Micro milling of polymeric micro injected specimens with randomly oriented Carbon Nanotube fillers

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ABSTRACT

The interest in the application of Carbon Nanotube (CNT) composites is recently increasing in several industrial sectors. The main reason for this growing attention is the reinforcing effect of the CNTs. However, the composite use is limited by technological issues concerning the manufacturing processes when small features are required. A multistage process chain could exploit the advantages of suitable processes to enhance the control of the filler orientation. This paper investigates the feasibility of milling micro features on micro injected specimens of POM/CNT and LCP/CNT composites. Design of Experiment is used to study a suitable experimental design to investigate the influence of the material and the process parameters on the machinability and the feature geometry. POM-based composites showed a better machinability and allowed a fabrication of more accurate features, while LCP showed high cutting forces and the presence of diffused burrs, preventing the fabrication of very small features.

KEYWORDS

Micro machining; micro milling; micro injection moulding; carbon nanotubes; polymer composites.

1. INTRODUCTION

Currently, products made of polymers are present in all the market sectors and fulfil numerous purposes. Indeed, a wide range of polymeric materials is available, thus allowing to choose the polymer having the most suitable characteristics based on the product specifications (e.g. great processability, low weight, chemical endurance and thermal insulation). Nevertheless, the need of new materials with specific property combinations that cannot be provided by standard polymers arises from the continuous demand of low cost and high performing miniaturized products.

The new market requests have been recently fulfilled by developing novel polymeric materials. The polymer-based composites are multiphase solid materials consisting of a continuous phase (i.e. polymeric matrix), which incorporates one or more discontinuous phases (i.e. fillers). In the finished structure of such materials the different components are separate and distinct: the fillers keep their relative positions in the matrix that surrounds and sustains them, and the matrix properties are enhanced by the fillers. The properties and the behaviour of composites depend on the intrinsic properties, form, structural arrangement and percentage of their constituents, and on the interaction between them. The interaction between matrix and fillers is a very influent and not easily predictable aspect of polymer composites. The filler distribution in the matrix is affected by the mutual interaction between the constituents, as well as the transfer of the filler behaviour to the composite.

Composites containing nanoparticles are particularly attractive since the filler dimensions in the order of nanometers increase the interaction surface, therefore enhancing the impact of the filler properties. The dispersion and orientation of the fillers significantly influence the nanocomposite characteristics, thus the processing operation is of utmost importance to obtain a homogenous compound with a uniform dispersion of the filler within the matrix (Hussain, 2006). Nanocomposites reinforced with carbon-based fillers have shown to generate great interest and close attention has been paid on carbon nanotubes (CNTs), since they have not only an excellent electrical and thermal conductivity, but also superior mechanical properties provided by the strength of the covalent carbon-carbon bonds. For the aforementioned reasons, nowadays the use of polymer-based composites with CNTs is increasing in a large variety of sectors, from automotive to aircraft and defence, bioengineering, electronics, sports, leisure and consumer goods (Breuer and Sundarraraj, 2004).

Polymer-based materials can be processed easily and at a low cost thanks to mass production technologies (e.g. injection moulding, hot embossing, etc.) that allow to replicate complex geometries with high accuracy. The replication of microscale features can be performed by the conventional injection of a molten polymeric material into a moulding cavity, achieving very high production rates and, thus, a cost reduction. Therefore, micro injection moulding is very suitable for the fabrication of high performing miniaturized plastic products and components. However, at the microscale this process involves high shear rates that are likely to influence the internal structure of the material and also to align and orient the fillers, in case of composites (Advani, 2007). Indeed, microscopic features require the material to go through very narrow channel that may stretch the molecules and favour the elongation of clusters and the alignment of fillers along the flow direction. The narrower the channels, the higher the shear rates and the effect on the internal structure of the material. This effect can be observed in Figure 1b: the fillers appear aligned with the flow direction, indicated by the arrow at the bottom left corner of the picture.

The images in Figure 1 are taken from specimens made of the same composite based on polyoxymethylene (POM), but fabricated using micro injection moulding with two different moulds (Figure 2), whose features produce different share rates. The sample in Figure 1a is made using the mould in Figure 2a, which has a lower share rate (3×10^3 s^{-1}) and, as a consequence, the fillers are not affected by the injection process. The sample in Figure 1b is made using the mould in Figure 2b, which contains five micro channels with submillimetric dimensions: 0.2 mm and 0.1 mm are the widths of the top and the bottom of the channels and the depth is 0.2 mm, as shown in the inlet of Figure 2b. In this mould the reduced dimensions generate a higher shear rate (8×10^5 s^{-1}), affecting the filler orientation in the matrix.

The composites present an anisotropic behaviour and the filler alignment affects their properties, hence, for some applications a different fabrication process is needed to enhance the control of the filler distribution. For this purpose, a multistage process chain can be used, exploiting the advantages of various manufacturing processes to obtain the desired product characteristics (Mahmoodi, Mostofa et al., 2013; Attanasio et al., 2017).

This paper studies an alternative way to obtain micro features, based on the machining of specimens fabricated using micro injection moulding, but with low shear rate, so that the filler position and orientation are not affected by the process. This method based on micro milling is promising for the small batch manufacturing of extremely accurate products with micro features, such as MEMS and microfluidic devices (Guckenberger et al., 2015).

The conventional machining processes for polymeric materials (Kobayashi, 2011) and for polymer-based composites (Mata et al., 2008; Sheikh-Ahmad, 2009; Karataş and Gökkaya,

2018) is challenging because of several characteristics of these materials, including the thermal expansion, moisture absorption, residual stress, ductility and, in case of composites, heterogeneous and anisotropic structure. Micro machining is even more difficult due to the lower tolerances involved. Despite the increasing interest in the topic, few papers have been found in the literature on micro milling of plastic materials.

Investigations on viscoelastic polymeric materials are reported by Kakinuma et al. (2012). Cryogenic micro milling was studied in order to increase the machinability of polydimethylsiloxane (PDMS), which is a challenging material because of its high elasticity and adhesion. The results demonstrated the possibility of using this technique for millimetric to sub-micrometric features. Khilwani et al. (2011) performed a research on the machinability of polymethylmethacrylate (PMMA) and polytetrafluoroethylene (PTFE) at the microscale. They manufactured micro channels with different feed rates, using two-flute end-mills with a diameter of 300 μm and 1 mm, and they measured the cutting force and the resulting surface quality in terms of roughness. The experimental results showed that both the roughness and the cutting force were lower for PMMA than for PTFE, when using the same cutting parameters. The micro milling of PTFE was affected by burr formation, which was probably caused by ploughing phenomena. Miranda-Giraldo et al. (2017) investigated the micro milling of a biopolymer (silicone hydrogel) applying compressed air to control the burr formation. Micro slotting operations with a 200 μm diameter mill were carried out with different air flux conditions and air temperatures, and the top burrs of the machined slots were measured.

Kuram (2016) studied the micro machinability of unreinforced polyamide 6 and polyamide 6 with glass-fibres as fillers, focusing on the influence of the cutting speed and feed rate on the cutting force, tool wear, burr formation and surface roughness. The experimental results pointed out that the fillers decrease the material machinability, thus increasing the tool wear, cutting force and surface roughness. Mahmoodi, Mostofa et al. (2013) and Mahmoodi, TabkhPaz et al. (2013) investigated the machinability of a polycarbonate (PC) matrix reinforced with graphene nano platelets and CNTs. Dog-bone specimens were realized by injection moulding and then mechanical micro machining was performed on them using a two-flute end-mill. Based on the experimental results, the machinability of nano-composites in terms of surface quality and chip size proved to be better than the machinability of the pure PC, due to the improved thermal conductivity and mechanical characteristics. Chu et al. (2015) performed micro slotting experiments using two-flute end-mills with 508 μm of diameter to investigate the machinability of a hierarchical three-phase composite consisting of an epoxy matrix, microscale glass fibres and nanoscale graphene platelets.

This paper focuses on the machining of micro features characterized by a high aspect ratio (i.e. ratio between height and thickness), which are typical of microfluidic devices. Such an application requires a very high geometrical and dimensional accuracy, thus these features represent a challenge in micro milling. Indeed, since the tools and features have a low stiffness, the cutting force is likely to produce their bending or vibrations that reduce the feature final quality. Hence, the knowledge of the relationships between process parameters, cutting forces and feature quality is of utmost importance since it could be used to meet the geometrical and dimensional tolerances by acting on process parameters to obtain appropriate force values. Several models describing these relationships have been developed based on different theories for cutting force prediction, for instance on semi-empirical models, also called mechanistic models (Seguy et al., 2008; Uriarte et al., 2008; Annoni et al., 2016). However, an effective modelling of the behaviour of composite materials is demanding, thus, in this case, extensive experimental campaigns are required to set empirical models.

This paper describes the feasibility study of manufacturing thin walls and pillars on specimens produced by micro injection moulding using two composites with polyoxymethylene (POM) and liquid crystalline polymers (LCP) as matrix materials and CNTs as fillers. In particular, the experimental study investigates the influence of the process parameters on the cutting force and the feature accuracy.

2. MATERIALS AND METHODS

The specimens were fabricated following three steps: firstly, the compounds were prepared, adding the fillers to the matrix, secondly, the composites were shaped using a micro injection moulding machine and finally, the obtained specimens were machined.

Two composites based on two polymeric matrixes were prepared. The selected polyoxymethylene (POM) was the Ultraform® N2320 003 from BASF with a grade for injection moulding. This material has a density equal to 1.4 g/cm^3 (ISO 1183) and a melt volume flow rate (MVR) of 7.5 cm^3/10 min, evaluated according to ISO 1133 with an extruding mass of 2.16 kg and at 190°C. The Liquid Crystal Polymer (LCP) was the Vectra® A950RX from Celanese Engineered Materials, an unfilled grade with a density of 1.4 g/cm^3 (ISO 1183). No MVR data are provided by the manufacturer.

The fillers were multiwalled CNTs produced by Bayer Material Science AG (Baytubes C150P) that have an average diameter and length of 10.5 nm and 770 nm, respectively, and a bulk density equal to 120–170 kg/m^3. For each polymer matrix two composites with 3% and 6% in weight of CNTs were compounded.

The compounding was carried out using a LabTech intermeshing co-rotating twin-screw extruder with a screw diameter of 16 mm and a screw length to diameter ratio L/D = 40. The main hopper was fed simultaneously with the polymeric granules and filler powder using two volumetric dosing units, which have been previously calibrated to convert the volumetric flow rate into the mass flow rate.

Once the extrusion process has reached a steady state, the extruded strands were cut to obtain pellets, which were then injected into a two-cavity steel mould (Figure 2a) using a DesmaTec FormicaPlast 1K injection machine. The polymers and their composites have been dried in order to avoid issues related to moisture during the compounding and moulding processes (POM and composites at 110°C for 3 hours, and LCP and composites at 150°C for 4 hours).

Before machining the micro features, several tests were performed on the materials before and after moulding and the results are presented in Bongiorno et al. (2017). Rheological analyses confirmed that no degradation of the material occurred and pointed out that the dispersion of CNTs in POM composites was adequate to achieve the network formation. This result was also confirmed from electrical analyses, which showed a significant increase in the conductivity of the composites with respect to the pure polymer. Based on the results of optical and Transmission Electron Microscopy (TEM) analyses, in the POM composites a good dispersion of the filler was observable, while the LCP composites exhibited big dense agglomerates of CNTs instead of a network.

The milling operations were performed on the micro injected specimens using a Kern Pyramid Nano ultra-precision CNC machine. The selected tool RIME HM79/04 (Figure 3) is a two-flute end-mill made of uncoated micrograin tungsten carbide (grade K20/K40; Co = 10 %, WC including doping = 90 %). The tool geometrical characteristics are listed in Table 1. Even if the absence of coating can reduce the tool life, when cutting polymers this condition is suggested in order to obtain sharper cutting edges and, consequently, to prevent the built-up-edge formation that could lead to a premature tool failure.

3. EXPERIMENTAL DESIGN

A suitable experimental design (Table 2) was studied according to Design of Experiments (DoE) to point out the effects of the feature dimension, material/composite and injection moulding temperature on the cutting forces and the feature accuracy. The specimens were made of one of the two polymers (POM, LCP) or their composites with the two different percentage of CNTs. Concerning the injection moulding process, two different mould temperatures were examined, since this parameter was found to be significant on the mechanical properties of the polymeric material (Baldi et al., 2014). Two types of micro features (thin-walls and pillars) were machined on the specimens with process parameters that were optimized based on a preliminary experimental campaign. In particular, three different thicknesses (200 µm, 100 µm and 50 µm) of thin-walls were studied, while the height was set to 400 µm. The round micro pillars had a diameter equal to 400 µm and a height equal to 300 µm. Based on preliminary experiments, the selected spindle speed was 45,000 rpm (cutting speed = 56.5 m/min) and the tests were conducted in dry condition. The thin-walls resulted from one-step full-slotting operations that were performed with a feed rate of 100 mm/min. The use of the same axial depth of cut on both wall sides is not recommended, because the wall is not supported by any material when milling its second side and, thus, it could be deflected by the cutting forces (Annoni et al., 2015). However, an axial depth of cut equal to the wall height ensures a better quality of both the wall sides. Indeed, it prevents the formation of step-like defects due to the wall bending in the subsequent passes. The micro pillars were machined using a helicoidal strategy, allowing to partially support them when milling the opposite side (Annoni et al., 2014). In this case, based on a preliminary experimental campaign, the feed rate was set equal to 100 mm/min and the axial depth of cut equal to 0.1 mm. Three replicates for each working condition were carried out, hence 18 specimens for each material were machined. The machining tests were completely randomized and a new micro end-mill was utilised for each specimen.

The analysed responses were the maximum cutting force value and the thin-wall thickness or pillar diameter error, which expresses the dimensional accuracy of the features. The cutting force was measured using a Kistler 3-components piezoelectric force sensor (type 9317C). The signals coming from the load cell were amplified by 3 Kistler charge amplifiers (type 5015A) and, subsequently, acquired by a National Instrument data acquisition system (cDAQ-9174) equipped with a National Instruments 9205 board. A sampling frequency of 40 kHz was selected in order to avoid aliasing effects and, thus, ensure a suitable signal resolution.

A Mitutoyo Quick Scope 200 optical coordinate measuring machine (measuring resolution = 0.5 µm, minimum accuracy = 2.5 µm) was used to acquire the geometry of the machined thin-walls and pillars. The dimensional error was calculated subtracting the nominal value of the thin-wall thickness or pillar diameter from the measured value. Positive error values correspond to actual feature dimensions that are bigger than the nominal ones.

4. EXPERIMENTAL RESULTS

In order to point out the effects of the material/composite, injection moulding temperature and thin-wall dimension on the cutting forces and feature accuracy, suitable models have been analysed. The ANOVA results are summarized in Table 3 and Table 4, while Figure 4, Figure 5 and Figure 7 detail the results in terms of maximum cutting force and geometrical accuracy.

The cutting force is influenced by the material and, in case of both thin-wall and pillar milling, the highest values were measured for pure LCP (Figure 4), probably due to its higher crystallinity degree. Regarding the other significant main factor, an increasing percentage of CNTs reduces the cutting force, thus reflecting the material machinability and the feature quality enhancement. The pictures in Figure 6 depict one representative specimen for each experimental condition and show that the burr amount is higher and the thin-wall quality is worse for the material with a lower machinability (LCP).

The interval plots in Figure 4 show that the measured cutting forces have a lower variance for POM-based materials. This fact could be due to the improved filler dispersion in the POM matrix, as observed from the rheological and TEM analyses (Bongiorno et al., 2017). Indeed, a better filler dispersion could lead to more stable cutting conditions, both within each specimen and between different specimens, and thus the cutting force variance between the three replicates should be lower.

The thin-wall thickness errors are reported in Figure 5 for POM and LCP composites. The LCP/CNT specimens have a higher amount of burrs compared to POM/CNT specimens (Figure 6), thus most of their 50 µm thick walls cannot be measured properly, while the 100 µm and 200 µm thick walls exhibit highly variable dimensions. The material and the CNT percentage have proved to influence the wall thickness error (Table 4) and Figure 5 shows that the error is generally lower for POM composites and for higher CNT percentages. This effect is related to the composite machinability. The wall thickness error is also affected by the feature nominal thickness for both materials. The lower the thickness, the lower the wall stiffness, therefore the wall is more likely to deflect during the milling of its second side. Due to this fact, the actual depth of cut value decreases and the mill is able to remove less material than expected, creating a thicker wall.

The errors, calculated from the pillar diameter measurements, are reported in Figure 7 for POM and LCP composites, while Figure 8 and Figure 9 show the pictures of pillars fabricated on POM and LCP composites (one representative specimen is reported for each experimental condition). As for the thin-walls, POM composites exhibit a better quality and less burrs. The ANOVA results (Table 3) show that the ability of reproducing pillars is influenced only by the CNT content, but the error is generally low, meaning that the helicoidal tool path is effective in decreasing the pillar bending.

5. CONCLUSIONS

This paper is focused on the machining of polymer composites. The injection moulding process can influence the properties of polymeric products and, in case of materials with two phases, as composites, the interaction between the two phases can also be affected by the involved high shear rates. Therefore, the choice of the correct process chain is critical to fulfil the product specification. In this context, machining could be a valid fabrication process in case of low volume productions.

In this study the milling process was used to manufacture micro features, such as walls and pillars with thickness or diameter of hundreds of microns, as an alternative to the micro injection moulding process of submillimetric features.

A Design of Experiment approach was adopted to investigate the influence of the process parameters on the machinability and the geometrical accuracy of the features. Two polymeric matrixes were investigated (POM and LCP) with two CNT content percentages (3% wt. and 6% wt.). The samples were injection moulded in a mould with low shear rate, selecting two mould temperatures. Finally, thin-walls with different thickness and micro pillars were manufactured.

The following results were obtained:

- Concerning the machinability, the POM based materials showed a higher stability, less burr and lower cutting forces. The diffused burrs originated in LCP machining prevented the fabrication of very small features. The cutting forces appeared influenced by also the CNT percentage, with lower forces corresponding to higher percentages.
- Concerning the measured geometries, the ANOVA results suggested that the material, the CNT percentage and, in case of thin walls, the nominal thickness, affect the feature dimensions.

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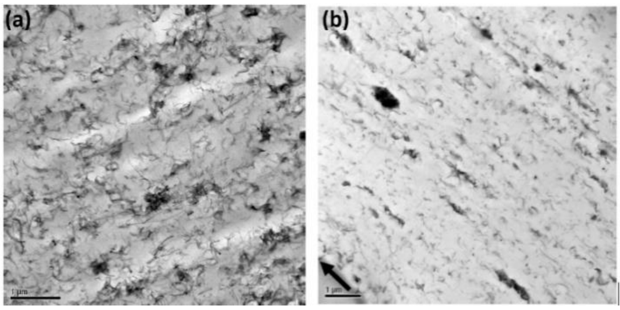

Figure 1 TEM images of CNT/POM composites after injection moulding with different share rates: (a) 3 x 10^3 s^{-1} and (b) 8 x 10^5 s^{-1} (Pagano et al., 2014)

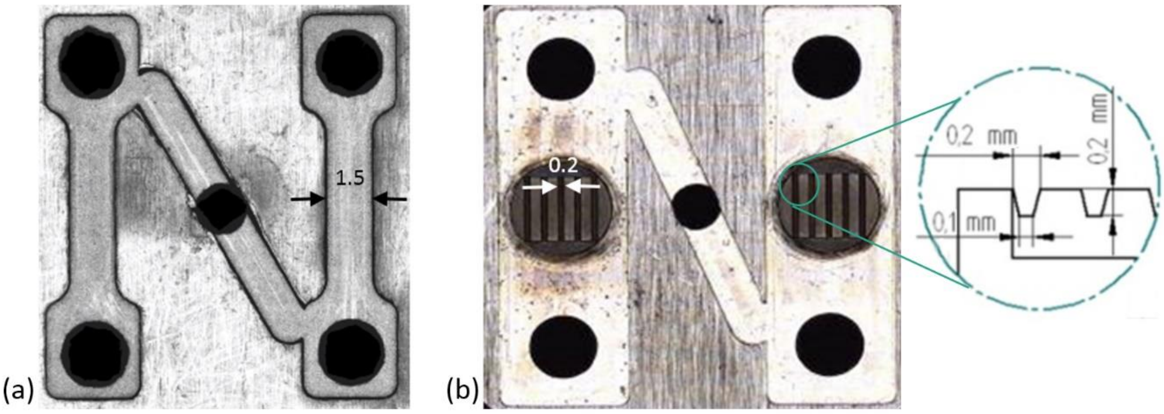

Figure 2 Pictures of the two moulds for producing (a) mini dog bones, (b) micro thin walls by means of an insert with micro channels, the inlet shows a drawing of a section of the circled area, perpendicular to the plane of the picture. All dimensions are in mm.

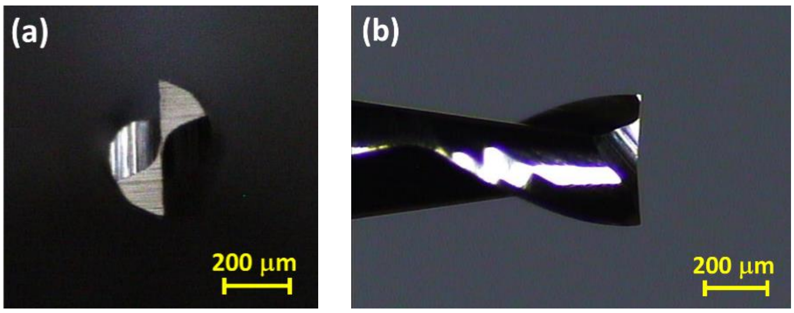

Figure 3 Optical microscope images of the selected tool: (a) bottom view, (b) side view.

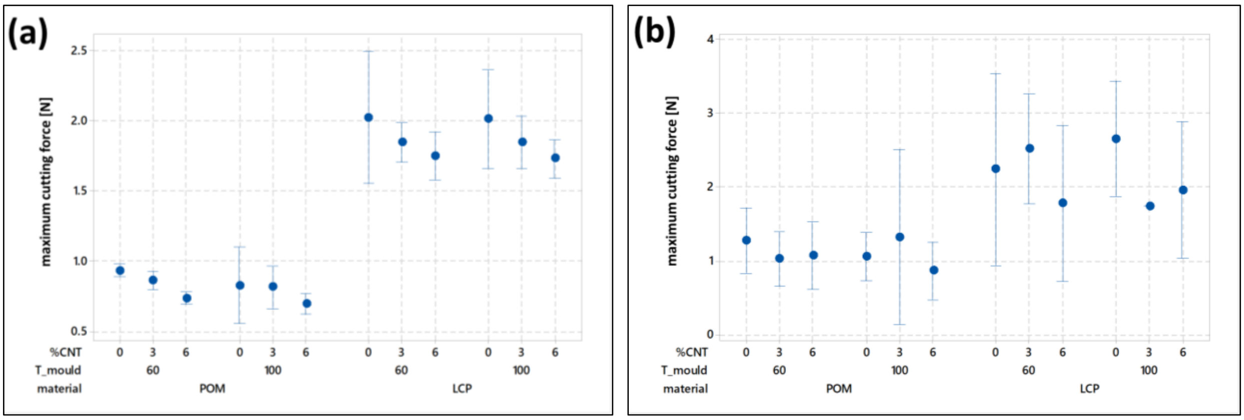

Figure 4 Interval plots of maximum cutting force for POM and LCP composites during (a) slotting and (b) pillar milling operations.

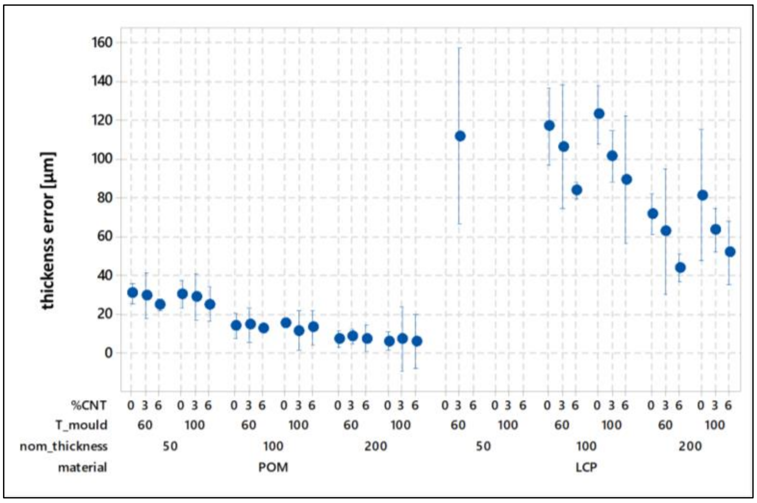

Figure 5 Interval plot of thin-wall thickness error.

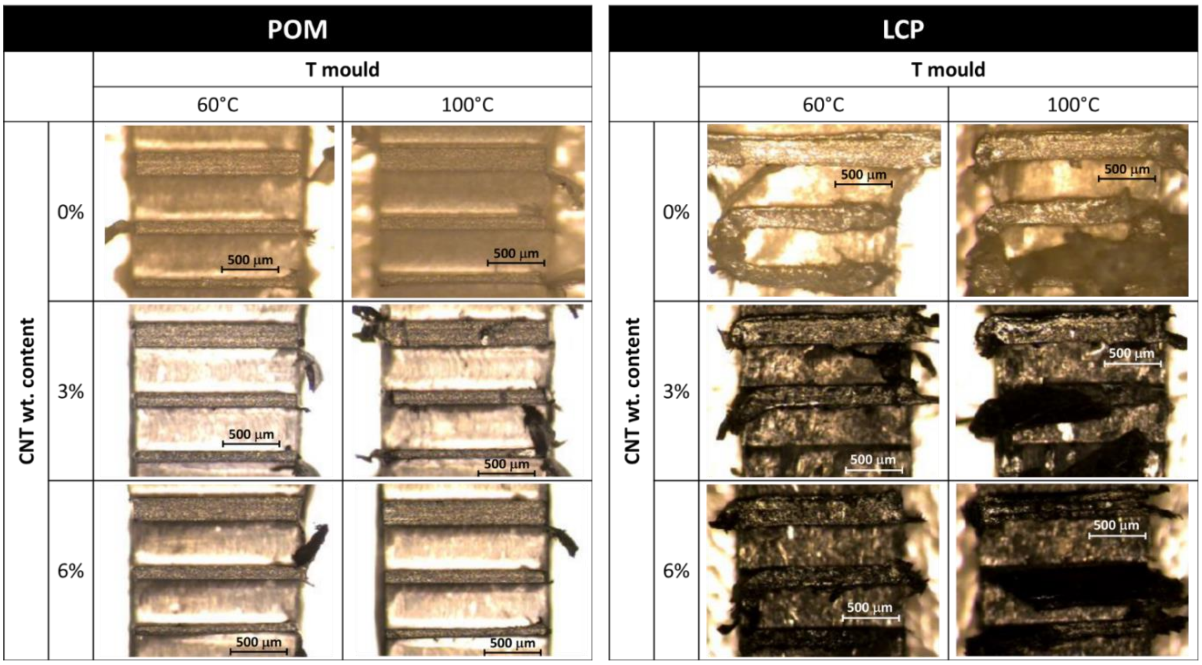

Figure 6 Top view of thin-walls on POM and LCP composites.

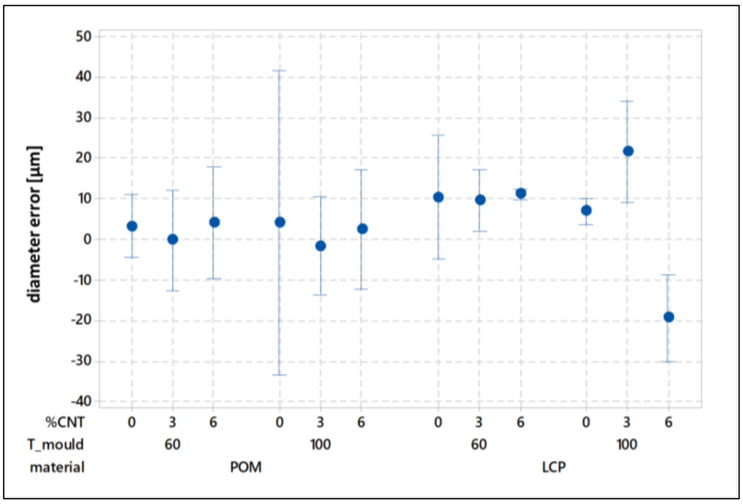

Figure 7 Interval plot of pillar diameter error.

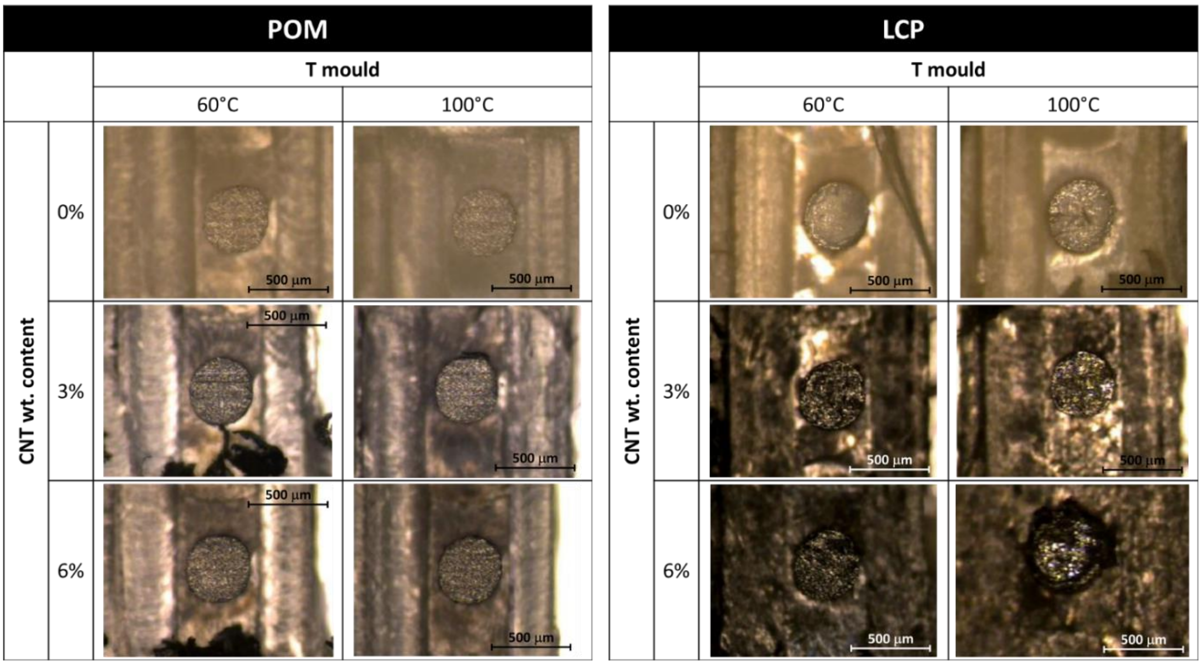

Figure 8 Top view of pillars on POM and LCP composites.

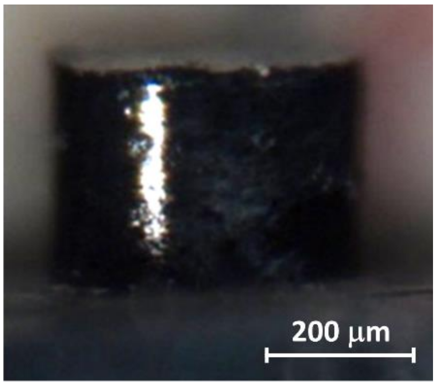

Figure 9 Side view of a pillar on POM composite (6% wt. CNT; T mould = 100°C).

Table 1 Tool geometrical characteristics.

Parameter	Value
Diameter	400 µm
Cutting length	400 µm
Radial rake angle	8°
Radial clearance angle	15°
Axial rake angle	0°
Axial clearance angle	7°
Helix angle	30°

Table 2 Experimental design (variables, levels and responses).

Factors	Levels	
Material	POM; LCP	
CNT wt. content	0%; 3%; 6%	
T_{mould}	60°C; 100°C	
Thin-wall thickness	50 µm; 100 µm; 200 µm	
Constant parameters		
Pillar diameter	400 µm	
Responses		
Maximum cutting force	Thin-wall thickness error	Pillar diameter error

Table 3 ANOVA p-values (dark grey = significant factor, grey = nearly significant factor, confidence level *α* = 1%) for the analysis on maximum cutting force in thin-wall and pillar milling, and the pillar diameter error.

			Main factors			Factor interactions			
			Material (M)	% CNT (C)	T mould (TM)	M*C	M*TM	C*TM	M*C*TM
Responses	Pillars	Maximum cutting force	**0.000**	**0.017**	0.631	0.285	0.940	0.398	**0.005**
		Diameter error	0.029	**0.009**	0.057	**0.000**	0.117	**0.001**	**0.001**
	Thin-walls	Maximum cutting force	**0.000**	**0.000**	0.206	0.148	0.335	0.889	0.878

Table 4 ANOVA p-values (dark grey = significant factor, grey = nearly significant factor, confidence level α = 1%) for the analysis on thin-wall thickness error.

			Main factors				Factor interactions					
			Material (M)	% CNT (C)	T mould (TM)	Nominal thickness (TH)	M*C	M*TM	M*TH	C*TM	C*TH	TM*TH
Response	Thin-walls	Thickness error	**0.000**	**0.000**	0.061	**0.000**	**0.000**	**0.014**	**0.000**	0.936	0.037	0.818