Processability of SS316L powder - binder mixtures for vertical extrusion and deposition on table tests

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Abstract:

Metal injection moulding (MIM) feedstocks are mixtures made of a solid metal powder and a viscous polymeric binder. Vertical extrusion of MIM feedstock has seldom been investigated in the scientific literature, but it is the enabling technology for extrusion-based additive manufacturing processes (EAM). The EAM process adopts the movement of an extruder, relative to a build table, to deposit thin strands (roads) of the mixture and grow a 3D object, layer by layer. EAM may find applications for the consumer as well as industrial products.

This paper addresses some of the challenges involved in achieving a consistent flow of molten feedstock mixture and discusses the consequences of the main process parameters on the extrusion-based manufacturing process. This work also relates the rheological behaviour of feedstock to the extrusion process, aiming at the dimensional stability of parts.

An experimental study was performed on SS316L feedstock with a water-soluble binder system, by varying the percentage powder content of the mixtures, the extrusion temperature, the extrusion rate, the deposition table speed and the nozzle shape. The study evidences and explains why higher powder loading values and lower extrusion temperatures are the most useful conditions in order to obtain a stable flow with reduced swelling/shrinking phenomena.

Keywords: additive manufacturing, extrusion, feedstock, viscosity

Nomenclature:

Acronyms		Units	
PIM/ MIM	: Powder/ Metal Injection Molding	kg	: kilogram
AM	: Additive Manufacturing	mm	: millimetres
FDM	: Fused Deposition Modelling	m	: metres
EAM	: Extrusion based Additive Manufacturing	min	: minutes
D_n	: Nozzle diameter	s	: seconds
L	: Nozzle length	^{o}C	: ^{o} Celsius
D	: diameter of extruded wire		

ϕ	: Solid loading (fraction)
ϕ_v	: Volumetric Solid loading (%)
T_e	: Extrusion Temperature
v_e	: Extrusion velocity
v_t	: Table velocity
$\dot{\gamma}$	: Shear rate
τ_w	: Wall shear stress
η_a	: Apparent viscosity
h_c	: critical nozzle height

1. Introduction

Powder Injection Molding (PIM) is a powder metallurgy technology suited to the manufacture of complex shaped metallic and ceramic parts, for a diverse range of applications. The typical PIM feedstock is made of metal or ceramic fine powder, homogenously mixed with a limited amount (usually less than 50% by volume) of a thermoplastic polymeric binder. Well established processing routes for different materials and products by powder injection molding are practiced for mass production [1]. The process chain is divided into 4 conventional steps; the first is compounding of metal or ceramic powder with polymer to obtain a homogeneous feedstock.

In the second step, in conventional PIM, the feedstock is injected through an injection molding unit, into a cooled mold to get a solid, powder-filled part. However, the feedstock can also be extruded to various shapes. For example, the production of thin-wall tubes can be utilized in applications such as filters, thermowells, heat exchangers, etc. [2] [3].

As a more recent development, the extruded filament can be deposited according to a designed path, as in Fused Deposition Modeling (FDM) techniques, for obtaining 3D parts. Several additive manufacturing (AM) techniques have been developed to build geometries by a layer-wise "3D printing" deposition approach [4] [5] [6]. Freeform extrusion [7], a relatively new type of powder-based AM, is an economically advantageous technique for manufacturing complex shaped metallic and ceramic articles [8] [9].

The mold-injected, extruded or 3D printed feedstock is a so-called "green" part. In the third step of the process chain, thermal and solvent-based debinding techniques are used to remove water-soluble or organic binder constituents. A porous structure is formed in a part thereafter. As the fourth step, the debinded ("brown") part is finally sintered to achieve desired densification and mechanical properties [10] [11].

In cylindrical extrusion, the shape of the extruded rod can result somewhat different from what expected since different phenomena, occurring at the die exit, can affect the material flow. The diameter of the extruded filament is not exactly equal to the nozzle diameter; in fact, the extruded filament undergoes swelling [12], immediately after the exit nozzle, followed by a shrinking due to cooling. Some defects may be visible on the extruded filaments. If restricting the attention to cylindrical extrusion, ovalization of wires may occur, partly due to the incorrect nozzle cylindricity and alignment. Another possible defect is the bending of the wire axis, immediately after extrusion or later during sintering. Another defect is the sagging, leading to narrowing of filaments with increasing length of the extruded, observed during vertical extrusion. This is due to the increasing weight of extruded matter, which pulls the plastic material immediately below the nozzle and before the polymer fraction cools down below the glass transition temperature. This defect is more severe when extruded at higher temperatures [13].

Not only the average dimensions of the extruded filaments are technologically relevant, but the extrusion instabilities can generate dimensional variations and surface irregularities [14], which are considered a defect of the extrusion process. Such instabilities usually occur at high flow rate and thus may limit the actual output rate attainable.

1.1 Effect of material parameters

The extrusion of powder/binder feedstock is a process, based on plastic forming, which requires specific material properties: the first requirement is flowability. The material to be extruded must be flowable during the extrusion process to form the desired cross-section as that of die or nozzle [15]. Since at the typical extrusion temperatures (which depends on the binder), the polymer fraction is melted, the flowability is strictly related to the viscosity of a multi-phase suspension of solid particles in a viscous melt.

The second requirement is green strength and stiffness; the extruded or 3D printed part must be strong and stiff enough to avoid ruptures and deformations either induced by its own weight or by handling it. Both requirements mostly relate to the interaction between the binder system and the powder characteristics and content [16]. [3]

In addition to binder viscosity, flowability is remarkably influenced by powder characteristics such as mean particle size, particle size distribution, particle shape and specific surface area, because these parameters relate solid loading, inter-particulate friction, and viscous flow during extrusion

[10]. The formulation of feedstock mixture interacts with the characteristics mentioned above and influences the final quality of parts. The spherical shape of powder particles is preferable, because it reduces internal-particulate friction and it avoids particle interlocking; spherical shape allows better flow, which in turns enable more homogeneous microstructure of parts. On the contrary, irregular particle shape increases mechanical interlocking, but it allows greater sintered density. The maximum theoretical volumetric packing of spheres of identical size is $\phi_{v\_max} = \pi/\sqrt{18} \cong 0.74$. On the other hand, multimodal particle distribution offers the benefit of higher possible solid loading, because of a denser packing, which subsequently results in good green strength, lower sintering distortion and high sintered density [17].

The viscosity of the powder-binder mixture is dramatically sensitive to solid content, i.e. to the amount of powder fraction: viscosity increases as the powder loading increases [18].

The high viscosity of the mixture is associated to a marked non-newtonian behavior; as a matter of fact, the polymeric binder is expected to show a pseudoplastic behavior; moreover, disaggregation of powder agglomerates during extrusion can also lead to a progressive reduction of viscosity on increasing flow rate. Provided a relatively high flow is maintained, a mixture with consistent powder content but convenient fluidity can be produced; a proper choice of powder content and extrusion rate is thus the key for obtaining a good quality extruded filament of adequate powder content [3].

1.2 Effect of extrusion unit and process parameters

The extrusion unit is responsible for taking the raw feedstock, heating it up in the loader and extruding it through a small nozzle to extrude or deposit a filament on the build-surface [19]. Precise flow control in the extruders is necessary to ensure the dimensional stability of the extruded filaments, and therefore of the parts made during the 3D printing (or vertical extrusion) process [20]. An extruder with perfect control of the flow of feedstock is expected to have an output rate precisely equal to the commanded, or desired flow rate. This control of the flow in an extruder is not trivial to achieve. Zinniel and co-authors [21] revealed that an uncontrolled flow rate, even with small fluctuations, can contribute to significant variations in the quality of the final 3d part, including gaps and extruded thickness (or diameter) variations. The discrepancy between desired and actual flow rate are due to three characteristics of molten plastic flow, including the time taken to ramp up/down the flow rate and the delays in the flow rate response.

Some literature discusses the volumetric flow errors, i.e. errors that result from discrepancies between desired flow rates and expected output flow rates, in terms of process variables and control system to improve flow accuracy of a pure thermoplastic melt in conventional FDM, Fused Deposition Modeling [22]. The extrusion rate and head velocity (i.e., the travelling speed) of the extruder must be synchronized to conform predicted flow rate at every point [23] [24]. The approximate dynamics of extruder and flow characteristics of the feedstock obtained from the viscosity model is useful for setting the travel velocity of the extruder to compensate for the non-ideal (non-Newtonian) flow characteristics of the feedstock. Another way which is being attempted for controlling the extruded flow rate is to utilize a modified control signal that compensates the real-time flow variation [25].

1.3 Objectives of the present paper

The currently available scientific literature that tries to correlate the material properties and the process parameters to the shape, size and stability of the extruded filaments is limited to the extrusion of pure polymeric melts, which obviously have significantly different rheological properties, if compared to the paste feedstocks used in PIM and related processes. In the literature, there is a lack of experimental information, and therefore a lack of knowledge about the effect of extrusion process parameters on the dimensional quality of extruded paste filaments to be used especially in AM processes. The general objective of this paper is to fill this lack.

Figure 1 schematically shows that each building unit of feedstock (a strand of wire) during extrusion or deposition relates with both material (powder and binder properties) and process parameters comprise of extrusion system and deposition system described in above Sections 1.1. and 1.2 through flow properties.

The specific objectives of this paper are:

- to develop an experimental proof for confirming the hypothesized correlations between material/process parameters and flow properties (as shown in Figure 1), and thereby product quality, here extruded wire;
- to identify the effect of gravity pull on dimentional stability of extruded wires;
- to understand the influence of feedstock flow properties on characteristics of extruded wires;
- to discuss the consequences of viscoelastic effects on quality of extruded products.

2. Materials and Methods

2.1 Materials and Feedstock Preparation

In this paper, stainless steel 316L feedstock is used with a diverse range of powder loadings. A commercially available stainless steel powder (grade: SS316L; by Sandvik Osprey) having mean particle size 8.8µm (distribution: D_{10} = 4.1 µm, D_{50} = 8.8 µm D_{90} = 15.9 µm) was used as a raw material (Figure 2 (a)). The chemical composition of powder is given in Table 1.

Commercial water-soluble binder (Embemould K83), a multi-component mixture (Density ρ = 1.05 g/cm^3), usually employed for powder injection molding, was used to formulate the feedstock. Binder constituents and powder were premixed in different volumetric proportions to accommodate the typical range (ϕ_v = 50 - 65 % [26]) of solid loading in MIM feedstock using turbula mixer at room temperature. Three mixtures having weight fraction ϕ = 0.875 (ϕ_v = 50%), 0.900 (ϕ_v = 54%) and 0.925 (ϕ_v = 63%) of powder loading were then separately compounded using a Brabender - Plasti-Corder mixer at 140 ºC for 30 minutes. These feedstock mixtures were processed through a twin-screw extruder at 140 ºC. This long feedstock preparation chain and the small granulometry of the metal powders have been chosen in order to obtain a highly homogeneous (as appreciated from Figure 2 (b)) feedstock for subsequent operations.

The viscosity of the feedstock mixtures was measured using a rheometer (Rheometrics RDA II) with parallel plates configuration. The sample of feedstock was placed between the parallel discs of 25 mm diameter maintained at a distance of 1 mm away from each other. The viscosity tests were performed in the temperature range between 110 and 140ºC, while the shear rate ($\dot{\gamma}$) was between 1 and 100 s^{-1}. This method is appropriate for viscosity measurements of SS 316L feedstock because the range of shear rate and wall shear stress during the deposition and extrusion are similar to those during the viscosity measurement.

2.2 Feedstock extrusion and deposition tests

The machine used for the tests is shown in Figure 3 (a); it includes (i) a work table operated through parallel kinematics (linear delta), with controlled acceleration of actuators, (ii) an extrusion system having two distinct pistons, respectively for feeding (oriented at 45° to the vertical) and for extruding (vertical) [27]. The feeding cylinder is heated and internally equipped with packed steel balls. The preheated flow enters the extrusion cylinder through a restricted gate. The pre-heating inside the feeding cylinder, the balls and the gate all provide shearing of the viscous flow and further homogenization of the feedstock. The CNC servo-control of the extrusion piston has an excellent positioning precision, below 0.0005 mm. The temperature is servo-controlled by means of three thermocouples: one at the feeding cylinder, one at the injection cylinder and one at the nozzle. The precise control of extrusion piston position and nozzle temperature ensures precise flow of molten feedstock. The flow rate variability only depends on homogeneity of the material, which has been maximized during the preparation and feeding phases.

This machine is suitable for extruding varieties of feedstock mixture in required extrusion shapes. A sectional view of extrusion system (Figure 3 (b)) shows the internal flow channels of the unit wherein synchronized movement of feeder piston supplies material to the extruder cylinder and the extruder pushes material out of the nozzle for controlled extrusion/deposition.

Two extrusion nozzles (shown in Figure 4) were used in this study to understand the effect of nozzle geometry on extrusion characteristics. The two main nozzle design parameters are the throat length L and the throat diameter D_n. Thin D_n conduits and long nozzle increase frictional losses at a lower temperature, due to the high viscosity of the tested feedstock. Conversely, too short throats might favor mid-air bending of the extruded wire [28]. Distinct nozzle diameters, D_n, (0.9 and 0.6 mm) have been used, to change the flow characteristics as apparent shear rate $\dot{\gamma}$ decreases with increase in D_n, according to the well known equation (1):

$$\dot{\gamma} = \frac{32Q}{{D_n}^3} \quad (1)$$

where Q is the extrusion flow rate. Pressure drop increases with increase in length L, as for equation (2) [29]:

$$\tau_w = \frac{\Delta P \, D_n}{4\,L} \quad (2)$$

where τ_w is the wall shear stress and ΔP is the pressure drop in capillary length of the nozzle. Nozzle 1 has been selected with larger diameter and smaller D_n/L ratio, in order to reduce shear rate and increase shear stress with respect to nozzle 2.

Another tested experimental parameter is the extrusion temperature, i.e. the temperature T_e at the nozzle. Too low an extrusion temperature (below 110ºC) is not feasible because it would require a very high pressure, with a risk of exceeding the available power of the extrusion unit and excessively increase the shear stress. At higher T_e (above 140ºC), the binder may degrade inside the feeding and extrusion cylinders and cause a discontinuity in the feedstock flow. A suitable range for T_e was therefore selected as 110ºC to 140ºC.

Wire extrusion experiments were performed in two test modes (a) vertical extrusion and (b) deposition on the table. In vertical extrusion experiments, wires were extruded of 180 mm length keeping worktable at a distance of 200 mm from the nozzle. Whereas, wires of length 180 mm were deposited on moving worktable keeping 5 mm apart from the nozzle. The different levels of table velocity v_t were selected according to the levels of v_e.

Experimental levels of above parameters are given in Table 2. About 150 experiments were performed to study the effect of solid loading (ϕ), nozzle geometry (D_n), extrusion temperature (T_e), extrusion velocity (v_e), table velocity (v_t) on the wire diameter. The v_e and v_t levels have been selected in order that only 3 possible values of v_t /v_e ratios are possible: ~0.755, 1.000 and 1.250. Feedstock wires of approximate length 180 mm were collected during vertical extrusion tests, whereas feedstock wires of length 180 mm were deposited on table during deposition on table tests. Thus about 150 wires are considered for analysis.

2.3 Characterization of extruded wires

Out of the extruded length (180 mm), a length of 15 mm on either side of wire was trimmed off, and the central 150 mm length was retained for measurement of wire diameter. Diameters of extruded wire were measured using a 3D vision measuring machine (make: Mitutoyo). All wire diameters were measured at 8 different locations chosen periodically on the selected length of 150 mm. The mean wire diameter (D) and its standard deviation (σ_D) were determined from the 8 measurements on each wire. The microstructure of the extruded wires was analyzed using scanning electron microscopy (Zeiss EVO 50XVP SEM).

3. Results

3.1 Rheology of feedstock at low strain rates

Figure 5 shows some of the results of viscosity measurements for the selected feedstock using a parallel plate rheometer. The shear rates during the high-pressure PIM processes are approximately 100 to 1000 times higher than the expected shear rates during extrusion tests. The viscosity behavior for such conditions, which is available for some feedstock in the literature, is not usable for this study. Feedstock are tested a deliberately lower range of shear rate to serve the purpose.

During the initial trials, an abrupt decrease in the apparent viscosity with shear rate was observed. This was because of the low level of shear stress resulting from a thin layer of the binder formed near the wall (wall slip), which acts as a lubricant and lowers the friction of the fluid flow. This wall slip effect was reduced (but not completely avoided) by using a roughened parallel plate surface.

Although the data are affected by the wall slip phenomenon, i.e. the actual viscosity values are underestimated, still they provide clear and useful information about the influence of temperature and solid loading. Figure 5 shows a very strong influence of the solid loading on the viscosity, and a limited influence of temperature in the investigated range. In all tested cases a very steep decrease in viscosity with increasing shear rate can be observed. This is because these feedstocks behave as shear-thinning fluids, showing pseudoplastic behavior. This shear thinning behavior can be expressed by the following exponential relationship that models the apparent viscosity η_a:

$$\eta_a = K\left(1 - \frac{\varphi_v}{\varphi_{v\_max}}\right)^{-m} \dot{\gamma}^{n-1} e^{E/RT} \quad (3)$$

where the consistency K (measured in Pa·s) and the sensitivity exponents *m, n* and β are material constants, *T* is the mixture temperature, T_0 is a reference temperature (in this case T_0 = 140 °C), ϕ_{v_max} is the maximum packing volumetric solid loading. A smaller value of *n* corresponds to higher shear thinning. Larger n-values are generally preferred for solid-binder mixtures, because they reduce segregation problems and because they reduce the sensitivity of the viscosity to the shear strain rate [30]. According to equation (3), viscosity is as an increasing linear function of the powder loading ϕ_v. Unlike injection molding, in wire extrusion operations larger consistency values might be preferred, because it is more important to have a stable and stiff material, rather a low viscous one.

A regression model on the logarithmic terms has been developed for the available experimental results, yielding the rheological constants. The regression model is very robust, with R^2=87% and quasi-normal residuals. The calculated consistency is K = 43 Pa·s. As expected from Figure 5, the calculated temperature coefficient is very low, β = 0.058. The estimated m-value is quite large, equal to 1.5, indicating a very strong dependence of viscosity on the solid loading.

The extrusion unit which will be used for the tests is a CNC controlled piston, which is extremely precise at the low speed values which are typical of additive manufacturing techniques. Therefore, the expected shear rate inside the extrusion nozzle should be highly constant and, due to wall slip, a negligible axial velocity gradient should be present at each cross section of the extrusion nozzle.

3.2 Dimensional variation of extruded wire during vertical extrusion tests

The measured diameters of the green extruded wires (D) after cooling were used to calculate the ratio D/D_n, which quantifies the diametric variation (shrinkage if $D/D_n < 1$ and swelling if $D/D_n >$ 1) during vertical extrusion and deposition on table tests. A statistical analysis of the experimental data, performed through a General Linear Regression Model of the data, has been used to assess the statistical significance of mentioned main effect and interactions.

The effect of extrusion temperature T_e and solid loading ϕ on the extruded wire, for the two nozzles, is shown in Figure 6. In most cases, shrinkage is observed rather than swelling. The reason is that in vertical extrusion testing, the weight of the extruded wire, while it is still plastic, reduces the initial die swelling and further thins the diameter, with simultaneous diameter shrinkage and elongation of the extrudate after discharge.

As it can be seen from Figure 6, there is a strong interaction of the nozzle diameter with both extrusion temperature and solid loading. Some shrinkage is observed, as long as the temperature remains within 130°C. When T_e reaches 140°C, the smaller *nozzle 2* (D_n = 0.6 mm) generates a little swelling, which is not compensated by the gravity loading. On the contrary, at 140°C the larger *nozzle 1* (D_n = 0.9 mm) produces a significant shrinkage, with D/D_n being about 0.84. The effect of the nozzle diameter, in combination with the other two parameters, is not trivial. Dissimilar shrinkage of the extruded wire with nozzle geometry is a proof of the distinct solidifying shrinkage of binders in the feedstock material [31]. In the smaller nozzle (D_n = 0.6 mm), larger and sudden shear deformation takes place in a short length (*nozzle 1* has a smaller L/D_n ratio), i.e. in a shorter time. This should intuitively lead to a larger swelling, due to the viscoelastic recovery, rather than to a larger shrinkage. The packing of the powder in the feedstock changes from close to loose as the material flows out of such a narrow section [32]. In the larger nozzle (D_n = 0.9 mm), a larger flow rate is allowed and the longer length of the die prevents swelling. The hot and fluid extruded wire therefore thins considerably because of its own weight (gravity effect). These different behaviours between the two nozzles emerge only at 130 °C of extrusion temperature. From a technological point of view, the use of temperatures below 130°C make the process less sensitive to the nozzle diameter.

A very important interaction can be observed between the D_n and the solid loading ϕ. It is clear from Figure 6 (b) that the smaller nozzle is more stable with respect to the mixture composition, because D/D_n is closer to 1 for all ϕ-values. Instead, the larger nozzle diameter has an acceptable shrinkage only in combination with the larger ϕ-value and at low temperature.

The smaller nozzle (diameter 0.6 mm) is not significantly sensitive to temperature nor to the solid loading. This result is also very important from a technological point of view, because not only smaller nozzles allow for better accuracy in 3D printing, but they also lead to better dimensional stability, according to the present results. With the 0.9 mm nozzle, that allows a larger, more massive flow rate, there is a significant shrinkage in vertical extrusion for both ϕ = 0.875 and ϕ = 0.90, especially at 140°C.

Shrinkage increases for feedstock with smaller solid loading ϕ (i.e. larger amount of plastic content) because when there is more polymeric binder content and larger flow rate, the melt is less viscous and it cools down at a slower rate, facilitating the deformation of the plastic green wire due to its own weight. When the feedstock with ϕ = 0.925 was used, the extruded green body is stiffer, it cools down rapidly, and the wire shows little deformation.

It must also be observed that the feedstock with higher powder loading (ϕ = 0.925) leads to a small variation of D/D_n with T_e for both nozzles. The shrinkage of this feedstock is very little and constant throughout the whole investigated experimental field. This result is of great practical technological importance, especially for 3D printing applications. In fact, lower binder content not only obviously facilitates the debinding and sintering operation and leads to greater sintered density of parts, but it is also clearly beneficial for the predictability of the extrusion process. If the D/D_n ratio is close to unity for a wide range of temperature and for any die dimension, as a consequence the extrusion process will be more predictable and stable to minor temporary variations of process conditions. The superiority of the ϕ = 0.925 feedstock is confirmed by the statistical analysis on the within-wire standard deviation of diameter (σ_D). ϕ is the only statistically significant factor, since σ_D = 0.141 when ϕ = 0.925, while it increases to σ_D = 0.223 when ϕ = 0.875. Most results presented here can be correlated to the expected viscosity of the mixture, as later discussed in Section 3.4.

3.3 Extrusion and deposition on moving table

In the EAM process, the actual deposited shape of the filament does not depend on gravity, because the distance between the nozzle and the table is very small. However, in EAM each new deposited filament does not remain cylindrical, because it is squeezed onto the previous layer or directly onto the table. Table deposition tests have been conducted in order to remove both the effect of gravity and the effect of squeezing from the results. Extrusion velocity v_e and table velocity v_t are related with each other from critical nozzle height (h_c), as in equation (4) [33]. The critical nozzle height is the distance between the nozzle and the table below which the extruded filament is significantly squeezed between the nozzle and the table which, therefore changing its cross section from round into approximately rectangular with rounded edges. In the present study, the selected levels of nozzle diameter D_n, extrusion velocity (v_e) and table velocity (v_t) are such that the critical nozzle height (h_c) varies from 0.6 to 1.2.

$$h_c = \frac{0.785\, v_e\, D_n}{v_t} \quad \text{(4)}$$

The deposition tests should be run with variable height, according to (Equation (4)), but the actual nozzle height was kept constant at 5 mm, about 5 times larger than the maximum critical value of 1.2 mm. This is to study the swelling/shrinking phenomenon only as a function of the velocities, avoiding shrinkage due to gravity and deformation due to a short nozzle-table distance (squeezing effect). As a consequence, the final filament shape will be nearly cylindrical, and its average diameter will be easily measured.

In the plan of experiments, 3 levels of v_t/v_e ratios have been tested. Figure 7 shows the effect of the two most influencing factors on the wire diameter, while depositing on a moving table: the v_t/v_e ratio and the extrusion temperature T_e. Mean diameter of extruded wire increases with increase in extrusion temperature from 110ºC to 140ºC. While in vertical extrusion tests most average values where below 1, i.e. indicated shrinking, here some swelling has been observed in many cases. In this case the wire bends on the deposition table, and this reduces the gravity-induced shrinkage. The friction between the wire and the table determines whether the deposited wire is pulled (and shrinks) or not (and does not shrink or even swells).

The results of the experiments, modeled through a General Linear Model, have a standard error of 0.033 mm, less than half than in vertical extrusion tests. This means that gravity increases the variability and reduces the predictability of the results.

As seen from Figure 7 (a), when the table velocity is high (e.g., $v_t/v_e = 1.25$), the filament is pulled by the friction between the deposited wire and the table. In this case, the contact angle between wire and table is smaller than 90°. When $v_t/v_e = 1$, the contact angle becomes close to 90°, suggesting that the wire solidifies with the same diameter of that extruded. At lower table velocity ($v_t/v_e = 0.755$), the filament is compressed onto the table and starts swelling. Unlike previous investigations, that had shown little influence of the table velocity on D/D_n [34], in the present study the interaction both T_e and v_t/v_e have a significant effect on the dimensions of the extruded wires. From a technological point of view, if the goal of reaching a solid wire diameter ratio close to 1 must be reached, the table velocity ratio should be increased from 0.755 to 1.25 as temperature increases from 110 to 140 °C.

The statistical analysis has shown that there is some interaction, i.e. combined effect of the solid loading percentage and the nozzle diameter on the swelling/shrinkage. This interaction is shown in Figure 7 (b). The most remarkable observation is that, as already demonstrated in the vertical extrusion tests, the swell/shrinkage of wires with $\phi = 0.925$ (corresponding to a polymer binder volume content of only 38 %) is substantially negligible with both nozzles. In fact, with large solid loading (towards $\phi = 0.925$), more powder grains are oriented in the flow direction, their packing states altered to loosen them to improve the distribution of binder for steady flow, due to the shear action during flow through the nozzle [35]. As in vertical extrusion tests, the smaller diameter nozzle outperforms the larger one, since it is closer to the acceptable D/D_n range for all ϕ-values.

A general linear model has been run also using the within-wire standard deviation of D_n (σ_D) as the response variable (Figure 8). As in vertical extrusion tests, an important parameter is the solid loading ϕ, but the standard deviation is also influenced by the v_t/v_e ratio, which was not present as a factor in previous tests (vertical extrusion experiments).

The effect of v_t/v_e is clear: the wires are less uniform when the table moves faster ($v_t/v_e = 1.25$). This is because the frictional stress that pulls the wire increases with the deposited length and induces diameter variations. The within-wire variation of D/D_n, i.e. the process stability, also depends on the powder content; once again, the most stable behavior is exhibited by the mixture with $\phi = 0.925$. Its high viscosity and large metal fraction makes it more resistant do deformation induced by the table.

To further demonstrate the important role of the solid loading ϕ, the radial distribution of powder in wire by different powder loading has been observed with the SEM. Cross-section of several (as deposited/green) wires were observed and representative micrographs are presented in Figure 9. It can be seen from the SEM micrographs that with the increase of powder content, powder distribution is more uniform throughout the wire and wire surface became smooth, resulting in an isotropic structure. In Figure 10 (a), magnified microstructure of as deposited wire shows the uniform distribution of powder in binder (similar to that present in as prepared feedstock of SS316L powder). The proposed extrusion and deposition system is capable of producing a stable and controllable flow of the filaments, which can be deposited in stratified layers to form a 3D build structure. A representative picture of 3D fabricated parts is also shown in Figure 10 (b). Extruded wires are uniformly deposited in each layer and adhered to previous layers to form a 3D structure.

3.4 Discussion

The General Linear Models used in Section 3.3 for the analysis of the vertical extrusion and deposition tests have been developed using the technological extrusion parameters: nozzle diameter, extrusion temperature, etc. However, according to equation (1), each experimental condition will generate a different state of shear strain, viscosity and shear stress on the material.

The expected viscosity of each experimental condition can be estimated and plotted vs. the standard deviation σ_d of each combination. The result is in Figure 11: there is an obvious large dispersions of results (scatter), but the negative linear correlation (in a log-log scale) of the standard deviation of the wire diameter with the apparent viscosity of the mixtures is statistically significant and evident from the plots. It can be concluded that in vertical extrusion, highly viscous feedstocks are preferable. In table deposition tests (Figure 11 (b)), table velocity is controlling parameter in v_t/v_e. Thus v_t/v_e is marginally interacting with viscosity in determining variation of diameter of extruded wire.

The pseudoplastic behavior is likely to have viscoelastic effects, i.e., partial recovery of elastic deformation that a viscoelastic fluid undergoes during the capillary flow of nozzle [36]. In both vertical extrusion and table deposition tests, a little swelling has been observed, more often shrinkage and elongation of wire was the dominant phenomenon. In fact, swelling is challenging to be recorded experimentally due to a limited period of its existence, as illustrated schematically in Figure 12.

At a constant extrusion velocity, any change of the diameter after swelling depends on gravity in case of vertical extrusion and on table velocity in case of deposition. The extruded wire leaving the nozzle, first swells as shown in Figure 12. At low table velocity or during the start of vertical test due to lower gravity pull, the wire swells considerably higher as shown in Figure 12 (a). The diameter of becomes smaller gradually when table velocity or gravity pull increases Figure 12 (b) and (c).

The existence of swelling is significant in case of (a) feedstock having low solid loading because of the dominance of viscoelastic behavior of binder and (b) higher shear rate corresponding to higher extrusion velocity (v_e).

4. Conclusions

Experimental investigations of extruded SS316L feedstock wires have been used for correlating flow behavior and target extrusion dimensions with feedstock and process characteristics. As follows, few concise interpretations of the study are reported. Dimensional variations in vertical extrusions are more severe than deposition on the table because part elongates before solidification with increasing self-weight and viscoelastic changes. Change of powder loading and extrusion temperature alters flow behavior, and rheological effects are counted in dimensional variations. From the viewpoint of accurate dimensional control, higher solid loading ($\phi \sim 0.925$ / 62 vol. %) and lower extrusion temperature ($T_e \sim 115^oC$) are favorable. Selected deposition parameters affect the dimensional variation of wire in connection with nozzle height. Increasing extrusion velocity and table velocity beyond 20 mm/s tends to stretch the wire due to plastic strain and shows considerably low D/D_n. However if maintained between 10 mm/s to 20 mm/s, the extruded diameter is equal to nozzle diameter. If managing the above range is not feasible for a specific application, feedstock flow can be controlled through relative adjustment between these parameters.

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