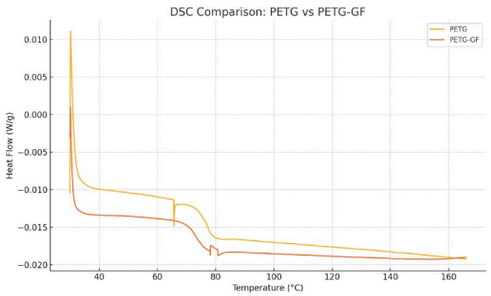
After these results were collected, the TA Universal Analysis software was used to analyze the data. The TA Universal Analysis software is an analytical tool used for TA instruments. The software visualizes data with interactive graphs, providing necessary data points for the research study.

4.1 Results

A quantitative method was used in order to determine the thermal properties of PETG and PETG GF. Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), and a Dynamic Mechanical Analysis (DMA) were used in order to record desired properties.

4.2 Results - DSC Data

Figure 1 Heat Flow



Thermal Data from DSC tests:

Table 2

	PETG	PETG GF	% Change
Glass Transition (Tg) (°C)	65.66	78.16	19.04
Crystallization (Tc) (°C)	165.64	154.36	-6.81
Melting Temperatures (Tm) (°C)	323.80	227.42	-2.31
enthalpy of fusion (ΔHf)	-1.19	-1.25	5.04
Specific Heat Capacity (Cp) (J/g·K)	1.27	1.18	−7.09

The glass transition temperature increased from 65.66 to 78.16, which is a 19.04% increase. This implies that the glass fibers being added to the PETG had a substantial effect on the stiffness of the material. Granted, the crystallization temperature decreased from 165.64 for pure PETG to 154.36 for PETG with glass fiber. The glass fiber most likely had a negative effect of the crystal growth, hence the lower value than that of neat PETG. The melting temperature saw a very minimal decrease, going from 323.80 for neat PETG to 227.42 for PETG with glass fiber. The small -2.31% change in melting temperature implies that glass fiber has a minimal/no effect on how PETG melts when heat is applied to it. The enthalpy (ΔH) showed a 5.04 percent increase, going from -1.19 to -1.25. This implies that the glass fiber addition minimally increases the thermal energy exchange for the PETG.

4.3 Results - TGA Data

Figure 2 Mass %

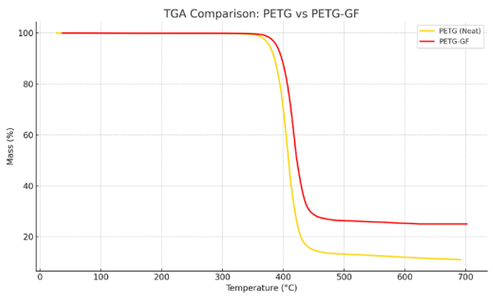


Table 3

	PETG	PETG GF	% Change
Onset Decomposition Temperature (Td)(°C)	377.32	389.2	3.15
Total Weight Loss (%)	89.01	75.0	-15.74
Residual Mass (%)	11.00	75.0	127.27%

The onset decomposition temperature saw only a 3.15 percent increase, having 377.32 °C for Neat PETG and 389.2°C for PETG with Glass Fiber. This implies that the thermal stability increased when glass fiber was added onto PETG. This suggests that PETG with glass fiber is harder to break down (decompose) than neat PETG. In addition, the total weight loss for PETG saw a -15.74 percent reduction. This is because the glass fibers assisted in minimizing the degradation of the PETG when combined with it; the neat PETG degraded normally. However, the residual mass percentage for PETG with glass fiber is nearly double when compared to PETG. This is simply because of the glass fiber residue that was left after the test was complete. It is expected that the glass fibers did not decompose with the TGA tests, explaining why there is glass fiber residue at the end of the test.

4.4 Results - DMA Data

Figure 3 Storage Modulus

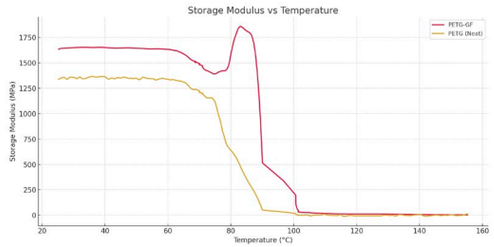


Figure 4 Loss Modulus

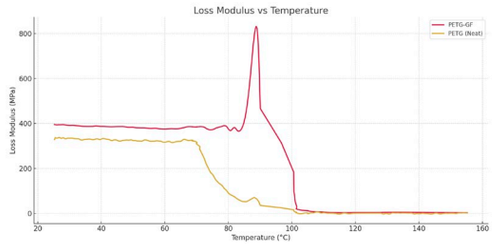


Figure 5 Tan Delta

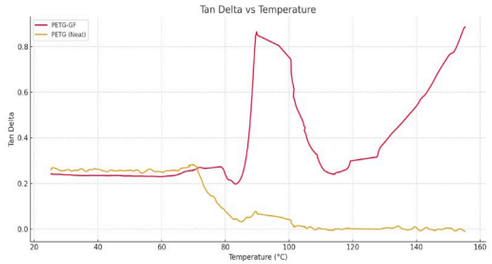
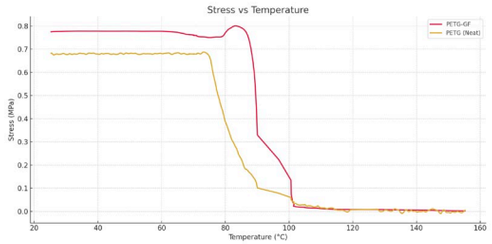


Figure 6 Stress



Overall, the graphs show a higher storage modulus, a higher peak for loss modulus, a higher tan delta, and a better stress graph when comparing PETG GF to PETG. The graphs show that PETG GF maintained a higher storage modulus than PETG. This means that PETG GF is stiffer than neat PETG because of the glass fiber added. PTEG GF had a higher loss modulus than the neat PETG. This means that PETG GF is better at absorbing mechanical energy than PETG. PETG shows a higher tan delta graph, meaning that PETG GF has a higher glass transition temperature, which is verified by the DSC data. The stress graphs show that PETG with glass fiber does better than neat PETG when stress is applied.

5.1 Discussion

The purpose of the study was to answer the question "To what extent does the addition of Glass Fiber affect the thermal properties of PETG produced via Fused Deposition Modeling?" Since the purpose of this study was to determine the thermal properties of PETG and PETG GF and compare the values, an experimental procedure with instruments such as a DSC, TGA, and DMA was taken because these instruments would yield the numerical values needed to analyze the effect of glass fiber. Overall, the data showed that the glass fiber had a minimal, but noticeable effect on the thermal properties of PETG. More specifically, the DSC showed a 19.04% increase in the glass transition temperature and a 7.09% decrease in specific heat capacity when glass fibers were added to PETG. The TGA showed a 3.15% increase in decomposition temperature, and a 15.74% decrease in thermal stability. The DMA showed a ≈ 25% increase in storage modulus. Overall, the glass fiber had a minimal effect on the PETG's thermal properties, but can be important for applications in the medical field.

5.2 Implications

There are many implications of the finding that the glass fiber had a minimal effect on the thermal performance when combined with PETG. This research can be applied to industries that need materials for high-temperature applications. To reiterate, the addition of glass fiber reinforcement does not substantially affect the thermal properties when combined with PETG. A study conducted by Saidova et al. [26] agrees with the results presented in this study, as they found that the addition of glass fiber affects the thermal properties, but other factors that the composite exhibits have a more direct impact on the thermal properties. This said, one cannot assume that since a reinforcement (like glass fiber) improves the mechanical properties of the composite, the thermal properties will also be positively impacted in turn. The results in this study found similar findings to those of Arrakhiz et al. [27], in which they found that doum (low-density polyethylene) drastically increased the mechanical properties of composites, but that the higher the doum concentration, the lower the thermal properties. A direct analysis of the glass fiber and doum should be conducted to observe similarities between the two reinforcements. In applications that require high temperatures in the automotive, medical, and aerospace industries, this study proposes to disregard glass fiber as a candidate for reinforcement.

For high temperature applications, other reinforcements should be explored, or plastics like polycarbonate or PEEK should be used.

5.3 Limitations and Future Research

This study helped develop the current body of knowledge surrounding polymer reinforcements, specifically glass fiber. This study shows that the effectiveness of glass fiber on thermoplastics like PETG is minimal when considering the thermal properties. It is currently acknowledged in the literature that the addition of glass fiber significantly increases the mechanical properties when combined with a thermoplastic, such as PETG. However, the thermal properties of the glass fibers when combined with PETG had a slight change. That said, this studu had many limitations that could have potentially influenced the data acquired.

Firstly, the inevitable porosity in PETG GF (as well as all thermoplastics produced via FDM) could have an effect on the results given by the instruments. In fact, porosity can be attributed to a lack of interfacial bonding strength between the GF and the PETG matrix, which could result in a reduction in the mechanical and thermal performance of FDM printed parts.

In addition, this study only tested PETG combined with 15% glass fiber. Although multiple trials were conducted to verify the composite's performance, having different concentrations of glass fiber content in PETG (ex. 10%, 20%, and 30%) could provide a better understanding of the results and the effect of the glass fibers.

In the future, research should be conducted to find out what reinforcement (ceramic, nylon, carbon fiber) optimizes the thermal properties when reinforcing PETG. This will reveal the extent to which PETG maximizes its thermal properties when in combination with a reinforcement. Additionally, using different manufacturing techniques to produce PETG GF (Material Jetting, SLS, etc.) should also be explored. This will reveal the extent to which different manufacturing methods have an effect on thermal and mechanical properties of polymer composites.

6.1 Conclusion

In conclusion, the results of this study suggest that glass fiber reinforcement does not have a positive effect on the thermal properties of PETG. These results, however, did affirm glass fibers' ability to increase the stiffness and strength of the composites. These conclusions were drawn after conducting three tests of the material: the DSC, which gave insight on the heat flow; the TGA, which gave insight on the weight loss; and the DMA, which gave information regarding stiffness and strength. The study helped fill the gap in the body of knowledge about the thermal effect glass fiber composites have on PETG. This study added that the thermal properties had only minimally increased for this composite.

The study conducted has many implications, focusing on industries that need certain materials for different applications. There is a widely debated issue on whether materials like glass fiber should be used for applications that require heat and high strength. This study supports the idea that glass fiber increases the mechanical strength when combined with a thermoplastic, but the thermal increase is minimal. This study can serve as a warning for industries like manufacturing and aerospace that this material is not suggested for high temperature applications, despite the conclusions drawn from other studies in the past. Instead, industries should invest in polymer materials with better molecular structures (like PEEK & polycarbonate) to better fit the need. This study had many limitations, so future research is needed to further investigate this phenomenon and affirm this notion for glass fiber reinforced composites.

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