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Fast cavity surface temperature evolution in injection molding: control of cooling stage and final morphology analysis.

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Injection molding experiments

Molded samples were produced by a 70-ton Negri-Bossi reciprocating screw injection molding machine. A rectangular cavity having a length L=110 mm, width W=12.7 mm, and thickness S=1.5 mm was adopted. A rectangular section gate, with a thickness of 1.2 mm, was located between the runner and the cavity. Fig. S1 shows the geometry with all dimension of the molded part adopted for the present experiments.

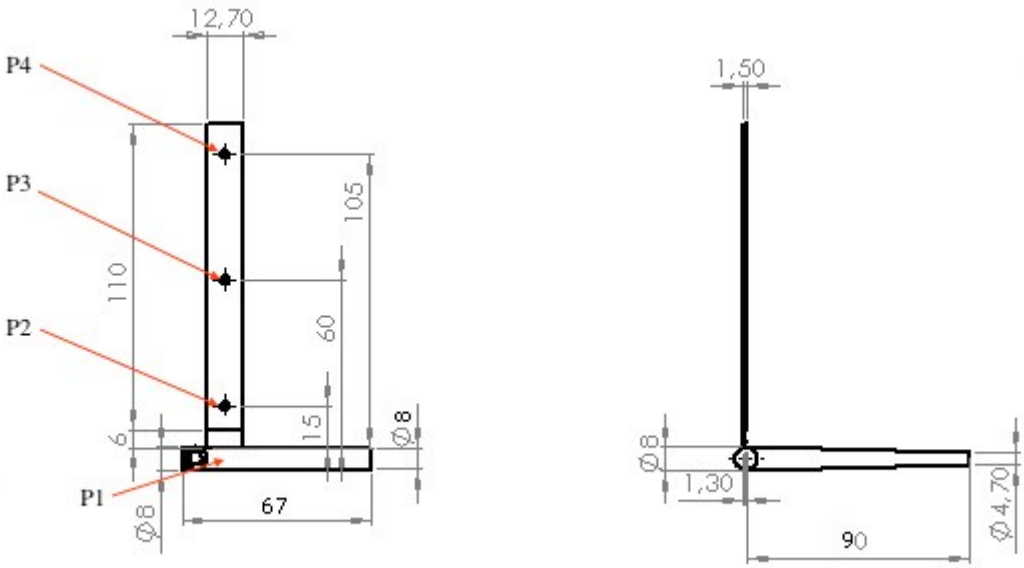

Fig. S1 Sketch of cavity geometry adopted in this work.

Five piezoelectric pressure transducers were located along the polymer flow path: one in the injector (P0), one just before the gate (P1) and three in the cavity (P2, P3 and P4) at 15 mm, 60 mm and 105 mm downstream from the gate position, respectively. A fast data acquisition system

(Kistler, DataFlow, acquisition frequency of 100 Hz) was adopted for collecting pressure and temperature evolution data.

Two multilayers heating devices, electrically connected in series, were located just downstream from the gate on each side of the cavity. The heating device consists of a CB-PAI film of about 50 μm sandwiched between two electrical isolating PAI layers, of 20 μm thickness. An additional thermal insulation layer, of 120 μm, was inserted between each heating device and the mold surface in order to reduce the heat dissipation toward the mold during the heating stage. A steel layer of 100 μm thickness protects the heating device from the molten polymer. An assembling scheme of the heating device is shown in Fig S2.

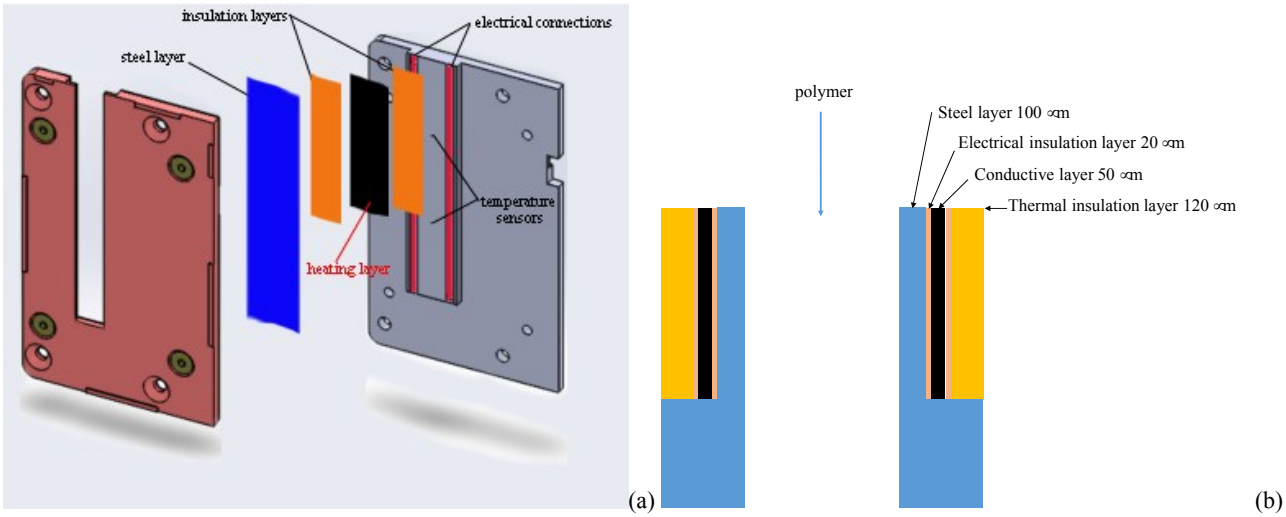

Fig. S2 (a) Assembling scheme of heating device; (b) scheme of assembled heating device

The heating devices adopted were 70 mm length and cover the whole cavity width. Fig S2a shows its position in the cavity. Temperature evolution was detected with a thin thermocouple probe (type T) on the steel layer, which gets in contact with the polymer while it fills the cavity.

Several preliminary tests were carried out in order to correlate the electric power supplied to the heating device with the asymptotic temperature reached on the surface. In this way, it was possible to set the temperature level without need of a controller.

An example of the temperature kinetics response of the sole mold/heating device (with an empty mold cavity) to an electrical pulse is reported in Fig. S3. As evident in the figure, the surface

temperature of the heater follows quite quickly the step change of the electric power supply. The time necessary to cool down the surface is higher than that necessary to heat it up. This is also dependent on the actual asymmetric configuration of the heater device. Indeed, by changing the thickness of the thermal insulation layer on the mold side and the power supplied, it is possible to reduce the cooling time without changing the temperature level reached.

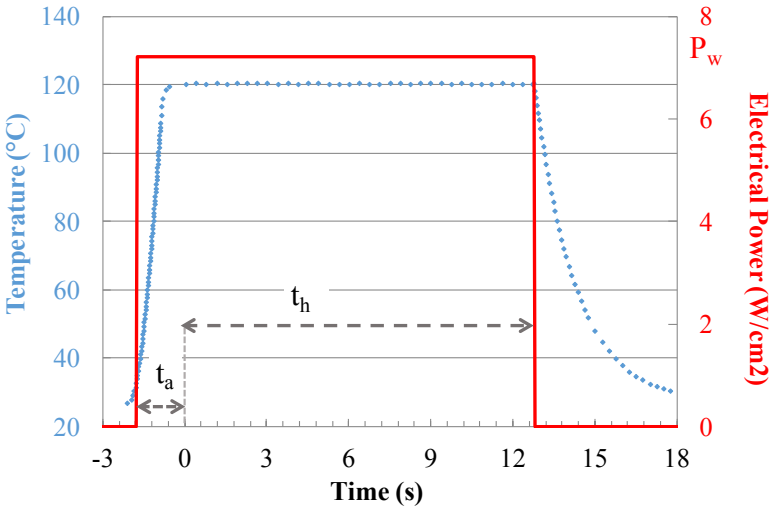

Fig. S3 Temperature evolution on the steel layer due to the activation of the heating device with a power density of 7.3 W/cm^2 (t_a=time that the heater is activated before polymer entrance into the cavity; t_h=time that the heater is held active after polymer entrance into the cavity).

All the injection molding experiments were carried out with an average volumetric flow rate of 2.8 cm^{3}s^{-1} (thus the cavity filling time was about 0.8 s); a melt injection temperature (T_i) of 220°C and a mold temperature of 25°C were adopted. Two holding pressures (P_{hold}) were adopted: 360 bar and 720 bar. A holding time of 9s after filling of the cavity was selected. For all tests, the heater was activated 2 second before the polymer reached the position P2; afterwards, it was kept active with the same power for additional heating times, t_h of 0.8, 1.3, 6, 12 and 20s. Three different levels of the electrical power were supplied to the heating device, 5, 7 and 9.5 W/cm^2, corresponding to three different asymptotic temperature levels (T_{level}), 90, 120 and 150°C, respectively.

The name of each sample tests includes sequentially the holding pressure ("H" for high holding pressure, 720bar, and "L" for low holding pressure, 360bar), the asymptotic temperature measured on the steel layer (T_{level}) and the heating time t_h (after the 2s of pre-heating).

Additional non-conditioned experiments were carried out for comparison. In particular, the P720 and P360 (Passive) experiments were carried out without activating the heater, whereas S720 and S360 (Steel) samples were carried out by replacing the heating device with a steel layer. Table S1 summarizes the operative conditions adopted for the injection molding experiments.

Table S1 Injection molding operating conditions. (Pw=electrical power density; T_{level}= temperature reached on the steel layer; t_h=time that the heater is activated after the polymer reaches position P2. t_a = time that the heater was activated before the contact with the filling polymer in P2: P_{hold}=holding pressure).

Test run	*Pw (W/cm²)*	*T_{level} (°C)*	*t_h (s)*	*t_a (s)*	*P_{hold}*
H-150.0	9.5	150	0.8	2	720
H-150.1	9.5	150	1.3	2	720
H-150.6	9.5	150	6	2	720
H-150.12	9.5	150	12	2	720
H-150.20	9.5	150	21	2	720
H-90.12	5	90	12	2	720
H-120.1	7	120	1.3	2	720
H-120.6	7	120	6	2	720
H-120.12	7	120	12	2	720
L-150.1	9.5	150	1.3	2	360
L-150.6	9.5	150	6	2	360
L-150.12	9.5	150	12	2	360
L-150.20	9.5	150	21	2	360
P360	0	25	0	0	720
P720	0	25	0	0	360
S360	0	25	0	0	720
S720	0	25	0	0	360

Morphological investigations

For morphology investigations, thin slices were cut out from molded samples by means of a Leica slit microtome, at about 15 mm downstream from the gate (in position P2) as illustrated in Fig. S4: the slices were cut from the central part of the molded slab along flow direction, parallel to the flow-thickness plane.

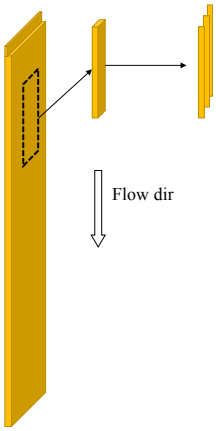

Fig. S4 Cutting procedures of slices from molded samples for morphological investigations.

Two optical images of the cross section in position P2 were taken: (i) with the slices oriented along the polarizer direction; (ii) with the slices rotated of 45° with respect to the polarizer direction. The change of brightness during a 45° rotation is generally proportional to the molecular orientation of the polymer.

In order to better characterize morphological distribution in injection molded samples, some of the slices were chemically etched according to the procedure reported by Bassett[29] and then observed by AFM. The etchant used was a solution of potassium permanganate in a mixture of 10:4:1 volume of concentrated sulphuric acid, orthophosphoric acid and distilled water, respectively (1 g of potassium permanganate in 100 ml of mixture). A 2 h period of etching at room temperature was generally sufficient to reveal the surface topography. AFM investigations were conducted in air and at room temperature with a Bruker Dimension instrument coupled with a Nanoscope V controller operating in tapping mode. A commercial probe tips with nominal spring constants of 42 N m^{-1}, resonance frequencies of 300 kHz, and tip radius of 7 nm were used.

Wide-angle X-ray diffraction patterns, with nickel filtered CuKa radiation, were obtained, in reflection mode from the sample surfaces, with an automatic Bruker D8 Advance diffractometer. X-ray patterns were analyzed by a deconvolution procedure performed according to a scheme reported in the literature[30] and summarized below. The full spectrum is considered as a

superposition of a number of peaks; each peak being described by a combination of a Lorentzian function and a Gaussian function.

Temperature evolution

Fig. S5 shows three examples of cavity surface temperature evolutions during injection molding experiments. As mentioned before, such results were obtained supplying three different electrical power densities to the heating device: 5, 7 and 9.5 W/cm^2. In Fig. S5, as in all the subsequent figures, t=0 corresponds to the time of the first contact of the polymer with position P2.

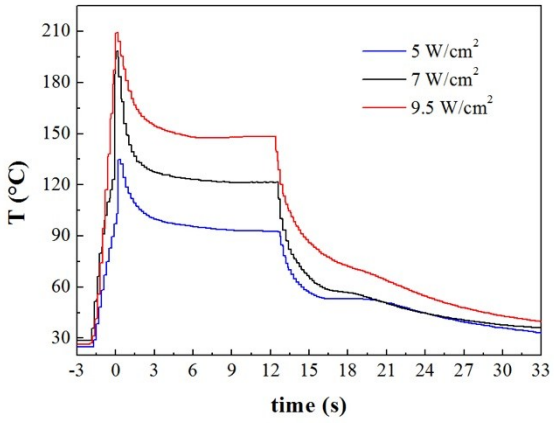

Fig. S5 Temperature evolutions obtained keeping active the heater device for 12 s after the first contact of the polymer in pos. P2 at different electrical power densities, 5, 6 and 9.5 W/cm^2.

Pressure evolutions

In Fig. S6, pressure evolutions measured in different positions along the flow path, for the non-conditioned Passive test P360, are reported.

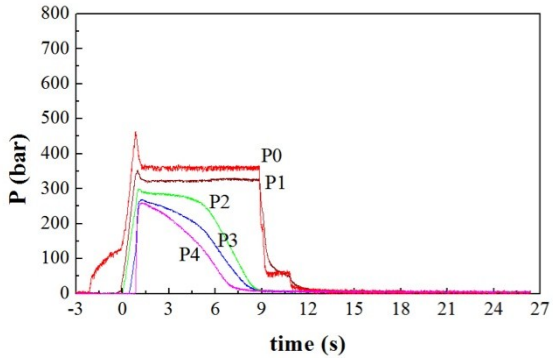

Fig. S6 Pressure evolutions for the experiments P360 (360bar as holding pressure), performed using the heating device only as an insulating layer. t=0 s is the time at which polymer melt reaches position P2.