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Role of elongational viscosity of feedstock in extrusion-based additive manufacturing of powder-binder mixtures

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Abstract

The 3D printing of metals and ceramics by the extrusion of a powder/thermoplastic binder feedstock is an extrusion-based additive manufacturing (EAM) technique and has received significant interest. EAM feedstocks are generally characterized by their shear viscosity. A quantitative comparison with the shear flow data, through an estimation of the Trouton ratio, indicates that the extensional viscosities are three orders of magnitude greater than their shear flow viscosity at a comparable shear rate obtained in three different high loaded polymers retained for this study. This experimental study addresses the unsolved issue of the role of elongational viscosity in the modelling of EAM of highly viscous melts. The study was conducted using three feedstocks with a water-soluble binder and high powder loading. The different powder materials used for this study are stainless steel, alumina and zirconia. Initially, the rheological properties of the feedstocks were assessed using capillary rheometers. A pressure drop model based on the shear and elongational components of the viscosity was proposed to predict the extrusion pressure during capillary tests. The model was adapted to develop a specific EAM machine, namely, an EFeSTO, equipped with a pellet extrusion unit. Experimental EAM tests were conducted, and the pressure drops were analytically predicted and experimentally measured. A total of 31 different combinations of extrusion velocities, nozzle diameters, 3D printed shapes and materials were tested through a total 184 experimental runs. The model predicts well the experimental pressures for the steel feedstock, whereas it underestimates the pressure for the two ceramic feedstocks owing to their different thermal properties. The results of this study clearly demonstrate that the pressure, and therefore the material flow during the EAM processes of viscous materials, cannot be modelled well without considering the elongational viscosity.

Keywords 3D printing · Highly viscous melt · Extrusion pressure · Elongational viscosity

1 Introduction

Powder injection moulding (PIM) is a convenient widespread manufacturing process for producing complex components in large batches [1]. It employs a feedstock usually composed of a thermoplastic polymeric binder, filled with metals or a ceramic powder. This type of feedstock can also be used for extrusion-based additive manufacturing (EAM) technologies for metallic and ceramic components [2]. There are two types

of EAM technologies. One is called direct ink writing (DIW), which has also been called robocasting, based on the direct use of a powder-binder/matrix slurry feedstock (without increasing the temperature for melting) [3]. The other is called fused filament deposition (FFD). It is a 3D printing process or additive manufacturing technique that applies a continuous filament. A filament-type thermoplastic polymer is melted before it extrudes from the nozzle and is deposited on the growing specimen. The headed printer extruder heat usually moves in two dimensions to deposit one horizontal layer at a time. The specimen or printer extruder head is then moved vertically by a small amount to begin a new layer. To realize a 3D component with a functional material, Nadernezhad et al. [4] investigated the extrusion of PLA/CNT nanocomposites dedicated to additive manufacturing using this FFD process. In our case, the FFD process has been modified for application with pellets instead of a filament using the EAM of powder-binder mixtures.

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56 Some EAM machines for the processing of such feedstocks
 57 are commercially available (including Markforged Metal X
 58 and Desktop Metal Studio); however, no commercial ma-
 59 chines are yet available for extrusion starting from the pellets
 60 of feedstock, instead of filaments or rods. EAM machines
 61 based on the extrusion of pellets allow for material diversity
 62 and are cost-effective.

63 The EFeSTO machine has been previously developed and
 64 was employed in this study. It combines a servo-controlled
 65 small pellet extruder unit with a robotic deposition table based
 66 on parallel kinematics [5]. One advantage of EFeSTO for the
 67 present study is that the torque (and therefore the pressure)
 68 applied by the pellet extrusion unit can be monitored during
 69 the processing.

70 Melt viscosity [6] is one of the important characteristics of
 71 a feedstock and is used to predict the rheological behaviour
 72 during highly viscous melt extrusion and in the correct design
 73 of a 3D printing process through the selection of appropriate
 74 extrusion parameters.

75 In previous studies associated with powder injection
 76 moulding, the rheology of highly loaded feedstocks has gen-
 77 erally been assessed through a capillary rheometer, used to
 78 characterize the shear viscosity behaviour [7]. A literature
 79 review dedicated to the laws of highly concentrated feedstock
 80 alloys is available in [8], where a shear viscosity model was
 81 proposed for superalloy powders. A capillary rheometer is
 82 generally preferred over other rheometers to reduce the esti-
 83 mation errors from a wall slip [9]. The shear viscosity is uni-
 84 versally and correctly considered the most important param-
 85 eter for highly viscous PIM feedstocks. However, during the
 86 EAM processes, the shear rates are comparably smaller, and
 87 the extrusion nozzles are shorter; therefore, compared with
 88 PIM, EAM processes induce a comparably lower amount of
 89 shear deformation, whereas the extruded filaments inevitably
 90 elongate. In a review on the EAM processes [10], the exten-
 91 sional viscosity was not mentioned. In a more recent review
 92 [11], the author recognized that elongational viscosity is gen-
 93 erally accepted as an important parameter for determining the
 94 pressure drops in additive manufacturing through a material
 95 extrusion. However, despite this common belief, the charac-
 96 terization of the elongational viscosity in scientific papers
 97 dealing with the EAM processes has generally been neglected.
 98 In [12], the authors list all of the relevant feedstock properties
 99 for the EAM of metals, and place a large emphasis on the
 100 shear viscosity, while neglecting to mention the elongational
 101 component. The shear viscosity is still frequently considered a
 102 unique or important property of highly viscous EAM feed-
 103 stocks, such as in [13], where the authors studied the EAM
 104 of zirconia, or in [14], where the authors studied the effects of
 105 the powder size on the properties of highly filled polymers for
 106 fused filament deposition (FFD). In [15], the authors charac-
 107 terized the viscosity of highly viscous polymers for FFD and
 108 recognized the importance of the material at extremely small

or “zero” shear rates; nevertheless, they modelled and repre-
 109 sented the shear viscosity only, and not the elongational
 110 viscosity. 111

112 One of the reasons why the elongational or entrance vis-
 113 cosity of viscous non-Newtonian fluids during the EAM pro-
 114 cesses has been neglected by the scientific literature is the
 115 inherent difficulty of knowing the instantaneous extrusion
 116 pressure during such processes or, even worse, the instanta-
 117 neous shear stress. In typical FFD machines, the instantaneous
 118 extrusion pressure is unknown. As an exception, in [16] the
 119 authors conducted a very interesting study using in-line rheo-
 120 logical pressure measurements in FFD. However, they did not
 121 characterize or isolate the extensional viscosity.

122 The purpose of the present study is to demonstrate that, for
 123 EAM with specific viscous melts, which take place at a low
 124 shear rate and within relatively short extrusion nozzles, the
 125 characterization of the feedstock based on the elongational
 126 viscosity is more important than the shear viscosity when
 127 predicting the flow.

128 The remainder of this paper is organized as follows: In the
 129 next section, the relevant rheological models are presented,
 130 highlighting the differences between the shear and
 131 elongational viscosity. The experimental materials, methods
 132 and equipment are then described. In the third section, the
 133 rheological model is validated based on capillary rheometer
 134 data. Finally, the results of extrusion and 3D printing tests
 135 using EFeSTO equipment are presented and discussed.

2 Rheological models 136

137 The rheology of powder-binder feedstocks has been exten-
 138 sively studied, and many models have been proposed to de-
 139 scribe the melt viscosity during the extrusion and injection
 140 moulding processes. The well-known constitutive equation
 141 for the shear viscosity of Newtonian fluids is as follows:

$$\eta_s = \frac{\tau}{\dot{\gamma}} \quad (1)$$

142 where τ is the shear stress and $\dot{\gamma}$ is the applied shear rate.
 143 In addition, η_s is the shear viscosity or the resistance of
 144 the fluid to shearing. The shear viscosity is a constant for
 145 Newtonian fluids, whereas the powder-binder feedstocks
 146 usually show a non-Newtonian characteristic [17]. In PIM
 147 applications, a shear-thinning (or pseudoplastic) effect is
 148 observed, where the shear viscosity decreases upon an
 149 increase in the shear rate [18]. The simplest way to de-
 150 scribe a pseudoplastic effect is the power law model,
 151 which demonstrates a non-linear relation between the
 152 shear stress and shear rate as follows: 153

$$\tau = K\dot{\gamma}^n \quad (2)$$

154 where K and n are material-specific parameters, namely, 204
 156 the consistency and shear rate sensitivity, respectively. 205
 157 The shear rate sensitivity n is the power law index, which 206
 158 is $n < 1$ for pseudoplastic fluids; shear thinning then be- 207
 159 comes more evident with a decrease in n . In a previous 208
 160 study [6], it was demonstrated that larger K values favour 209
 161 a better stability of the extrusion of the metal-binder feed- 210
 162 stocks, in terms of both the pressure signal and filament 211
 163 quality. 212

164 Experimental measurements of the shear viscosity can be 213
 165 conducted using a variety of instruments, given the wide range 214
 166 of viscosities that feedstock materials can present [19]. The 215
 167 most common are capillary rheometers, which can be used 216
 168 from 2 to 3000 s⁻¹ [20]. For a capillary rheometer, pressure 217
 169 is applied using a piston, and the apparent shear rate ($\dot{\gamma}_a$) and 218
 170 shear stress at the wall (τ_w) are determined from the extruded 219
 171 flow rate for non-Newtonian fluids: 220

174
$$\tau_w = \frac{\Delta P_{cap}}{L/2R} \quad (3)$$

173
$$\dot{\gamma}_a = \frac{4Q}{\pi R^3} \quad (4)$$

176 where ΔP_{cap} is the pressure drop at the capillary, L is the 217
 177 capillary length, R is the radius, Q is the volumetric flow rate 218
 178 and $\dot{\gamma}_a$ is the apparent shear rate, i.e. the true shear rate of a 219
 179 Newtonian fluid. For shear-thinning fluids, Rabinowitsch cor- 220
 180 rection for determining a more realistic value of the true shear 221
 181 rate $\dot{\gamma}_w$ must be employed [21]: 222

184
$$\dot{\gamma}_w = \frac{(3n + 1) 4Q}{4n\pi R^3} \quad (5)$$

183 Nozzles used in EAM machines for highly viscous poly- 223
 186 mers [2] are generally extremely short, with length over diam- 224
 187 eter (L/D) ratios of well below 10. For short capillaries ($L/D <$ 225
 188 25), an additional pressure drop ΔP_e at the entrance must be 226
 189 accounted for owing to the sharp decrease in diameter from 227
 190 the barrel where the material is compressed before entering the 228
 191 capillary. Bagley's correction is often used for this purpose 229
 192 [17]: 230

195
$$n_B = \frac{\Delta P_e}{2\tau_w} \quad (6)$$

196 Bagley's corrected shear stress at the wall can be calculated 231
 197 as follows: 232

200
$$\tau_w = \frac{(\Delta P_{cap} + \Delta P_e)}{2(L/R + n_B)} \quad (7)$$

201 Bagley's correction depends on both the geometry of the 233
 202 capillary and the material characteristics. The role of the en- 234
 203 trance pressure drop in the short capillaries has been 235

204 considered by many authors to be related to the so-called 205
 206 elongational or extensional viscosity [22]. Indeed, for a deeper 207
 208 understanding of the rheological of the feedstock during the 209
 209 extrusion process of EAM, the contributions of the shear vis- 210
 210 cosity and the elongational viscosity need to be explicitly 211
 211 quantified. Numerous models have been proposed to describe 212
 212 the elongational viscosity of polymer melts, e.g. using a flow 213
 213 through a tube with an abrupt contraction as a measure [23]. 214
 214 For elongational rheometry experiments of non-Newtonian 215
 215 fluids, the elastic and viscous contributions can be separated 216
 216 [24]. When characterizing highly viscous materials, the roles 217
 217 of the capillarity and gravity are generally neglected. 218

219 One of the most accredited models for estimating the en- 220
 220 trance pressure drop (ΔP_e) was developed by Cogswell [25], 221
 221 who assumed that the pressure drop can be modelled by de- 222
 222 fining the shear viscosity (η_s) and elongational viscosity (η_E) 223
 223 dependent terms [26]. This model is only accurate at low 224
 224 deformation rates (as in EAM applications). As an alternative 225
 225 to Cogswell's model, Binding and Gibson's model [27] can 226
 226 also be used to accurately describe the pseudoplastic effect of 227
 227 PIM feedstock over a wide range of shear rates when consid- 228
 228 ering the contributions of the shear and elongational viscosity. 229
 229 A simple rheological model, comparable with Binding and 230
 230 Gibson's model, is proposed herein to analyse the results of 231
 231 twin-bore capillary rheometers. 232

2.1 Rheology of feedstock: shear and elongational viscosities

233 Polymer processing through the mixing and printing of a high 234
 234 loaded polymer usually involves medium and large strain rates 235
 235 in shear and extensional flows, and the viscosity of the feed- 236
 236 stock depends on the binder composition and properties of the 237
 237 powders, mixing parameters and conditions. In the case of a 238
 238 high loaded polymer, Arabo [28] concluded that an extensional 239
 239 (or elongational) flow is important and has therefore 240
 240 attracted significant interest in the powder forming processes. 241

242 In a twin-bore rheometer, there are two nozzles: The nozzle 243
 243 on the left is a long capillary ($L/D \gg 10$) and the nozzle on the 244
 244 right has a negligible length, i.e. virtually a "zero shear" de- 245
 245 formation. A simple model has been developed based on the 246
 246 data obtained from a twin-bore capillary rheometer. This mod- 247
 247 el was then validated, as shown later in this paper, on a differ- 248
 248 ent twin-bore rheometer with a different L/D ratio. The model 249
 249 assumes that the total pressure at a long (left) capillary is 250
 250 considered the sum of two components as follows: 251

252
$$P_{tot} = \Delta P_{left} = \Delta P_{ent} + \Delta P_{cap}, \quad (8)$$

253 where ΔP_{ent} is the entrance pressure variation owing to the 254
 254 abrupt change in section between the barrel and capillary. 255
 255 This ΔP_{ent} value can therefore be directly measured from 256
 256 the right bore, which is associated with the calculation of 257

253 the elongational viscosity (η_E) and elongational strain rate
 254 $\dot{\epsilon}$. The second term ΔP_{cap} is associated with the shear
 255 deformation and shear viscosity, which can be calculated
 256 at the long (left) bore after subtracting ΔP_{ent} . True correc-
 257 tions have been applied to the capillary rheometer data to
 258 more accurately describe the non-Newtonian behaviour of
 259 the feedstock, namely, the above-mentioned Bagley's and
 260 Rabinowitsch corrections. Moreover, owing to the high
 261 viscosity of the powder-binder feedstock, the assumption
 262 of no wall slipping, typical of capillary rheology, is re-
 263 moved. Therefore, the apparent shear rate was corrected
 264 as follows:

$$\dot{\gamma} = \dot{\gamma}_a - \dot{\gamma}_0, \quad (9)$$

266 where $\dot{\gamma}_0$ is an experimental constant determined by the
 267 least squares minimisation; this expresses the shear rate
 268 reduction owing to a wall slip. The shear viscosity (η_s) is
 269 modelled using a power law equation as a function of the
 270 corrected shear strain:

$$\eta_s = K \dot{\gamma}^{n-1}. \quad (10)$$

273
 274 The right capillary provides a negligible shear resistance,
 275 and therefore its pressure reading, the major cause of which is
 276 the entrance pressure, can be entirely associated with the
 277 elongational viscosity (η_E):

$$\eta_E = \frac{\sigma_E}{\dot{\epsilon}}, \quad (11)$$

278 where σ_E is the elongational stress at the orifice and $\dot{\epsilon}$ is the
 280 elongational strain rate. The elongational viscosity can also be
 281 modelled using a power law equation as a function of the
 282 apparent shear rate:

$$\eta_E = l \dot{\gamma}_a^{y-1}, \quad (12)$$

284 where l and y are the consistency and sensitivity parameters
 285 associated with the elongational viscosity. The elongational
 286 strain rate is independent of the capillary length, although
 287 the capillary diameter does have an influence. For a given
 288 apparent shear rate $\dot{\gamma}_a$, the elongational strain rate can be esti-
 289 mated based on the following:

$$\dot{\epsilon} = \frac{\dot{\gamma}_a}{4}. \quad (13)$$

292
 293 Under Cogswell's model assumptions, the elongational
 294 stress can be calculated as a function of the entrance pressure
 295 drop as follows:

$$\sigma_E = \frac{3}{8}(n+1)\Delta P_{ent}. \quad (14)$$

298 After substituting the terms η_s , η_E , $\dot{\gamma}$, $\dot{\epsilon}$ and σ_E in Eq. (8),
 299 the total pressure drop in the left capillary can be finally
 300 expressed in the following way: 301

$$\begin{aligned} P_{tot} &= \Delta P_{cap} + \Delta P_{ent} = \eta_s \dot{\gamma} \frac{4L_l}{D} + \eta_E \dot{\gamma}_a \frac{2}{3(n+1)} \\ &= K \dot{\gamma}^n \frac{4L_l}{D} + l \dot{\gamma}_a^y \frac{2}{3(n+1)} \end{aligned} \quad (15)$$

305 3 Materials, equipment and methods

306 3.1 Feedstock characterization

307 Three different feedstocks were used for this study. A feed-
 308 stock with a solid loading of stainless steel (SS 316L) powder
 309 was prepared by mixing a water-soluble Embemould K83
 310 binder (eMBE, GmbH) and gas-atomised (SS 316L) powder
 311 (Sandvik Osprey) in a Brabender Plasti-Corder mixer. Parenti
 312 et al. [29] used the same binder for thermoplastic processing,
 313 applying a combination based on polymers with water-soluble
 314 components. The binder is specifically devoted to aqueous de-
 315 binding for the PIM process. After a DSC analysis, they con-
 316 cluded that it is multi-constituent with three different ingredi-
 317 ents and that the highest associated melt temperature is ap-
 318 proximately 118 °C. The density of the water-soluble material
 319 K83 is 1.05 g/cm³. Mixing of the K83 binder and powder was
 320 performed at 145 °C for 30 min to produce a homogeneous
 321 feedstock without introducing air bubbles.

322 This feedstock mixture was further processed through a
 323 twin-screw extruder at 145 °C to obtain a highly homoge-
 324 neous and pelletised feedstock for the subsequent operations.
 325 Two commercial (INMATEC, GmbH) ceramic feedstocks,
 326 having a solid loading of alumina-based ceramic powder
 327 (INMAFEED K1008) and zirconia-based ceramic powder
 328 (INMAFEED K1009), were procured. The chemical compo-
 329 sition of the stainless steel, alumina and zirconia powders is
 330 shown in Table 1.

331 In Table 2, the relevant physical and thermal properties of
 332 the investigated feedstocks are shown. Physical and thermal
 333 properties of the feedstock play an important role in the sta-
 334 bility and phase change during extrusion and 3D printing.

335 All powders used in the present study are fine powders with
 336 d_{50} of less than 10 µm, allowing components with a fine
 337 microstructure and smooth surface to be produced through
 338 the EAM process.

339 The volumetric powder loading φ of the two types of com-
 340 mercial ceramic feedstock was clearly selected by the produc-
 341 er. The powder loading of the steel feedstock with the best
 342 value for extrudability was selected according to a previous
 343 study [6].

t1.1 **Table 1** Chemical composition (by wt%) of powders used in the present study

t1.2 Element	Cr	Ni	Mo	Mn	Si	C	P	S	Fe
t1.3 SS 316L steel	17.90	11.70	2.30	1.41	0.72	0.02	0.02	0.006	65.7
t1.4 Compound	Na ₂ O	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	ZrO ₂	Y ₂ O ₃	MgO	CaO	
t1.5 Alumina	0.1	0.03	1.8	96	-	-	0.9	1.3	
t1.6 Zirconia	0.04	0.01%	0.02	0.25	94.5	5.15	-	-	

344 The thermal conductivity (*k*) of the feedstock is non-
 345 proportional to the solid content (i.e. weight of the powder
 346 in the feedstock) because the heat flow is limited by the binder
 347 system, with a continuous matrix forming a layer between
 348 particles. The thermal conductivity *k* of each feedstock was
 349 calculated using an equation provided by Lobo and Cohen
 350 [30]:

$$\frac{1}{k} = \frac{1-\varphi}{k_b} + \frac{\varphi}{k_p}, \quad (16)$$

352 where φ is the volumetric powder loading and k_b and k_p are
 353 the nominal thermal conductivities of the binder and powder,
 354 respectively.

355 The heat capacity C_p of the feedstock is calculated up to the
 356 suggested operational temperature (145 °C for alumina,
 357 175 °C for zirconia and 130 °C for stainless steel), through
 358 an analysis of the DSC curve [31]. The three powders differ
 359 considerably in terms of the heat capacity, the estimate of
 360 which is also provided in Table 2. The values were measured
 361 based on differential scanning calorimetry (DSC) tests, con-
 362 ducted under the ASTM D3418-15 standard, using a differen-
 363 tial scanning calorimeter (DSC2010, TA Instruments). The
 364 pan was aluminium, and the test was performed in a nitrogen
 365 atmosphere, with a gas flow rate of 40 ml/min. The applied
 366 heating rate corresponded to 5 °C/min. The DSC curves of the
 367 feedstocks are plotted in Fig. 1, which show a comparison
 368 between the curves of the metal and the ceramic feedstock.
 369 Both ceramic feedstocks present a peak at the same tempera-
 370 ture of 60.8 °C corresponding to the melting point of the
 371 industrial binder. However, the stainless steel feedstock pre-
 372 sents a single fusion peak at 62.9 °C with a latent heat of
 373 fusion of 26.1 J/g. The solidification temperature is observed
 374 to be 38.0 °C. The DSC curve also shows indistinct peaks at

64.4 °C, 96.9 °C, 110.5 °C and 158.3 °C, corresponding to the
 melting and solidification of different components (PEG,
 PMMA, surfactants and additives, respectively) in an
 Embemould K83 binder.

Once the properties ρ , C_p and k are known, the diffusivity α
 can be calculated as a derived variable. Table 2 shows that the
 thermal diffusivity α of zirconia is the largest (owing to its low
 heat capacity), followed by alumina, whereas the diffusivity of
 the steel feedstock is significantly smaller (owing to its higher
 density).

3.2 Capillary rheometers

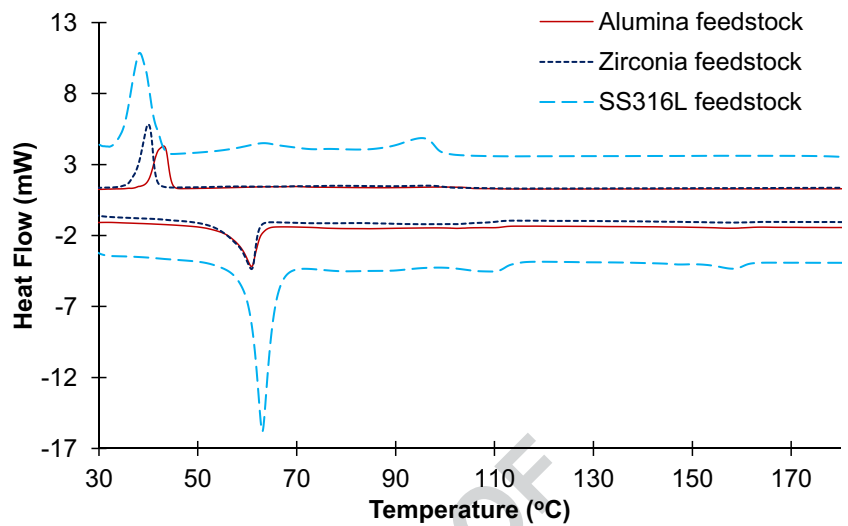
The rheological properties of all feedstocks were deter-
 mined using two different twin-bore capillary rheometers
 (Fig. 2a), labelled as rheometers A and B (Malvern
 Panalytical). The selected test temperatures were 145 °C
 for alumina, 175 °C for zirconia and 130 °C for stainless
 steel, over a wide range (50 to 1000 s⁻¹) of shear rates. A
 sample of each material was positioned in a cylindrical
 barrel with moving pistons. Defining the piston speed,
 the material is forced into a long capillary of known di-
 ameter D_l and length L_l at the bottom left of the barrel and
 into an extremely short capillary with D_r and L_r on the
 right. The values of D_r and L_r for both rheometers are
 given in Fig. 2c and used for the calculations described in
 Sect. 4.1 for validation. Pressure transducers are placed
 immediately above the capillaries; the output of this test is
 therefore the pressure from each bore. This setup allows
 the determination of the shear viscosity from the left capil-
 lary and the elongational viscosity from the right capil-
 lary, according to the model presented above.

t2.1 **Table 2** Physical and thermal
 t2.2 properties of feedstock used in the
 present study

Feedstock	d_{50} (µm)	φ (vol%)	ρ (kg/m ³)	k (W/m K)	C_p (J/kg K)	α (vol%)
Al ₂ O ₃ -binder	1.9	60	2400	0.63	1528	0.17
ZrO ₂ -binder	0.6	47	2550	0.43	794	0.21
SS316L-binder	8.8	62	5320	0.66	1668	0.07

d_{50} is the mean diameter of the powder; φ is the powder loading (vol%) in the feedstock; and ρ , k and C_p are the density, thermal conductivity and heat capacity of the feedstock, respectively

Fig. 1 DSC curves of alumina, zirconia and stainless steel feedstocks



405 **3.3 Description of specific EFeSTO equipment**

406 The EFeSTO machine, shown in Fig. 2b, has been used both
407 for extrusion and 3D printing tests. The work table is free to

move in the *X*-, *Y*- and *Z*-directions and is governed by a 3-axis 408
parallel kinematics linear delta system. The printing head is 409
stationary and composed of a feeder where the pellets of the 410
feedstock are placed, as well as a screw plasticiser and an 411

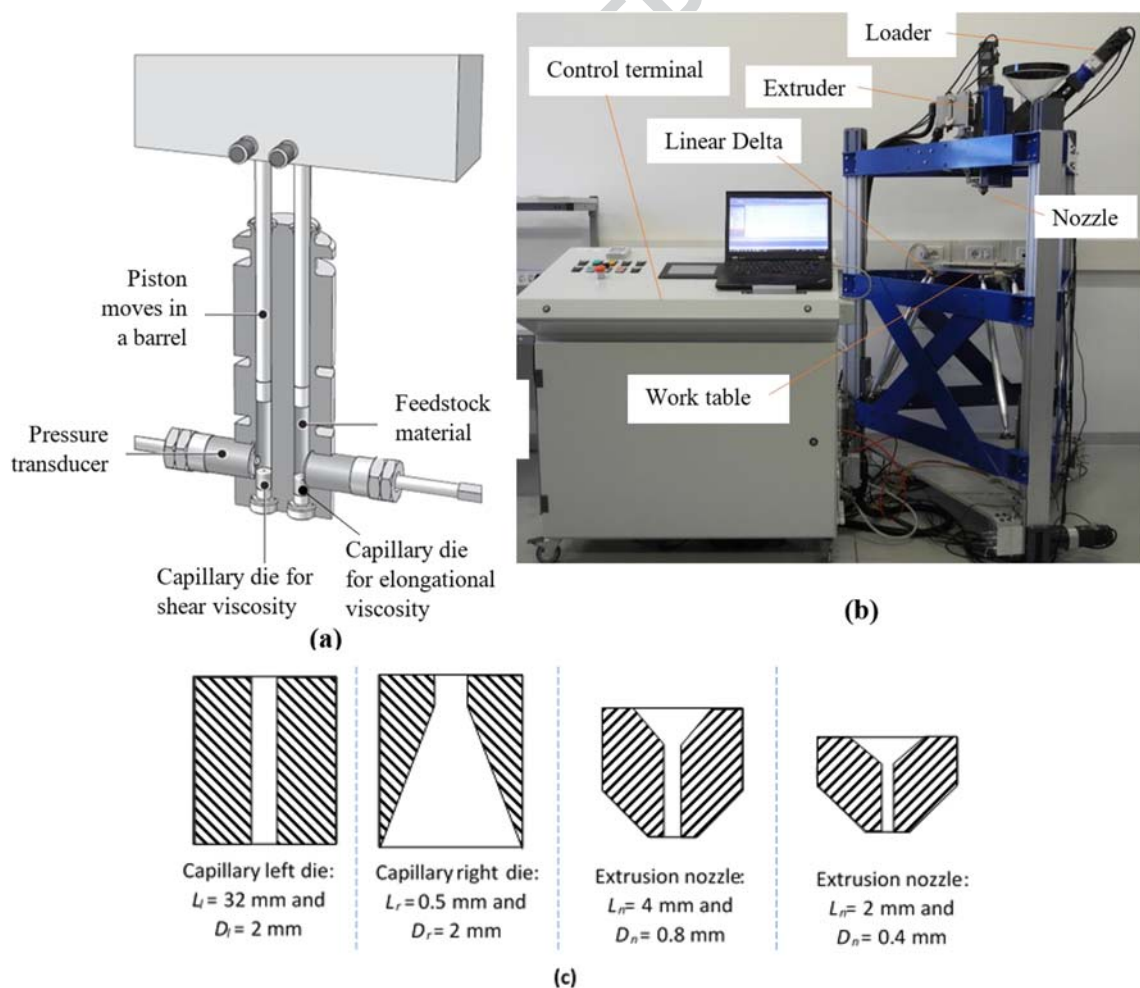


Fig. 2 Main components of a capillary rheometer and b EFeSTO machine and c schematic of die and nozzles

412 injector piston. In the extruder system, the feedstock is
 413 inserted into the feeder and falls into a first loader chamber,
 414 which plasticises the material; it is then injected into a second
 415 extruder chamber, where a CNC piston directly pressurizes the
 416 melt material through the nozzle. For this study, two different
 417 nozzles were employed, with a nozzle diameter (D_n) of 0.4
 418 and 0.8 mm, respectively.

419 Three electric resistors (in the plasticisation chamber, in the
 420 extrusion chamber and at the nozzle) provide heat to the ma-
 421 terial, and four thermocouples provide a temperature control.
 422 Thermal insulation between the high-temperature
 423 plasticisation unit and the actuator unit is achieved using a
 424 water-cooling circuit. The stroke of the extrusion piston is
 425 synchronized with the g-code of the deposition table and
 426 therefore stops during rapid movements of the table, e.g. be-
 427 tween consecutive layers of the 3D printed part.

428 For each experimental run on EFeSTO, the electric current
 429 absorbed by the piston drive was recorded and transformed
 430 into torque M versus the time signal. Data from the extruder
 431 motor was collected using Melsoft MR Configurator software.
 432 The data were stored in a local memory support, and owing to
 433 the length of the operations, a continuous pressure reading
 434 was considered infeasible. Therefore, data were collected dur-
 435 ing intervals of 50 s each at different times throughout the
 436 tests. The sampling frequency was 20 Hz. During the extru-
 437 sion and 3D printing tests, the torque measurements were
 438 conducted more frequently at the beginning and end of the
 439 tests, with 2 min between consecutive readings. In the central
 440 part of each test, the torque was measured with a longer time
 441 between readings: 10 min for extrusion and 5 min for 3D
 442 printing. The torque M versus time signals were then convert-
 443 ed into pressure P_{tot} signals.

444 Among the individual samples of the pressure readings, the
 445 average total pressure P_{tot} was calculated along with the stan-
 446 dard deviation SD_P and coefficient of variation $COV_P = P_{tot}/$
 447 SD_P . As an example, in Fig. 3, P_{tot} is plotted versus time
 448 during a sequence of extrusion and printing tests. The typical
 449 long-run trend of the pressure signal undergoes an initial in-
 450 crease in pressure and stabilization and a marginal increase at

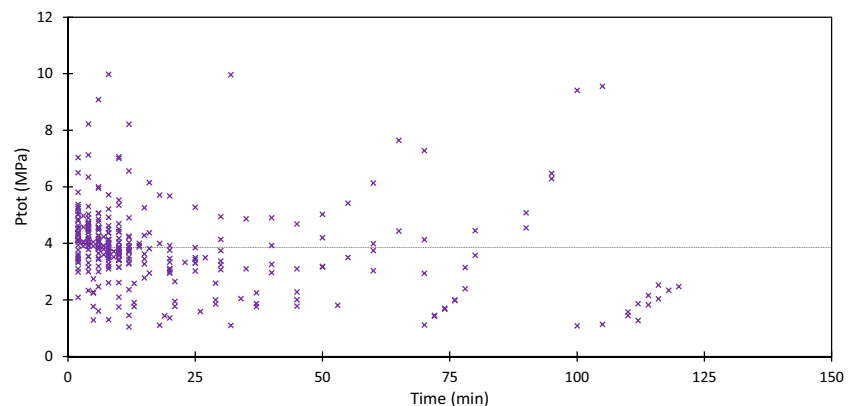
the end of the piston stroke. This is coherent with the flow of
 pseudoplastic fluids: The initial increase corresponds to an
 activation of the flow, and stabilization occurs because of the
 steady-state extrusion regime. The pressure increase at the end
 of the stroke likely occurs because the piston attempts to ex-
 trude the material, which forms a dead zone at the corners of
 the extrusion chamber.

3.4 Experimental plan implemented using EFeSTO

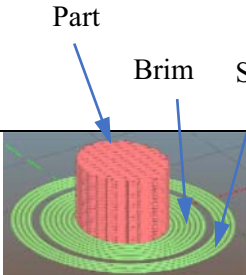
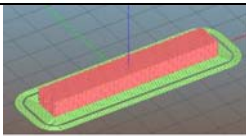
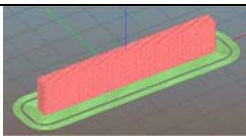
Two main types of tests were conducted: free continuous ex-
 trusion tests and 3D printing tests. During each test, as de-
 scribed before, the average extrusion pressure at piston P_{tot} ,
 as well as its standard deviation SD_P and coefficient of varia-
 tion COV_P , were recorded at regular intervals. During contin-
 uous extrusion, the extrusion piston moves at a constant
 speed, whereas during real 3D printing, it experiences multi-
 ple starts and stops, which might influence the measured
 values of SD_P . For each feedstock, three different shapes were
 3D printed (shown in Table 3): cylinders with a base diameter
 of 10 mm and a height of 10 mm and bars with a rectangular
 cross section with a 6 mm height, 60 mm length and 10 mm
 width. The rectangular bars were printed in both a horizontal
 and vertical configuration, placed on a face with dimensions
 of 60 mm × 10 mm.

The parameter settings used for extrusion and 3D printing
 tests, and designed to produce different apparent shear rates,
 are given in Table 3, namely, two nozzle diameters D_n , three
 extrusion velocities V_e , three materials and four types of test.
 The layer height h does not have an influence on the pressure
 readings and therefore is not listed in Table 3; however, it was
 varied around a centre value of half the nozzle diameter. A full
 factorial experimental plan would have required 72 different
 experimental conditions, plus replicates. Table 3 lists only 31
 out of 72 possible experimental conditions, which were used
 to keep the experimental cost within a reasonable limit. The 31
 tested conditions were replicated a minimum of 2 and a max-
 imum of 5 times, for a total of 184 different tests. Multiple

Fig. 3 Sequence of pressure readings for extrusion pressure (P_{tot}) of stainless steel feedstock: average measured pressure $P_{tot} = 3.86$ MPa with coefficient of variation $COV_P = 8.1\%$



t3.1 **Table 3** Experimental plan for extrusion and 3D printing tests

Shape/Test	Material	Extrusion velocity V_e (mm/s)		
		7.5	12.5	17.5
		D_n (mm)	D_n (mm)	D_n (mm)
 Cylinder	Al ₂ O ₃	0.4	0.4	0.4
	SS 316L	0.4–0.8	0.4–0.8	
	ZrO ₂			
 Rectangular horizontal bar	Al ₂ O ₃	0.4	0.4	0.4
	SS 316L	0.4–0.8	0.8	0.8
	ZrO ₂	0.8	0.8	0.8
 Rectangular vertical bar	Al ₂ O ₃	0.4	0.4	0.4
	SS 316L	0.4		
	ZrO ₂	0.8	0.8	0.8

487 pressure readings were recorded during each test, resulting in
 488 the availability of a very large dataset.

489 **4 Results and discussion of rheological data**

490 The rheological models given in Eqs. (9), (10) and (12) were
 491 applied to the capillary rheometer data (through linear regres-
 492 sion). The corresponding material parameters ($\dot{\gamma}_0$, K , n , l and
 493 γ) are provided in Table 4.

494 The values of consistency l of the elongational viscosity are
 495 three orders of magnitude higher than the shear viscosity

consistency K at a comparable strain rate. This means that
 the ratio of the elongational viscosity function to the shear
 viscosity function is high, corresponding to a Trouton ratio
 of η_E/η_s . This Trouton ratio is approximately 100 in the case
 of 316L feedstock and 200 s^{-1} .

The consistency K of the SS316L steel feedstock is signif-
 icantly smaller than that of the other two materials at lower
 than $1200\text{ Pa}\cdot\text{s}$. Indeed, to verify the flow stability, samples of
 the extruded SS316L feedstock were collected at a shear rate
 of approximately 600 s^{-1} . The surfaces of the rods are shown
 in Fig. 4a and b. For the left (longer) capillary, the quality
 observed at the outer surface of the rod is smooth, whereas

t4.1 **Table 4** Power law parameters and correction shear rate for the three feedstocks

Feedstock	K (Pa.s)	n	l (kPa.s)	γ	$\dot{\gamma}_0$ [s^{-1}]
SS316L	1187	0.678	1530	0.133	16
Alumina	5219	0.279	1086	0.210	1
Zirconia	3622	0.592	6568	0.050	7

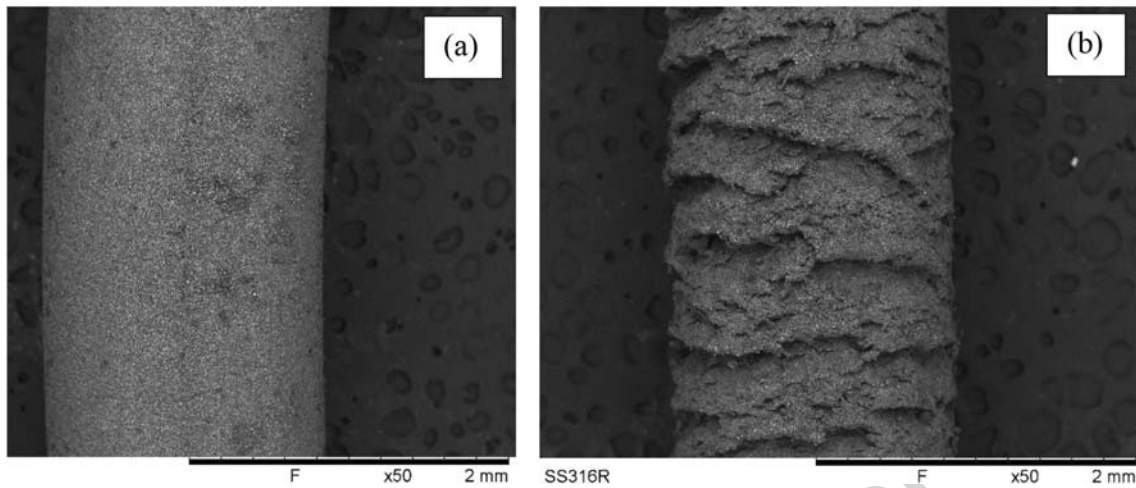


Fig. 4 Extruded roads of SS316L feedstock from **a** left and **b** right capillaries at shear rate of 600 s^{-1}

508 the capillary extruded from the right (shorter) shows a shark-
 509 skin defect. This type of instability can be observed in a poly-
 510 mer extrusion when the capillary/nozzle length is extremely
 511 small ($L \approx 0$) and is connected with a rapid detachment of the
 512 melt flow at the exit of the capillary [32]. This sharkskin
 513 problem has previously been observed for EAM processes
 514 [33].

515 The rheological models can be applied to the conditions of
 516 the planned extrusion and 3D printing tests (given in Table 3),
 517 and the corresponding expected feedstock viscosities at the
 518 nozzle can be estimated. A comparison of the shear and exten-
 519 sional viscosity characteristics of the three high loaded
 520 polymers corresponding to the extrusion and 3D printing test
 521 settings was conducted. In Fig. 5, a comparison between the
 522 shear and elongational viscosity is shown for all studied com-
 523 binations. In the nozzle with $D_n = 0.8 \text{ mm}$, a lower shear rate is
 524 clearly calculated. The shear rate tested with the alumina is
 525 larger owing to its lower n -value, which determines a stronger
 526 Rabinowitsch correction.

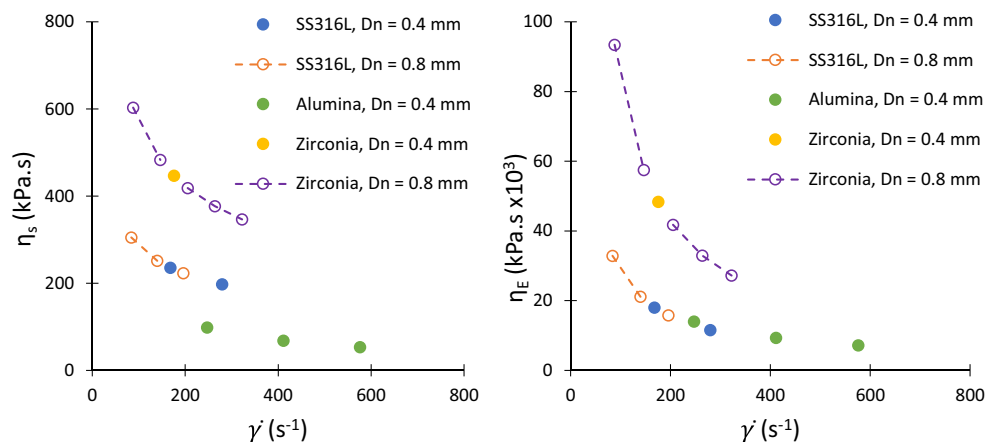
527 The elongational viscosity of the zirconia feedstock is sig-
 528 nificantly larger than that of the other two materials as com-
 529 pared with the shear viscosity, which is due to the larger

elongational consistency l for zirconia of greater than 530
 6500 kPa.s. 531

4.1 Validation of pressure drop model 532

The pressure drop model presented in Eq. (15) can be applied 533
 inversely, and if the viscosity values are known, the total pres- 534
 sure drops can be calculated. The rheological parameters given 535
 in Table 4 for the stainless steel feedstock were obtained 536
 from a Rosand capillary rheometer (rheometer A, $L_1 = 17 \text{ mm}$, 537
 $L_r \approx 0$ and $D_1 = D_r = 1 \text{ mm}$) and were used to predict the total 538
 pressure P_{tot} required when using another rheometer with a 539
 different capillary configuration (rheometer B, $L_1 = 32 \text{ mm}$ 540
 and $D_1 = D_r = 2 \text{ mm}$). In Fig. 6 the predicted pressure is com- 541
 pared with the test with equipment B, showing a good agree- 542
 ment. Rheometer B has a double length in the left capillary, 543
 but with nearly the same L_1/D_1 ratio, and hence the pressure 544
 requirement owing to the shear viscosity is similar. By con- 545
 trast, the right bore of rheometer B has a double diameter and 546
 requires a significantly lower pressure ΔP_{right} owing to the 547
 elongational viscosity. In conclusion, the total pressure re- 548
 quired by rheometer B is up to 40% smaller, primarily because 549

Fig. 5 Shear and elongational viscosity of three feedstocks versus shear rate corresponding to extrusion and 3D printing test settings



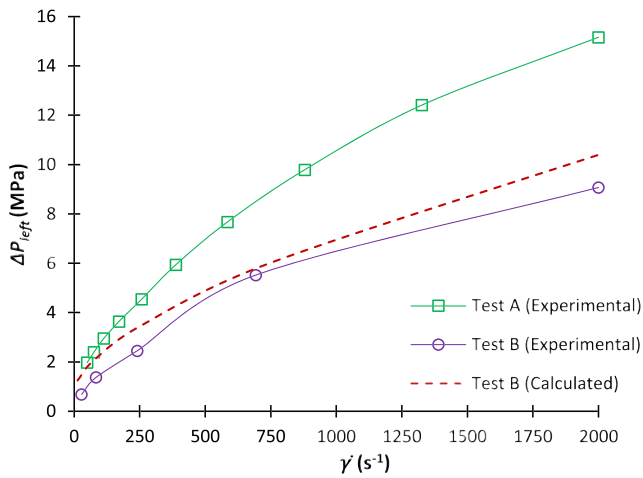


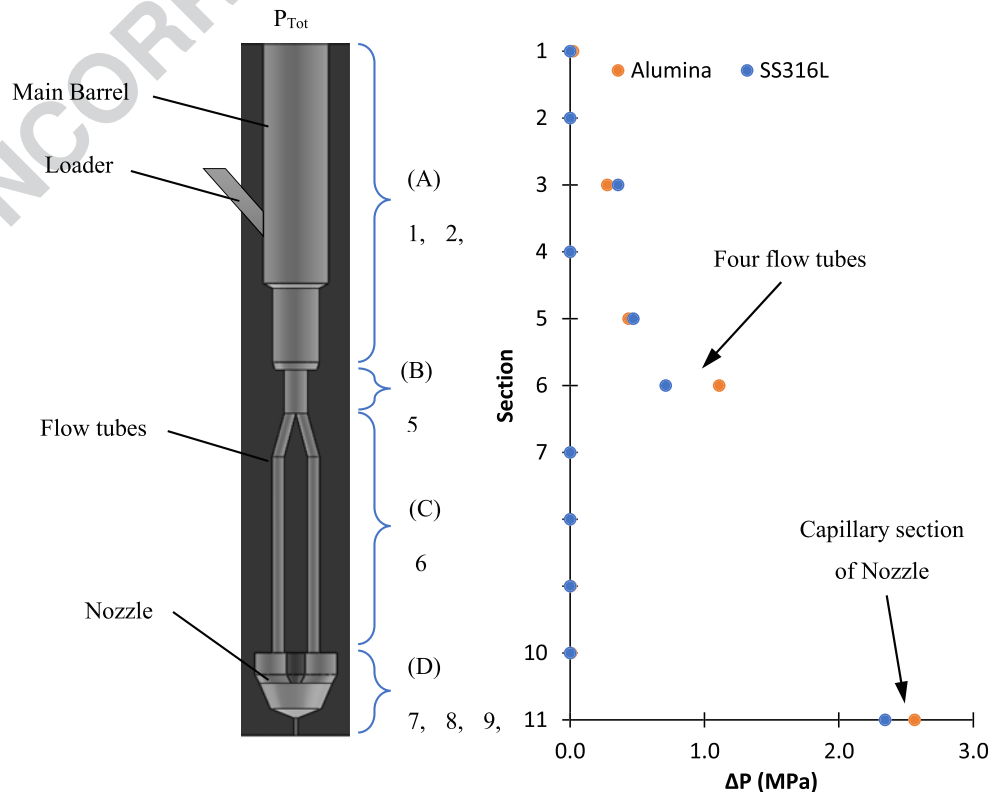
Fig. 6 Experimental and calculated $P_{tot} = \Delta P_{left}$ for different capillary configurations and strain rates

550 of the elongational viscosity. This is an important confirmation
 551 of the important role of the elongational viscosity in the
 552 extrusion of powder-binder feedstocks.

553 **5 Results of extrusion and 3D printing tests**

554 The methodology used for predicting $P_{tot} = \Delta P_{left}$ with Eq.
 555 (15) at the left side capillary rheometer can also be adapted to
 556 predict the total extrusion pressure during extrusion and 3D
 557 printing tests on an EFeSTO machine. The extrusion unit is

Fig. 7 Simplified cross-sectional view of extrusion system and flow channels used for feedstock



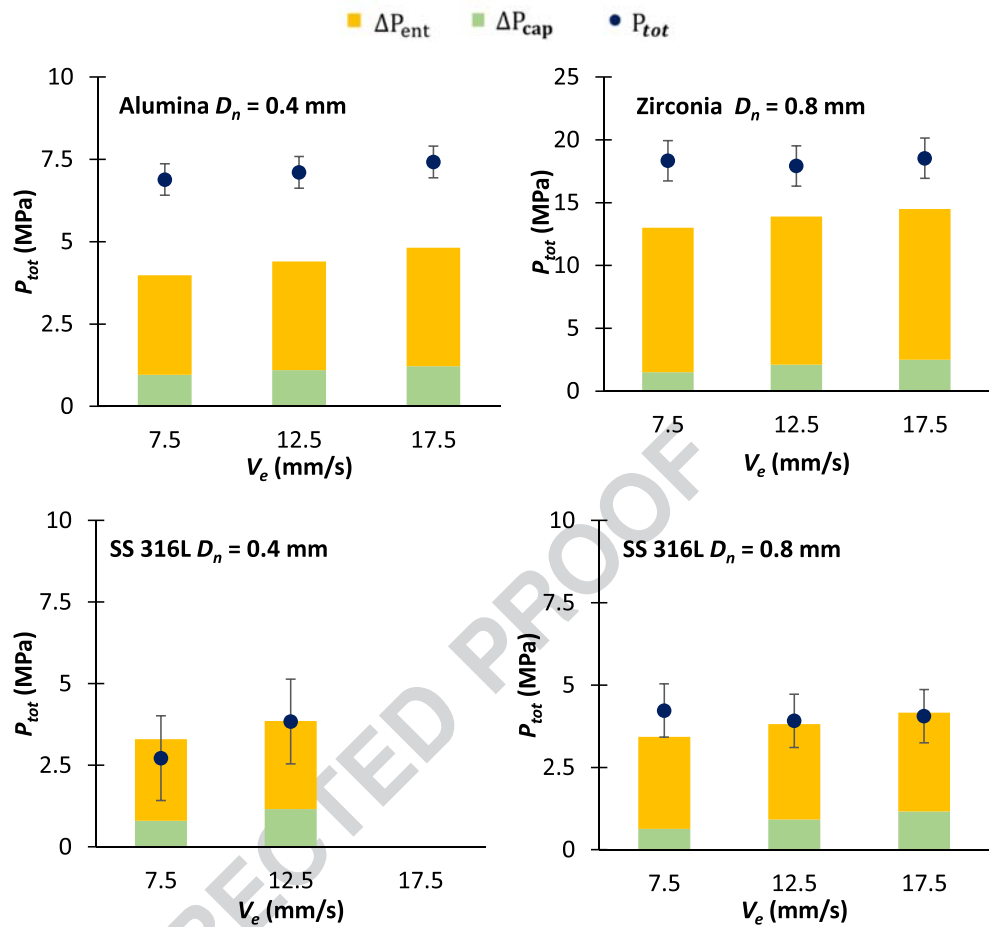
558 geometrically complex and can be reconducted into a series of
 559 cylindrical capillaries, the pressure drops of which can be
 560 estimated using Eq. (15).

561 Figure 7 shows a cross section of the flow channels of the
 562 extruder, with the respective pressure drop in the channels
 563 during the feedstock flows shown on the right side. From
 564 the main extruder chamber (A), the material flow is divided
 565 into two identical sections (B), which are further split into two
 566 long tubes (C) from each section. The material flow from
 567 these four channels flows into the nozzle region (D).

568 The cross-sectional area of the flow channels also accounts
 569 for the determination of the pressure drop through the extru-
 570 sion unit and is subdivided into 11 zones with simple geom-
 571 etries. The elongational fraction of Eq. (15), ΔP_{ent} , has been
 572 computed only for the entrance of sections 3, 5, 6 and 11
 573 because these sections represent a restriction of the flow. In
 574 the shear fraction of Eq. (15), ΔP_{cap} has been measured at
 575 all sections after section 2, but is significant only at sections 6 and
 576 11 because of the extremely high aspect ratio L/D_n . The right
 577 side of Fig. 7 shows a representative plot of the pressure drop
 578 across the 11 sections measured at $V_e = 12.5$ mm/s and $D_n =$
 579 0.4 mm for the alumina and steel feedstocks.

580 The total pressure drops under all experimental conditions
 581 were measured and compared with the experimental values.
 582 This comparison is summarized in Fig. 8, which shows that
 583 the measured pressure (P_{tot}) and the calculated pressure compo-
 584 nents ΔP_{cap} and ΔP_{ent} increase linearly with an extrusion at
 585 velocity V_e because of the larger flow rate.

Fig. 8 Experimental and calculated extrusion pressures for different feedstocks and extrusion velocities; error bars are plotted equal to the pooled standard deviation of the V_e values of each graph



586 Zirconia requires more than double the pressure required
 587 by the alumina and SS316L. The alumina feedstock P_{tot} re-
 588 quires between 6 and 8 MPa of pressure, whereas the SS316L
 589 feedstock requires P_{tot} at below 6 MPa. The predicted pres-
 590 sures properly capture the actual measure pressured for stain-
 591 less steel, whereas they underestimate the actual pressure re-
 592 quirements of the two ceramic materials. This underestimation
 593 is probably connected with the different thermal values of the

594 ceramic feedstock (having a larger thermal diffusivity, as
 595 shown in Table 2), and they likely cool faster than the steel
 596 as soon as they approach the exit of the nozzle, with a viscos-
 597 ity increase that cannot be captured by the model inside the
 598 extrusion unit. Although the model underestimates P_{tot} for the
 599 ceramic feedstocks, it clearly gives an indication of the rela-
 600 tive importance of the shear ΔP_{cap} and elongational ΔP_{ent}
 601 components of the pressure. Here, ΔP_{cap} is significantly larger

Fig. 9 Coefficient of variation COV_P of the pressure signal for the different types of tests and materials

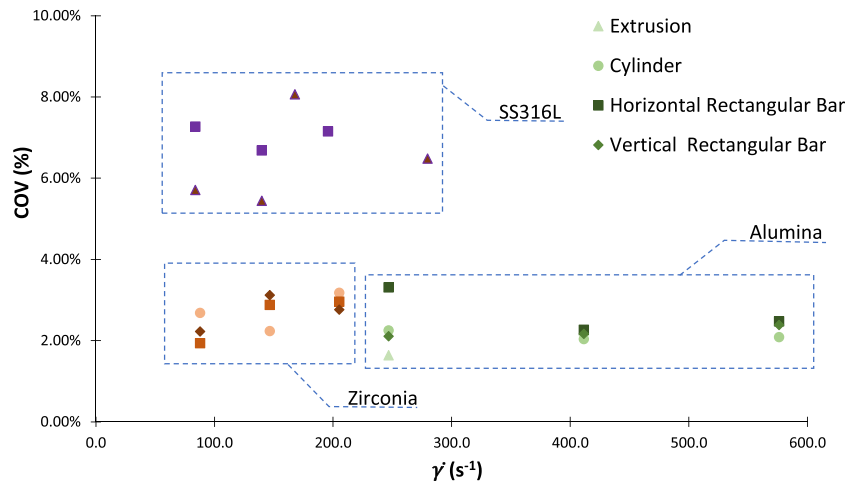
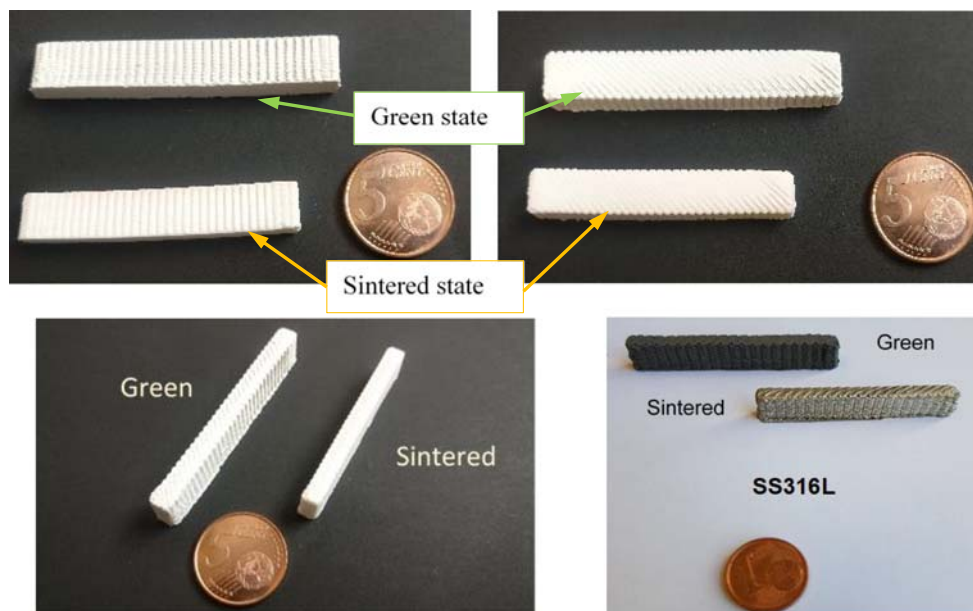


Fig. 10 Green and sintered alumina in rectangular bar-shaped parts printed in a vertical (top left) and horizontal (top right) configuration and green and sintered steel (bottom right) and zirconia (bottom left) parts



602 for all cases, as further proof of the dramatic importance of the
 603 elongational deformation, rather than the shear deformation,
 604 during the EAM processes of highly viscous feedstocks.

605 **5.1 Quality and stability of the extrusion and 3D**
 606 **printing process**

607 A complete representation of the mechanical and geometrical
 608 sintered properties of the 3D printed ceramic materials is not
 609 the main focus of the present paper, which is aimed at the
 610 consequences of the rheology of viscous melts on the pressure
 611 values. Previous results of the sintering properties regarding
 612 this aspect are available in [34, 35]. In the section, the corre-
 613 lation between the variability of the pressure signal and the
 614 variability of sintered quality is investigated.

615 As shown in Table 3, several samples of different shapes
 616 have been 3D printed in their green state, i.e. when the powder
 617 is mixed into the thermoplastic polymeric binder. The pressure
 618 signals during each test were recorded, along with their coef-
 619 ficients of variation COV_p (the ratio between the standard
 620 deviation of each pressure sample and the mean sampled pres-
 621 sure). The results were statistically analysed and clearly show
 622 that COV_p depends on the feedstock material, with stainless
 623 steel being significantly less stable than zirconia and alumina.

By contrast, the error COV_p in the pressure signal measured
 using the four different types of printing (free extrusion, cyl-
 625 626 627 628 629 630 631 632 633
 634 635 636 637 638 639 640 641 642 643
 644 645 646 647 648 649 650 651 652 653 654 655 656
 657 658 659 660 661 662 663 664 665 666 667 668 669 670 671 672 673 674 675 676 677 678 679 680 681 682 683 684 685 686 687 688 689 690 691 692 693 694 695 696 697 698 699 700

After 3D printing, some of the samples underwent de-
 634 binding and were sintered to better understand the variations
 635 in their surface quality characteristics. Because this study is
 636 focused on the extrusion pressure, the parts were printed with-
 637 out outer contour roads to enhance the variations owing to the
 638 start and stops and directional changes. All samples therefore
 639 show an extremely rough surface finish in a green state, which
 640 mildly improves after sintering. Representative 3D printed
 641 parts in their green and sintered states are shown in Fig. 10,
 642 and their sintered properties are reported in Table 5.

Owing to the sintering, shrinkage reduces the waviness on
 644 the surface, although the structure of the surface texture re-
 645 mains unchanged. To further recognize the role of the printing
 646 parameters on the surface quality, surface characteristics of 3D
 647 printed components are also analysed using SEM. The obser-
 648 vations indicate that the surface quality of the components is
 649 not correlated with COV_p and depends only on the material to
 650 be printed and on the infill and layering parameters, as is well-
 651 known for all EAM processes. As an example, Fig. 11 a to c
 652 compare the surface characteristics of green SS316L samples
 653 printed with different layer heights h . Because the surface
 654 quality of the parts is not influenced by the variations in pres-
 655 sure, a quantitative report of the surface quality data is omitted
 656

t5.1 **Table 5** Sintered properties of alumina, zirconia and S316L at extrusion speed of 12.5 mm/s

t5.2 Material	Alumina	Zirconia	SS316L
t5.3 Density (g/cm ³)	3.60	5.65	7.11
t5.4 Elastic modulus (GPa)	81.5	27.33	77.26

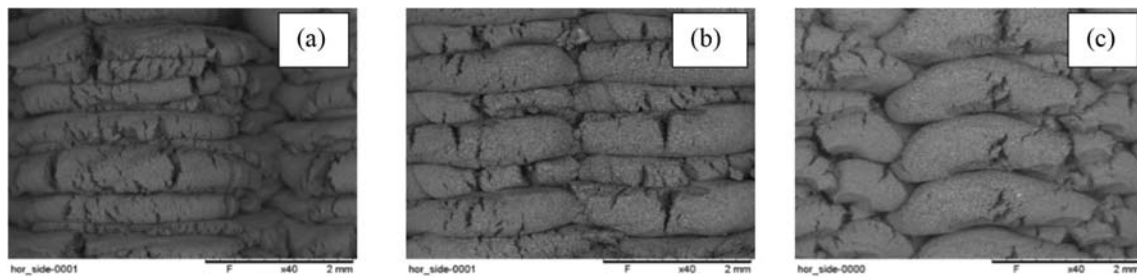


Fig. 11 SEM images for side walls of SS316L samples, printed with a 0.8-mm nozzle and different printed layer heights h of **a** 0.3, **b** 0.4 and **c** 0.5 mm

657 herein for brevity. Zhou et al. developed some numerical op-
 658 timisation approaches to increase the tensile strength and con-
 659 trol the volumetric shrinkage values through different cost
 660 functions dedicated to the polymer FFD process and opti-
 661 mized the processing parameters [36]. This promising method
 662 will be adapted in the future. An estimation of the shrinkage is
 663 a real challenge in additive manufacturing, and Fotovvati et al.
 664 proposed an analytical expression to quantify the size depen-
 665 dency of the dimensional percentage errors with a polynomial
 666 function in the DMLS manufactured features [37]. This meth-
 667 odology will be adjusted during the FFD process in the future.

668 **6 Conclusions**

669 This work is focused on the measurement and prediction of
 670 the instantaneous pressure occurring during the extrusion and
 671 EAM operations of highly viscous powder-binder feedstocks.
 672 A pressure prediction model was developed when considering
 673 both the shear and elongational viscosity contributions. The
 674 material parameters were calculated from capillary rheometry
 675 data, which were also used to validate the model by verifying
 676 its agreement with the experimental viscosity measurements.
 677 An extensive extrusion plan and 3D printing tests were
 678 applied to three different materials (steel, alumina and zirco-
 679 nia) over a range of different nozzle diameters, extrusion ve-
 680 locities and 3D printed shapes. The results indicate that the
 681 pressure requirements owing to the elongational viscosity are
 682 dominant with respect to the contributions of the shear
 683 viscosity.

684 The results also indicate that, among the investigated pa-
 685 rameters, the stability of the pressure signals depends on the
 686 material feedstock and not on the shear rate or shape of the 3D
 687 printed parts.

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