Thermal Stability studies on PLA –SS316L reinforced filaments for FDM 3D printing produced through Conical Twin Screw Method

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Abstract:

This study focuses on the fabrication and processability of stainless steel (SS) 316L powder-reinforced polylactic acid (PLA) composite filaments with solid loadings ranging from 2.5 wt. % to 10 wt. %, with increment of 2.5 wt. %. A conical twin-screw extruder with four distinct heating zones was employed for melt compounding, followed by water cooling and filament spooling. The extrusion process was carefully optimized to achieve a consistent filament diameter of 1.75 ± 0.05 mm, with the highest extrusion temperature set at 170°C. Thermal stability was assessed through Thermo Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC), revealing that the composite filaments remained stable up to 350°C, with melting points in the range of 150°C to 160°C.

Keywords: *PLA matrix, SS316L reinforced, 3D printing, TGA, DSC.*

1. Introduction

Material extrusion is a type of additive manufacturing process, used in 3D printers where the material has drawn through a nozzle, heated, and then deposited. This nozzle moves horizontally whereas the build platform moves vertically. By this way, layers are formed. As the heating progresses the previous layer attaches with the current layer [1]. Temperature and the chemical agents control the bond between layers. Fusion Deposition Modelling (FDM) is a commonly used additive manufacturing technology in various engineering fields [2]. Over last several years researchers have been able to produce complicated geometric characteristics rapidly in a variety of situations using additive manufacturing technologies[2,3]. The concept was first introduced by Stratasys and Scott Crump included the development of FDM technology at the company in 1989. Using this method, 2D layers are successively laid off along the vertical axis to create 3D structures. Since Hull's original invention, AM is thought to be a rapidly expanding field that has seen significant technological advancement [3]. FDM used to produce a wide variety of moulded components derived from the filament. The excellence of moulded parts through this process is quantified by tensile strength, surface roughness, and the flexural strength [4]. FDM process, which uses the layer-by-layer manufacturing approach to create parts made of porous materials such as polymer, concrete, metal or a composite is managed by a rapid prototype computer [5]. Using 3D design software, a digital design is created and generated for FDM and then divided into multiple layers. The printer receives this layer data and uses it to reproduce the design layer by layer until the full model is created. Additionally, the re-melting of the existing layer when the successive layer built enhances the layer growth [6].

2. Experimental Setup

PLA granules with an average size of 4 mm and a density of 1.2 g/cc (supplier: Rever Industries) are employed as the matrix in this investigation. The reinforcement is provided by the stainless steel 316L grade powder (source: INDO-MIM Pvt. Ltd), which has an average particle size of 30 µm, a tap density of 4.94 g/cc, and a pycnometric density of 7.95 g/cc.

To create composite filament, a counter-rotating conical twin screw extruder is employed. The machine has a main hopper underneath it and two gravimetric feeders, A and B, at the top that are used to fill it with powders and granules. Direct guidance of the material from the main hopper onto the conical twin screw enclosed in a closed chamber occurs. For each of the two feeders, the pace at which material enters the hopper can be independently adjusted. Metal powder is supplied more slowly than PLA because of its higher density. The filament die assembly, which is located at the other end of the conical twin screw, is where the necessary filament size is obtained. The experimental setup for the filament production is shown in Figure.1.

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are used to analyze the thermal stability of the composite filaments that meet the necessary testing requirements and are less than printable size.

Figure1. Experimental setup used for composite filament fabrication.

3. Results and Discussions

3.1 Thermogravimetric analysis

One type of thermal study that is typically done following filament production is thermogravimetric analysis (TGA). It is advised to research the temperature stability of the filament material prior to 3D printing. TGA is a common method for determining the thermal stability of any material, including polymers [10]. The change in mass content of a sample exposed to a variety of temperatures in a controlled atmosphere is used to evaluate thermal stability. In order to prevent thermal reactions, TGA entails bringing the sample's temperature up to the degradation temperature either linearly or in phases that are separated by isothermal intervals while a gas is present. The sample material's thermal stability within the tested temperature range is confirmed by no mass change in response to a temperature increase.

As seen in Figure.2(a), TGA is performed in the current investigation using an HITACHI-STA 200 simultaneous thermal analyzer. In an environment of nitrogen gas, all of the samples are progressively heated from room temperature to 550ºC at a rate of 10ºC per minute. Nitrogen gas is purged at a rate of 100 milliliters per minute. The test samples, which are smaller than 5 mg in weight each, are taken out of the extruded filaments of all compositions at random intervals and put into the crucibles that correspond to them in the main chamber. After that, the crucibles inside the heating chamber are gradually heated from ambient temperature to 550ºC as part of the heating process. As the samples are heated, the temperatures and changes in mass inside the chamber are noted. Most organic polymers are thermally stable up to 230°C. As the temperature is gradually increased, the weight of the PLA decreases, leaving SS 316L behind in the sample tray. The residual mass of the samples obtained from TGA confirms the composition of all the filled strands (wt% of SS 316L). TGA curve for five different composite filaments are shown in figure.2(b)

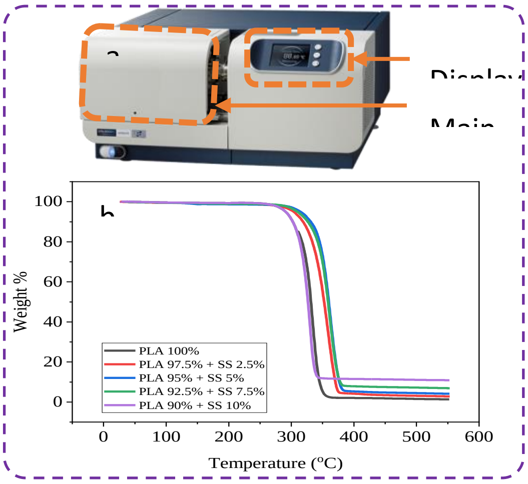

Figure.2 Thermogravimetric analysis (a) experimental setup and (b) plot depicting the thermal stability of the different composite filaments.

A straight line on the TGA plot means there is no weight loss of the composite, which means there is no degradation of the polymer in that temperature range. At higher temperatures, the metal powder may remain intact and the polymer may degrade. This polymer deterioration causes the weight loss of the test specimen, which can be realized from the sudden vertical drop in the graph. Further increase in temperature causes the weight loss of the composite specimen due to complete deterioration of the polymer inside. As a result, the highly filled composite samples stand out on the graph due to their high loading weight compared to the other samples. The weights of the various composites recorded after the polymer is completely decomposed remain constant and hence are observed in the form of a straight line on the graph. The vertical axis of these lines identifies the composition of the corresponding composite material.

3.2 Differential Scanning Calorimetry (DSC)

The melting, crystallization, and glass transition temperatures of the polymer composite filaments are monitored in this work using DSC. Every time, the composite sample is added to the sample pan with a predetermined infill load. The sample and reference pans are sealed and maintained inside the DSC cell, where they are heated using a single heating source to reach 550°C in an inert nitrogen medium at the same rate of 10°C per minute from room temperature. At 100 ml/min, the nitrogen purging rate is continuously maintained. As shown in Figure 3., the plots are obtained for the five various infill loadings and are analyzed for each composite filament's melting and degradation temperatures.

Determining the polymer's melting point and safe operating temperatures is the aim of DSC investigations. Endothermic events, such as melting and deterioration, occur when heat is absorbed by the substance and changes its condition. These occurrences are distinguished by a DSC plot's downward peak. Table 1 lists the determined melting (Tm) and degradation (Td) temperatures. When adjusting the printing temperature during a 3D printing process, this information is useful. The filament is kept safe by the degradation temperature, over which burning of the polymer may occur.

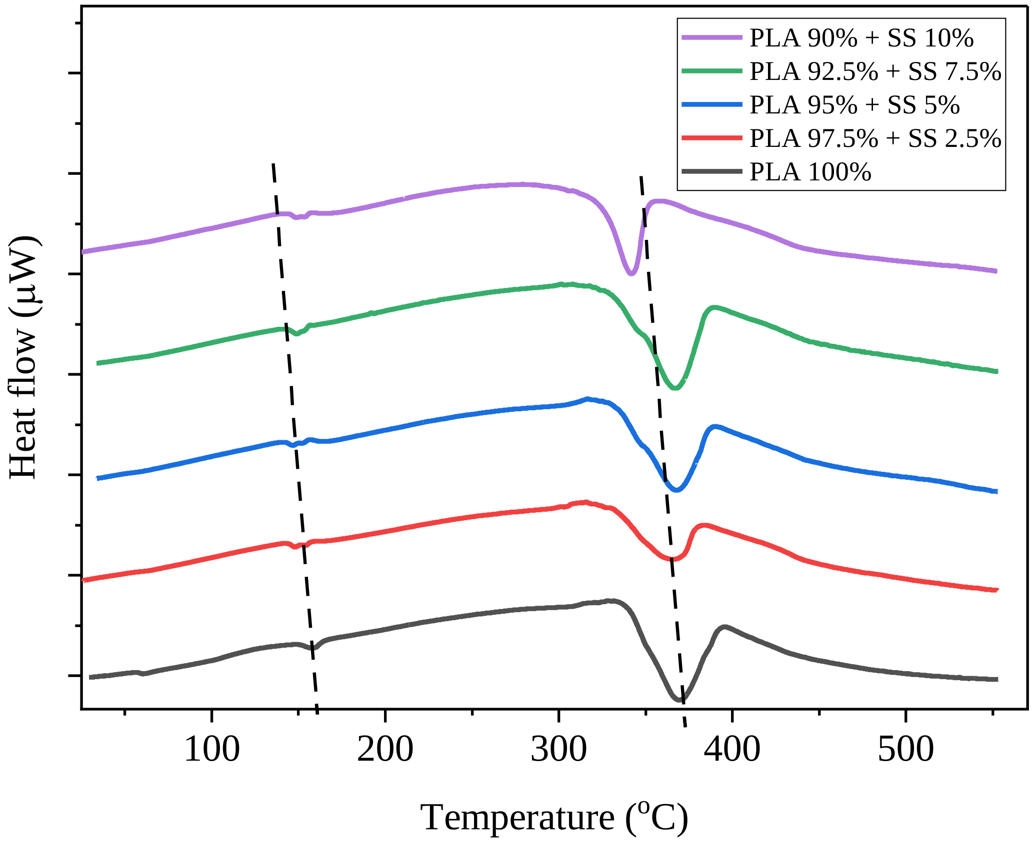

Figure.3 Differential scanning calorimetry plot – Effect of temperature on heat flow of the composite filaments.

The heat flow versus temperature plot indicates that the melting temperatures of all the composites, which include 0, 2.5, 5, 7.5, and 10% PLA + SS316L, are nearly identical, falling between 151°C and 159°C. The temperature range between 345°C and 370°C is when the breakdown of the aforementioned polymer composites happened.

Table.1 Melting and degradation temperatures of composite filaments

	0 wt. % SS	2.5 wt. % SS	5 wt. % SS	7.5 wt. % SS	10 wt. % SS
T_m (°C)	159	154	153	151	152
T_d (°C)	370	367	367	367	345

When determining the maximum temperature for 3D printing and filament extrusion, the melting point information shown in Table 1 is crucial. All of the filaments had melting points between 150 and 160 degrees Celsius. Once the filament reaches 345°C, it can start to deteriorate. The temperature for filament printing is set at 210°C based on this information.

4. Conclusions

In this study, the manufacturing processibility of PLA composite filaments loaded with SS 316L powder at varying loadings from 2.5 weight percent to 10 weight percent is examined, with increments of 2.5 weight percent. TGA and DSC coupled experimental setup is used to study the thermal stability of the composite filaments. According to the findings, all of the filaments are thermally stable but will eventually break down at temperatures higher than 350°C. All of the composite filaments have melting points between 150°C and 160°C.

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