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Mode I critical energy release rate of additively manufactured polyamide samples

Dan Ioan Stoia, Liviu Marsavina, Emanoil Linul

Department of Mechanics and Strength of Materials, Politehnica University of Timisoara, 1 Mihai Viteazu Avenue, 300 222 Timisoara, Romania

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ABSTRACT

One accepted cause that generates low mechanical strength and/or fracture properties is the presence of defects in the structure of the material. Additive Manufacturing does not except this rule, especially the powder bed fusion technologies that relays on powder spreading through a mechanical blade or roller followed by laser sintering or melting. The paper presents experimental investigations on fracture properties of polyamide PA2200 samples obtained by selective laser sintering. The mode I critical energy release rate and mode I fracture toughness were determined in accordance to ASTM D 5528 – 01 for four sets of samples: one set without induced geometrical defects, and three other sets having interlayer and intralayer defects. The results consist of geometrical evaluation of the samples and error computing on one-hand and fracture properties on the other hand. In addition, a study on absorbed energy in the defect section of samples was conducted, leading to a direct correlation between the defect percentages (0.0%, 0.1%, 0.3% and 0.5%) and the absorbed energy.

1. Introduction

Without reaching its maturity yet, the Additive Manufacturing (AM) is considered a key technology for the Third Industrial Revolution [1]. The development stages of these technologies include various studies on geometrical [2–4] and mechanical [5–7] properties of the parts, which will eventually lead to process optimization.

Some studies covering the mechanical behavior of AM samples can be found in the literature [8–10]. However, most works evaluate the fracture toughness or fracture behavior of materials obtained by processes other than Selective Laser Sintering (SLS) [11-18]. The interlayer fracture of non-reinforced Acrylonitrile Butadiene Styrene (ABS) and Carbon Fiber (CF) reinforced ABS composites manufactured by Fused Filament Fabrication (FFF) was studied by Young et al. [11]. Their results reveal a significantly reduction of mode I fracture toughness of the FFF samples compared with Hot-Press Molded (HPM) reference. Kishore et al. [12] assessed the influence of process parameters on the interlayer strength of reinforced ABS, processed by extrusion. The supplementary infrared preheating for preventing the temperature drop under glass transition temperature leads to better fracture energy. The authors observed that the phenomenon is greater influenced in printing at low speeds. Interfacial fracture toughness of ABS and fiber-reinforced polylactic acid composite was tested by Khan et al. [13] for mode I, mode II and mixed mode I/II. The speed of printing confirms its great influence on fracture toughness for Fused Deposition Modeling (FDM). As expected, the nozzle temperature is also directly and positively correlated with the fracture toughness. Using a double compliance method, Xu et al. [14] determine the mode I and mixed modes I/II fracture toughness for CF reinforced composites at low temperatures. They prove the possibility of determining the strain energy release rate from the applied load and resulted displacement, by knowing the compliance of the sample, without measuring the crack propagation. Double Cantilever Beam (DCB) samples of ABS were printed by Aliheidari et al. [15] through FDM process and tested. The interlayer fracture properties were correlated with nozzle and bed temperatures and layer height for underlining how process parameters will influence the fracture strength. A direct and positive correlation between nozzle temperature and fracture energy was observed by the authors, and also an optimum layer thickness was determined. Noori [16] studied the effect of deposition height of polylactide (PLA) on interlayer fracture energy. The rectangular strip samples obtained by filament extrusion were subjected to semicircular grooves for inducing a stress concentration area. The tensile tests reveal the interlayer fracture energy according to the deposition height and annealing. Best results of the author were obtained for extruded samples that did not undergo annealing process. A Multiphysics multi scale modeling of the powder bed in order to identify the possible manufacturing defects was conducted by Mindt et al. [17]. They

* Corresponding author. *E-mail address*: emanoil.linul@upt.ro (E. Linul).

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Nomenclature		HPM	Hot-Press Molded
		K _{IC}	mode I fracture toughness
ABS	Acrylonitrile Butadiene Styrene	L	length of the sample
ao	initial delamination	PA	Polyamide
AM	Additive Manufacturing	PCC	Pearson's correlation coefficient
В	width of the sample	PD	percentage of defects
BD	Brazilian disc	PLA	polylactide
CF	Carbon Fiber	P-value	Pearson's correlation coefficient
CTS	compact tension-shear	R	radius of hinge features
DCB	Double Cantilever Beam	R^2	correlation coefficient
Е	Young's modulus	SCB	semi-circular bend
ECT	edge cracked triangular	SLS	Selective Laser Sintering
ENDB	edge-notched disc bend	W	absorbed energy to failure
F force		XY, YZ, XZ orientation planes	
FFF	Fused Filament Fabrication	δ	displacement at F _{max}
F _{max}	maximum force	Δ	displacement
G _{IC}	critical energy release rate	Φ	diameter of induced defect
H height of the sample		θ	Poisson's ratio

describe by numerical methods the potential defects occurrence during additive manufacturing process. Phenomena like: ejection of large particles, nonuniformities of the powder bed due to particle interaction, non-uniform powder bed spreading due to particle dragging, porosity caused by gas enclosing were described as main causes of part defects [18]. A heat transfer model for laser powder bed fusion was develop in order to estimate the formation of defects in fused part. Parameters such laser power, scanning speed, layer thickness and hatching distance were found to dramatically influence the defect formation. The simulation relay on thermo-physical properties of the considered materials and allow determining the lack of fusion number in relation to the manufacturing parameters. Higher lack of fusion is associated with higher scan speed and lower temperatures, no matter the material used [18].

On the other hand, most investigations on AM samples obtained by the SLS process were focused on tensile behavior [5,19] and only few on the evaluation of fracture parameters (fracture toughness, crack path, crack propagation angle, etc.) [20–23]. Nevertheless, all these studies do not take into account the presence of possible defects in order to analyze the fracture behavior. The mode I fracture toughness of polyamide and alumide samples obtained by SLS additive process was determined by Stoia et al. [20,21]. The authors found the fracture toughness in both materials was highly influenced by the spatial orientation of the samples. Alfano et al. [22] focused on the analysis of fracture behavior in adhesive bonded samples with 3D printed nylon substrates. Circular and square cross-section channels were embedded into the substrates in order to determine the load-displacement response difference in delamination. The higher total work of separation was recorded for square cross-section channels, concluding that subsurface channels are significantly influence the dissipated energy. Recently, Linul et al. [23]

studied the main fracture properties of laser-sintered polyamide using single edge notch bend samples in symmetric and asymmetric four-point bending fixtures. The authors found that process energy and printing orientation significantly influence the mode I and mode II fracture toughness values. In addition, they reported that, regardless of process energy and printing orientation, mode I fracture toughness is greater than mode II.

As a result of the SLS layer-by-layer manufacturing process a layered structure is obtained (Fig. 1a), which in some cases (this phenomenon is very much dependent on process parameters) performs as a laminated/ layered object. Furthermore, it was observed that during the tensile and three-point bending (by using un-notched and notched samples) tests of SLS printed samples the interlayer fracture occurred prior to intralayer fracture (Fig. 1b) [5,19–21,23]. These two observations were decisive in choosing the DCB fracture tests for PA2200. In the same time, the DCB test results are useful for cohesive zone modeling in defining the traction-separation law.

The SLS process use a laser beam for sintering the powder particles quasi-uniform distributed in a previous step. In addition to mechanical distribution of powder, electrostatic charge may also contribute to powder set, and therefore to future discontinuities or defects in the part structure. Therefore, the paper presents a study on how the induced internal defects are influencing the geometrical parameters (shape and size of the samples) and fracture properties (mode I critical energy release rate and mode I fracture toughness) of laser-sintered polyamide. The experimental results were statistically compared with those obtained from defect-free samples manufactured in the same conditions.



Fig. 1. PA2200 layered structure (a) and interlayer fracture under tensile/three-point bending tests (b).

2. Materials and methods

2.1. Materials

The material used in this study is polyamide PA2200 commercially available (Electro Optical Systems - EOS GmbH, Krailling, Germany) in powder form. The physical, chemical and mechanical properties of this were comprehensive described in previous studies [5,19,24], some of them being determined in close relation to the process parameters. For example, the engineering and true stress-strain curves resulted from tensile test on PA2200 samples manufactured with the same printing parameters are plotted in Fig. 2 [19].

In particular, the adequate mechanical properties and high detail resolution are important characteristics of PA2200 processed by SLS. The polyamide PA2200 is a material suitable for additive manufacturing process due to relatively large sinterization window. All these properties make it suitable for a wide range of technical applications. The SLS printed parts can be used in various industrial casings and housings destinated for automotive industry, but also in biomedical field, for constructing of disposable surgical guides for example. The PA2200 powder possesses a biocompatibility certificate according to EN ISO 10993-1 and USP/level VI/121°C and food contact approval in compliance with the EU Plastics Directive 2002/72/EC [25,26].

2.2. Sample design

The sample design is based on specifications of ASTM D 5528 – 01 [27]. An initial model of Double Cantilever Beam (DCB) was constructed in SolidWorks 2020 (3DS North American HQ, USA) as a primary 3D part. In addition to the standard geometry hinge features were added to the model, in order to avoid the necessity of attaching an external one before testing (Fig. 3). These features will provide a pin location through which the force will be applied. The obtained model is a reference one, and was coded 0.0%. The main geometric parameters of the DCB sample are represented by the sample width (b), sample length (L), sample height (h) and initial delamination (a_0).

Using the reference model, right in the proximity of the initial crack end, three different defect area were designed resulting in additional three models coded: 0.1%, 0.3% and 0.5%. The geometrical defects spread on 22 mm \times 22 mm square area, positioned in relation to the DCB sample like in the Fig. 4. Considering that the singular stress field of a crack is around 50% of the total crack length [28,29] most of the defects are placed on the area dominated by the crack tip singularity.

All individual defects have circular cross sections of 0.1 mm radius. The difference between samples consists in how large the total defect area is, compared to the 22 mm \times 22 mm = 484 mm² spreading area. The area of one defect element (circular shape) is $\pi \cdot 0.1^2 = 0.0314$ mm².



Fig. 2. Engineering and true stress-strain curves for PA2200 processed by SLS.

Therefore, 0.1% of defect in 484 mm² is 0.484 mm² which is the equivalent by approximation with 0.0314 mm²·16 defects = 0.5024mm². Following the same procedure, the number of defects was determined for the other two percentages, obtaining that 0.3% defects are representing 47 individual sections and 0.5%, 77 respectively. A random defect distribution was used for every model, resulting in dot maps like in the Fig. 4. In the literature, there are different algorithms for the random distribution of particles inside a predefined region [30,31]. However, the choice of the optimal algorithm is made depending on the particle size, the tested material, the sample geometry, etc. A single criterion was used for defect distribution, in order to avoid the local summation effect: the minimum distance between two defect sections was 4 radii. There are studies in the literature that present the size of the molten pool being bout 90-300 µm [17,18,32,33]. These were determined experimentally and by numerical simulations. Studies on lack of fusion and ejection of particles from the sintering/melting site indicate that for powder consisting of particles of 40–90 μ m (PA2200 case) some of the defects may have circular shapes. Based on this, a random distribution of 200 μ m circular defects spread over a square area (22 mm imes22 mm). A square distribution area was chosen because it is easier to reduce it to unity, for further processing the distribution of defects.

The materialization of the defects on 3D geometry was done in the following way: all defect sections were symmetrically *cut extruded* 1 mm in relation to the delamination planes. The delamination planes are coincident to the material surfaces adjacent to the un-sintered layers (0.2 mm, meaning two layers). The defects were created longer that the distance between delamination planes (Z direction of DCB) in order to be intersected by the crack no matter on which plane will propagate. Geometrical details can be observed in the Fig. 5. A synthetic presentation of the samples with their corresponding defects is described in the Table 1.

Other sample configurations, including Brazilian Disc (BD) [34], Semi-Circular Bend (SCB) [35,36], Edge Cracked Triangular (ECT) [37], Edge-Notched Disc Bend (ENDB) [38,39] and Compact Tension-Shear (CTS) [40,41], are available and can be used for investigating the fracture behavior of different engineering materials under mode I and also mixed mode I/II and I/III fracture. Moreover, the DCB configuration was preferred, over the other available sample geometries, because it allowed quite easy: (i) automatic creation of the crack, (ii) large defect induction surface and (iii) determination of mode I critical energy release rate of additively manufactured polyamide samples. In addition, both by positioning the sample in the printer and by its low volume, it requires low material consumption.

2.3. Samples manufacturing process

The samples were additively manufactured by selective laser sintering (SLS) on EOS Formiga P100 (EOS GmbH Electro Optical Systems) machine. The sample positioning in the building environment and error checking was accomplished in Materialise Magics 10.0 software [42], considering a safety distance of 15 mm between parts and building envelope limits. A total number of 24 samples were arranged in 4 building layers, each containing 6 individual samples of 0.0%, 0.1%, 0.3% and 0.5% samples respectively.

Since this particular sample shape is not desirable for SLS process [2,43], some stiffness ribs were constructed for connecting all samples in one building layer as a whole. This will prevent twisting and bending of the parts when cools [44,45]. The large bottom surface of every sample was placed on the XY building plane. Next, the volume was sliced using an incremental distance of 0.1 mm. The laser trajectories can be observed in the Fig. 6a, red lines being contour lines while green lines are hatching lines. In the detail of the Fig. 6a, it can be observed one hatching line that avoids the internal section of the individual defect, leaving a non-sintered area enclosed by a sintered contour.

After completing the geometrical preparation, the process was start under the following building parameters: building chamber and removal



Fig. 3. Shape and size of modified DCB sample, in mm.



Fig. 4. Random defect spreading on the induced defect area, in mm.



Fig. 5. Induced defects and delamination planes in 3D space, in mm.

Table 1

Design defect	characteristics	for all	l samples.
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Code	Defect size φ [mm]	Spreading area [mm ²]	Number of defects[–]	Total defect area [mm ²]
0.0%	-	-	-	no defects
0.1%	0.2	484	16	0.501
0.3%	0.2	484	46	1.445
0.5%	0.2	484	77	2.418

chamber temperature were set to 170.5 °C and 159 °C, respectively; energy density of 0.067 J/mm², by setting the power to 25 W, the laser velocity to 1500 mm/s and scan spacing to 0.25 mm. A scaling factor of 2.1% was applied on each direction in order to compensate the shrinkage at cooling. All parameters were selected based on previous studies [5,19–21,23] and experience of the authors. Fig. 6b presents the final aspects of DCB samples, at the end of the post-processing (air blasting and cleaning) phase. The samples still possess the stiffness ribs, these being removed in the next step, before acquiring the linear measurements.

2.4. Methods

After removing the stiffness ribs by mechanical cutting, no other process implying material removing or addition was conducted. For every sample the linear dimensions b (sample width), L (sample length), h (sample height) and a_o (initial delamination) were acquired. These were use later on for determining the mode I critical energy release rate and mode I fracture toughness. The dimensional assessment was done at room temperature, using a digital caliper of 0.05 mm accuracy. Every dimension was measured 3 times and an average value was stored.

The fracture tests were conducted on Zwick Roell Z005 quasi-static testing machine equipped with a 5 kN (of 0.1% accuracy) load-cell. The DCB samples were fixed on the machine's grips using half-hinges and metallic pins that assembles to the built-in half-hinge of the DCB. The samples were aligned and centered to the flat and parallel surfaces of the grips. The two hinges are positioned symmetrical on both top and bottom surfaces of sample, located at a_0 distance to the crack tip (initial delamination). These serves as force insertion points, accordingly to the Fig. 7.

The experimental tests were carried out at room temperature according to the ASTM D5528-01 standard [27]. A constant crosshead speed of 5 mm/min was used up to the fracture point. The loaddisplacement data were stored using a sampling frequency of 600 Hz. Fig. 7 presents the fixing configuration of DCB sample together with some details related to the initiation and propagation of the crack.

The different defect incidence in each group of samples require establishing the statistical correlation between the number of defects and the fracture properties. This correlation was performed using the conventional Pearson's correlation coefficient (PCC), which assumes a linear relationship between presence of defects and fracture toughness. The PCC value will always be between -1 and 1. The negative values will prove an inverse relationship between the input and output parameters, while a positive value shows a direct relation. A zero value for PCC will indicate no correlation between parameters [46,47]. In addition to Pearson's correlation, the significance of obtained data was checked by computing the P – value by the probability test [48,49].



Fig. 6. Layer of the middle section (a) and final aspects (b) of DCB samples.



Fig. 7. DCB samples before crack initiation (a), during the crack propagation (b) and after fracture test (c).

3. Results and discussions

3.1. Overall dimensional assessment

The dimensional investigation targets to identify if the presence of defects in the middle section of the samples will or will not have overall geometrical consequences. To this end, the measurements conducted on every sample (L, b and h) were used to compute the relative error on each direction, related with theoretical dimensions. The theoretical or nominal dimensions are the actual size of the 3D model, scaled up by 2.3%, symmetrically. The model scaling is a procedure to compensate the shrinkage of the parts at cooling.

The relative dimensional errors are presented in the Fig. 8 in average value for all four categories of sample. The relative errors of defect-free

samples are presented in wireframe representation, while the other three categories of samples in full color. A linear increase in error values can be observed as the sample defects grow in number, but the errors cannot be discussed all together due to non-symmetry (XY plane versus Z direction) of the building process.

The error of length L (Fig. 8a) is very small in value, under 1% and no tendency among induced defect or defect-free samples can be identified. This is caused by the relative way of computing the error, since the total length is very high in value (nominal 140 mm) compared with dimensional changings associated with shrinkage at cooling.

The error of width b is much larger and a linear tendency can be observed (Fig. 8b). The error values are increasing (in the negative direction) in relation with the number of defects induced in the sample. The smaller (nominal 22 mm) value of this dimension makes the cooling





shrinkage be more significant, especially for the 0.5% samples, where the voids created by the defects allow a larger contraction of the part.

The error of height h is the highest of the three and also linearly growing with the defect number (Fig. 8c). This level of errors is usually expected for Z direction, the growing direction of the part. The geometrical instability in this direction is caused by the free top surface of the powder layer during the process, which allows not only thermal expansion in Z but also allows local particle relocation due to electrostatic effects.

3.2. Fracture properties assessment

For each group of samples (0.0%, 0.1%, 0.3% and 0.5%), the force, F and the displacement, Δ are automatically recorded and plotted using built-in data acquisition system and software. To compare the influence of percentage defects (PDs) on mode I fracture behavior of polyamide PA2200 samples, Fig. 9a presents the most representative F- Δ curves for each category. From this graph, it can be easily observed that the PDs significantly influences the magnitude of the forces. The DCB defect-free samples have the highest maximum force, even up to 48.3% higher than samples with 0.5% PDs. On the other hand, the linear-elastic area (see detail in Fig. 9a) and the size of the displacements do not show major differences between the four types of samples. An important aspect to mention is that with the increase of the PDs, the final fracture of the samples becomes more and more brittle. It seems that the presence of defects speeds up the failure process.

It is well known that the area under the F- Δ curves represents the total absorbed energy (W) [50–52]. Given this fact, Fig. 9b shows a comparison of the W- Δ curves of DCB samples during the experimental test. Like F- Δ curves, in this case it is observed that the presence of

defects considerably decreases the value of the W. Thus, defect-free samples (0.0% PDs) have a W value of up to 221.64 J, while those with most defects (0.5% PDs) store a maximum W of 132.92 J, showing an almost double decrease. Following the analysis of the fractured samples, it was observed that the PDs did not show any effect on the kinking angle in the tested DCB samples.

An important aspect to be analyzed is given by the energy absorbed between the maximum force and the final fracture of the DCB samples. This value highlights the influence of defects on the absorbed energy. Fig. 9a shows the individual load-displacement curves with details on maximum force (F_{max}), displacement at maximum force (δ) and area under the curve beyond maximum force ($W_{0.0\%}$, $W_{0.1\%}$, $W_{0.3\%}$ and $W_{0.5\%}$).

Further, the hatched area of Fig. 10a is represented in Fig. 10b in the form of W versus PD. As can be seen in Fig. 10b, defect-free samples (0.0% PDs) have the highest energy compared to defect-induced samples (0.1%, 0.3% and 0.5% PDs). The samples with 0.0% PDs absorb an amount of 66.65 J energy until final fracture, 44.09% more energy than the samples with 0.1% PDs, in which case a large drop of W can be seen (between 0.0% and 0.1% PDs). Beyond the F_{max} , between the defectinduced samples, no significant differences are observed in terms of W, only 14.13% extreme percentages (W_{0.1%} and W_{0.5%}). For defectinduced samples, the W absorbed after F_{max} decreases linearly with increasing of PDs, no drop being visible as in the previous case (from 0.0% to 0.1% PDs). Some inconsistencies of the increase and decrease of the force with the increase of the displacement are caused by the distribution of the defects in the sample. In this study, a random defect distribution in the defect area was taken into account, so it was not controlled how many defects are in the vicinity of the crack tip and how to align them. For this reason, the influence of the defect was estimated



Fig. 9. F- Δ (a) and W- Δ (b) curves of DCB samples with different defect area (0.0–0.5%).



Fig. 10. F- Δ curves (a) and W-PD variation (b) of DCB samples with different defect area (0.0–0.5%).

by absorbed energy (W-area under the curve) rather than interpreting the results from the slope perspective (brittle/ductile effects). However, as it can be observed in the Fig. 10b, the absorbed energies are decreasing with the defect percentage.

The expression for the critical energy release rate (G_{IC}) of a DCB is given by Eq. (1) [27]. According with Bažant [53] the scale effect applies to different types of materials like concrete, cellular materials, and so one; however there are no available studies for AM components obtained through SLS.

$$G_{IC} = \frac{3F_{max}\delta}{2ba_0} \quad [N/mm] \tag{1}$$

where F_{max} is the maximum force, δ is the displacement at F_{max} , b is the sample width and a_o initial delamination. G_{IC} is a measure of fracture toughness.

Performing the calculations in Eq. (1) and replacing the G_{IC} in Eq. (2) [54], we obtain the value of mode I fracture toughness (K_{IC}).

Table 9			



where E = 1400 MPa is Young's modulus and ϑ = 0.41 is Poisson's ratio of the investigated material [5].

Table 2 shows the geometric parameters of the DCB samples (L, b, h and a_0) together with the main investigated fracture properties (G_{IC} and K_{IC}).

As expected, the mode I critical energy release rate (G_{IC}) and mode I fracture toughness (K_{IC}) values decrease with increasing PDs, Fig. 11.

Moreover, the investigated fracture properties decrease approximately linearly as the PDs increases, presenting a high correlation coefficient ($R^2 > 0.975$). The G_{IC} and K_{IC} properties can be easily predicted using the equations proposed in Fig. 11, of course in the range of 0–0.5% PDs and for the proposed type of defects. In the range of investigated PDs, the energy release rate decreases by 23.49% from 0.0 to 0.5% PDs, while K_{IC} shows a decrease of only 13.14%. Both fracture properties show the largest decrease from 0.1 to 0.3% PDs, of 14.52% for G_{IC},

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PD [%]	Sample lengthL [mm]	Sample widthb [mm]	Sample heighth [mm]	Initial delaminationa _o [mm]	Maximum forceF _{max} [N]	Critical energy release rateG _{IC} [N/mm]	Fracture toughnessK _{IC} [MPa·m ^{0.5}]
0.0	138.67	21.75	6.69	24.50	18.254	0.489	0.907
	138.67	21.75	6.78	24.50	18.288	0.434	0.854
	138.73	21.69	6.86	24.50	20.620	0.595	1.001
	138.71	21.70	6.65	24.50	18.377	0.523	0.939
	138.69	21.68	6.74	24.50	19.288	0.453	0.873
0.1	138.70	21.68	6.75	24.50	13.333	0.463	0.883
	138.70	21.62	6.88	24.50	13.980	0.417	0.837
	138.64	21.67	6.80	24.50	17.154	0.596	1.002
	138.63	21.72	6.86	24.50	12.578	0.382	0.802
	138.59	21.76	6.62	24.50	18.269	0.586	0.993
0.3	138.62	21.73	6.84	24.50	16.893	0.462	0.882
	138.69	21.67	6.88	24.50	14.891	0.421	0.841
	138.66	21.69	6.74	24.50	16.563	0.467	0.886
	138.67	21.72	6.68	24.50	14.624	0.338	0.754
	138.63	21.64	6.79	24.50	17.605	0.403	0.823
0.5	138.70	21.67	6.77	24.50	10.661	0.301	0.711
	138.81	21.71	6.73	24.50	13.591	0.248	0.646
	138.85	21.62	6.80	24.50	14.548	0.383	0.803
	138.67	21.69	6.88	24.50	17.788	0.486	0.905
	138.52	21.68	6.76	24.50	16.272	0.490	0.908



Fig. 11. G_{IC}-PD (a) and K_{IC}-PD (b) variation of DCB samples for different defect area (0.0–0.5%).

respectively 7.33% for K_{IC} . On the contrary, the smallest decreases in properties, of 1.96% (G_I) and 1.22% (K_{IC}) are observed between 0 and 0.1% PDs. Although each individual group of DCB samples had the same pattern of distribution of defects, the obtained results show relatively large standard deviations. This may be due to both the manufacturing process and the initiation and propagation of the crack at the tip of delamination [55–57].

Variation G_{IC} - K_{IC} is presented in Fig. 12 depending on the percentage defects. Regardless of PDs, the mode I fracture toughness has values between 1.84 and 2.08 higher than mode I critical energy release rate. The two material properties (G_{IC} , K_{IC}) vary linearly from each other. Moreover, G_{IC} can be determined using Eq. (1), if K_{IC} is known. The proposed equation has a very good R² correlation coefficient of 0.998. Of course, the lowest values are obtained for the highest PDs.

$$G_{IC} = 0.994 \cdot K_{IC} + 0.411, \quad with \ R^2 = 0.998$$
 (3)

Fig. 13 shows a comparison of the fracture toughness results for different orientations of the samples in the SLS printing chamber. In order to avoid the discrepancies that could appear, K_{IC} results obtained on the same type of material were used, respectively the samples were printed on the same SLS machine and in the same conditions [23]. From the present investigation, the K_{IC} values obtained without inducing defects were used.

According to the building direction, the fracture toughness results are similar with those reported by Brugo and co-workers [10]. The K_{IC}



Fig. 12. G_{IC}-K_{IC} variation for different defect area (0.0–0.5%).

results indicate that the interlayers are the weakest part of the sample, creating a favorable and easy direction for crack propagation. YZ samples shows the lowest values of fracture toughness, with a sharp drop of 59.89% when compared with the XZ configuration and differences of only 16.68% when compared to XY configuration. Therefore, by making a crack parallel to the printing direction significantly favors the initiation and propagation of the crack at much lower forces than if the fracture plane (along the crack) were created perpendicular to the layer plane.

3.3. Parameter correlations

The level of significance in recorded data was evidenced by running the probability test. The defect-free sample parameters (G_{IC} , K_{IC} , L, b, and h) were considered reference data and compared by computing the P-value with the same parameters obtained for 0.1%, 0.3% and 0.5% samples. The results are presented in the Table 3 and indicate with a 95% confidence the differences that are found between reference data and data of defect-induced samples, for each considered parameter.

The statistical correlation between defect percentage (input data) and G_{IC} , K_{IC} , L, b, and h (output data) was calculated using Pearson's correlation (Table 4). Both strain energy release rate and mode I fracture toughness are highly correlated in the negative way to the defect percentage.

The negative sign indicates that lower fracture properties will be obtained by increasing the geometrical defects in the structure. The defects will act as a crack inhibitor but will diminish the sintered material volume in a more significant way.

From dimensional perspective, the data also correlates with defect percentage, the less influenced geometric parameter being the sample length (L). This is because the shrinkage at cooling on this length is less influenced by the local shrinkage in the defect area.

4. Conclusions

This paper presents the effect of geometrical defects induced in the middle section (in front of the crack tip) of Double Cantilever Beam (DCB) polyamide samples on their geometrical parameters (width-b, height-h and length-L) and fracture properties (mode I critical energy release rate- G_{IC} and mode I fracture toughness- K_{IC}). The Selective Laser Sintering (SLS) manufactured samples include defect-free (0.0%) and 0.1%, 0.3%, 0.5% induced geometrical defects on which linear measurements and fracture tests were conducted. The following conclusions can be drawn:



Fig. 13. SLS building directions (a) and K_{IC} variation with sample orientation plane [23] (b).

Table 3

P-values for GIC, KIC, L, b, and h parameters.

Parameter	Induced defect [%]			
	0.1	0.3	0.5	
G _{IC} [N/mm]	0.0700	0.5800	0.0775	
K _{IC} [MPa⋅m ^{0.5}]	0.0728	0.5942	0.0767	
L [mm]	0.0922	0.3576	0.7806	
b [mm]	0.0957	0.2762	0.0607	
h [mm]	0.4473	0.4538	0.1742	

Table 4

Correlation coefficient: induced defect vs. G_{IC} , K_{IC} , L, b, and h parameters.

Defect/ Parameter	G _{IC} [N/ mm]	K _{IC} [MPa∙m ^{0.5}]	L [mm]	b [mm]	h [mm]
(0.0%, 0.1%, 0.3%, 0.5%)	-0.98675	-0.99212	0.417857	-0.69055	0.901257

- The transversal (b and h) dimensions of the samples are highly influenced by the presence of geometric defects, while the longitudinal dimension (L) is less influenced.
- \blacksquare It was found that K_{IC} has values almost two times higher than $G_{IC}.$
- The G_{IC} and K_{IC} values decrease almost linearly with increasing of percentage defects, by 23.49% for G_{IC} and 13.14% for K_{IC} respectively.
- Two linear equations, for predicting G_{IC} and K_{IC} properties on the percentage defects range 0–0.5%, are proposed.
- \blacksquare An equation for determining G_{IC} as a function of K_{IC} has been proposed.
- The data analysis indicates statistical correlations between defect-free and induced defects samples in all investigated parameters (G_{IC}, K_{IC}, L, b, and h).
- A very strong negative correlation (close to −1) was obtained for fracture properties, which means that as the geometric defects in the structure increase, the G_{IC} and K_{IC} will decrease.

CRediT authorship contribution statement

Dan Ioan Stoia: Conceptualization, Formal analysis, Investigation, Writing - original draft, Writing - review & editing. **Liviu Marsavina:** Conceptualization, Resources, Writing - original draft, Writing - review & editing, Funding acquisition. **Emanoil Linul:** Conceptualization, Investigation, Writing - original draft, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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