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A two-colour optical system for the on-line characterization of extruded-blown polymer microfibres

A Bendada

National Research Council, 75 De Mortagne Boulevard, Boucherville, Quebec J4B 6Y4, Canada

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Abstract
We describe a novel two-colour optical system that we have developed for the on-line measurement of the temperature profiles of extruded-blown polyester microfibres. The key feature of the developed system is the use of two specific wavelengths that correspond to fundamental absorption bands of polyester. The two wavelengths are located in the far-infrared region, which allows accurate low-temperature measurements. Moreover, they are quite spaced out apart, which allows the achievement of a very good temperature resolution. A particular characteristic of the new optical device is the use of mirror reflectors as collection optics to overcome problems of optical chromatic aberrations. On-line experimental data obtained from trials carried out on an industrial-scale mutihole extrusion die are presented and discussed.

Keywords: plastics processing, instrumentation, optical sensing

1. Introduction

Extrusion-blowing is a commercially important way of forming fibres from thermoplastic polymers. The detailed description of the process has been presented elsewhere [1–3] and only a brief account is given here. In the process, a stream of hot polymer is extruded through numerous small orifices in a linear die into convergent streams of hot air. The force of the hot air upon the polymer results in the rapid attenuation of the polymer into very small-diameter fibres. The fibre diameter can be as low as 5 μm. Figure 1 shows a schematic diagram of the extrusion-blowing process.

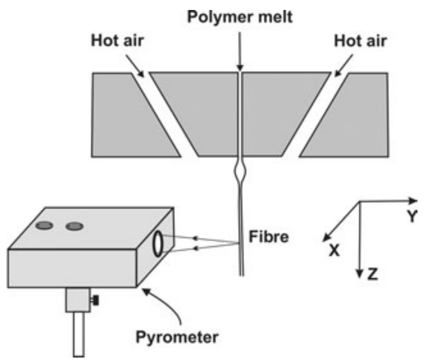

Figure 1. Cross-section of the extrusion-blowing die with the two-colour pyrometer. The z-direction corresponds to the main axis of fibre motion, while the x-direction represents the transverse axis parallel to the holes' alignment.

Because of the commercial importance of extrusion-blowing [4], the process has been of great scientific interest. To obtain a better understanding of the process, researchers have taken both on-line and off-line measurements of structure development during extrusion-blowing [5–8]. The viscosity of polymers and their speed of crystallization are features extremely sensitive to temperature. It is therefore necessary to control the cooling profile of the fibres during the manufacturing process in order to obtain a product of constant and uniform quality. The temperature of the fibres being extruded is difficult to measure because the fibres are particularly small, semi-transparent, moving and vibrating at high frequencies. Optical techniques such as infrared pyrometry and thermography exhibit a determining advantage in such processes where a vein of a polymer melt flows down in the air and cannot tolerate contact measurements that would lead to breaking the fibre stream.

In extrusion-blowing, some researchers have already tried infrared thermography to monitor the temperature of the fibres [9–12]. But the measurement of the true fibre temperature via thermography is not an easy task. This is due to the limited space resolution of the infrared cameras and the complex behaviour pattern of the polymer emissivity [13]. Even by using a camera with a very competitive space resolution and by adjusting the maximum optical magnification that is compatible with the process, the image of a fibre only a few microns in diameter cannot completely cover the photon detector of the camera. On the other hand, thermography needs the emissivity of the polymer fibres to be known in its spectral bandwidth and assumes an opaque grey-body behaviour for the polymer. In reality, the energy emitted by a polymer material is not evenly distributed in the infrared spectrum. Polymers are semi-transparent and usually exhibit few narrow, strong absorption bands created by the vibrations of particular chemical bonds [14]. From an infrared radiometric point of view, this represents special regions where thin plastic fibres are opaque and radiation is emitted only from the surface. Using an infrared detector with selective narrow-band filters in these regions, it is possible to accurately measure the fibre temperature rather than a combination of the fibre and background temperatures [15–17].

The current work describes a new on-line optical technique for the measurement of temperature profiles in extrusion-blowing of polyester. As previously mentioned, polymer transparency and background radiation may be overcome by using absorbance regions of the polyester spectrum. Polyester has few absorbance peaks that can be potential candidates for radiometry applications. On the other hand, smallness and movement of the fibres obviate the use of single-band pyrometry because, once the pyrometer has been focused so that the fibres fill its field of view, vibration may cause the fibres to be out of the field of view some of the time and their number to change continuously with time. One solution is to focus so that the same number of fibres is always in the field of view so that the signal from the fibres remains constant, no matter where they are in the field of view. However, now the relationship between signal and temperature must be determined for each target size, in other words for each fibre diameter, which is unknown, and changing with time. Proper use of two-colour pyrometry overcomes the latter problem. The ratio of radiances received by the pyrometer is independent of target size and of the position of the target within the pyrometer field of view [18]. The articles by Moreau *et al* [19, 20] are a typical illustration of the aforementioned technique for small, moving and opaque targets. The authors developed a two-colour device to monitor the cooling rate of typically small in-flight metallic particles after crashing into a substrate. For calibration, they used a tungsten ribbon lamp as a reference temperature source. They thoroughly described how they introduced a correction factor into the calibration curve to take into account the non-grey behaviour of the impacting particles and the tungsten ribbon. Since they used two relatively close short wavelengths ($\lambda_2 - \lambda_1 \approx 200$ nm), they assumed the correction factor to be roughly constant through the temperature range of the particle cooling process. Other researchers, namely Borca-Tasciuc and Chen [21], investigated theoretically the applicability of two-colour radiometry to transparent silica fibres in the fibre drawing process. This is very similar to the aim of our work. These researchers first developed a photothermal radiometry technique (PRT) that combined the use of an excitation laser and a two-colour system to measure the temperature of metallic vibrating wires. They used two quite separated wavelengths ($\lambda_1 - \lambda_2 \approx 1.3$ μm) in the mid-infrared region, which led to good temperature sensitivity. Then they analysed via Mie scattering theory the possibility of extending the technique to optical fibres [22, 23]. Calculation results suggested that two-colour radiometry could be used to measure the temperature of optical fibres by choosing an appropriate excitation laser and the two wavelengths of the ratio radiometer.

2. Pyrometer design

Standard two-colour thermometry measures the radiated energy of an object in two narrow wavelength bands and calculates the ratio of the two energies, which is a function of the temperature of the object. The temperature measurement is thus dependent only on the ratio of the two energies and not on their absolute values. Any parameter, such as target size, that affects the amount of energy in each band by an equal percentage has no effect on the temperature indication. This makes a two-colour thermometer inherently more accurate. However, there are some conditions that have to be considered when using two-colour thermometers to measure temperature. These conditions include stray reflections off the target and background radiation when the target does not fill the entire field of view of the thermometer. This is typical of a target which is completely surrounded by a hot background. The two-colour pyrometer will measure the composite radiant signals streaming from the target surface for each spectral channel, compute the ratio of these two signals, and display the temperature equivalent of this ratio. Unfortunately, although quite naturally, this indicated temperature is neither that of the target nor that of the background, but rather a combination of the mixed signals. Even if the ratio of two emissivities at the two employed wavelengths is known, it cannot correct for these conditions (i.e. reflected and directly emitted energies from the background). These can be corrected only by removing the interfering hot background. But none of these conditions are problems if the background is cool. Nevertheless, a question arises: for a given target, below what temperature can the background be considered cool? This issue has been taken into account during the calibration procedure of the pyrometer described hereafter.

Wavelength pairs for ratio thermometry are chosen for the following reasons:

(1) the ratio of signals in the selected wavelength pair is sensitive to temperature changes,
(2) some knowledge of the emissivity value in each wavelength region is available, and
(3) values are known for the relative absorptions of intervening media in each wavelength region.

Although the principles of ratio thermometry may be applied over any wavelength region, measurements are largely limited to few wavelength regions for polyester. Polyester is a semi-transparent material that exhibits only few narrow

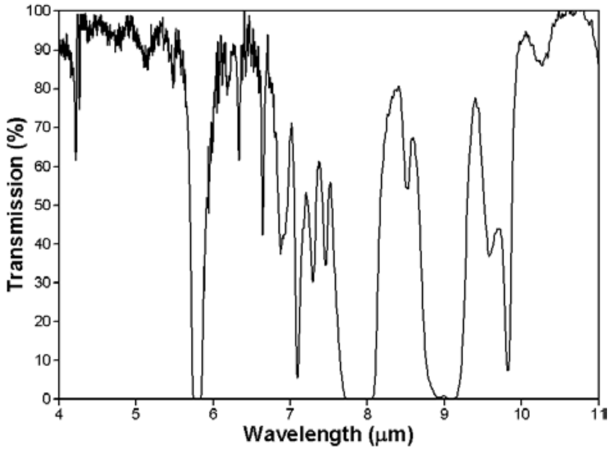

Figure 2. Transmission spectrum of a polyester film 10 μm thick.

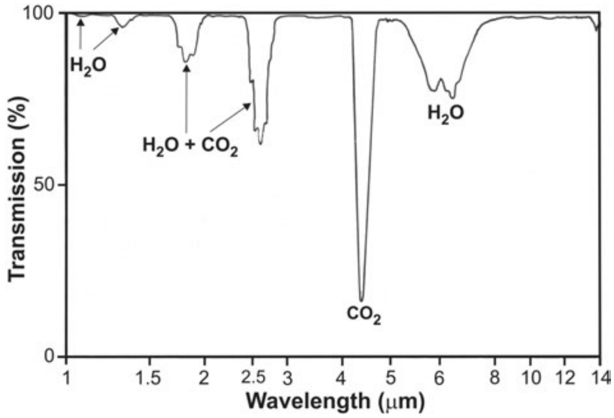

Figure 3. Infrared transmission through 1 m of air at standard conditions.

absorption bands that can be used for accurate radiation thermometry. Figure 2 shows a typical transmission spectrum of a polyester film 10 μm thick as recorded with a standard laboratory infrared spectrometer. We may observe here that the thickness of the latter specimen corresponds to the smallest diameter of the manufactured fibres as observed with scanning electron microscopy (SEM). From figure 2, it can be clearly seen that the main absorption bands that cause internal transmission to be zero ($\tau = 0$) are 5.80, 7.95 and 9.01 μm. It is worth noting that these absorbance peaks are not sensitive to polymer temperature and crystallinity changes, or to the stretching direction of the polymer fibres [24–28]. On the other hand, the reflectance at the air/polymer interface is typically 3% ($\rho = 0.03$) for most plastics throughout the infrared spectral region [14, 29]. This is not precisely true but it is close enough for our purpose here. As a consequence of the previous observations and according to Kirchhoff's law and the conservation of radiant energy considerations [30], the emissivity ($\varepsilon = 1 - \tau - \rho = 1 - 0 - 0.03 = 0.97$) values at either wavelength can be considered roughly the same. A grey-body assumption (i.e. the emissivity ratio is one) is therefore reasonable in this case.

Furthermore, it should be mentioned that the above three wavelengths were selected assuming perfect transmission of the infrared radiation through air. The infrared transmission through 1 m of air at 20 °C and 55% relative humidity is shown in figure 3 [31]. As we can see, absorption by water and carbon dioxide molecules cannot be neglected even at such a close distance for certain spectral bands. The 5.80 μm spectral band must then be avoided since it is sensitive to atmospheric moisture that would attenuate energy coming from the target and affect the accuracy and repeatability of the infrared instrument. Finally, the 'optimal' wavelength pair suitable for ratio thermometry of polyester is the 7.95/9.01 μm pair. We should point out here that the location of the remaining wavelength pair in the far infrared (i.e. near the Planck law maximum for low-temperature targets [30]) is a relevant factor which could lead to a significant improvement of the signal-to-noise ratio when radiation signals are expected to be weak. This is the case here for the small-diameter fibre stream whose temperature is expected to be close to the environment temperature. However, the long-wavelength pair would normally exhibit in the same time the drawback of providing lower sensitivity to temperature changes. Indeed, temperature resolution would be better if both wavelengths were on the short-wavelength side of the blackbody radiation curve. On the other hand, we may also mention that the loss of temperature resolution is to a certain extent compensated for the large separation between the two selected wavelengths ($\lambda_2 - \lambda_1 \approx 1$ μm).

After the determination of the wavelength pair suitable for the application of two-colour thermometry to extrusion-blowing of polyester, a pyrometer has been devised according to a conventionally employed design [32]. Zinc sulfide (ZnS) lenses were used to collect the infrared radiation emitted from the hot target and imaged it onto two 1 mm × 1 mm HgCdTe detectors, cooled with liquid nitrogen. This kind of cooling is somewhat impractical for industrial applications but it has the advantage of providing very high detectivities. We might mention also that liquid nitrogen exhibits a cooling autonomy of around 8 h, which actually makes the use of such a mode of cooling not so unpleasant. The detectors, which were specially designed to meet the needs of the current application, exhibited high D* peak values at 8 and 9 μm wavelengths ($>6 \times 10^{10}$ cm Hz$^{1/2}$ W^{-1}), respectively, and responsivities larger than 8000 V W^{-1}. This provided a very good signal-to-noise ratio over the required temperature excursion range in extrusion-blowing. One can assume, for this kind of application, that the polymer fibre temperature is mainly between 30 and 300 °C. A dichroic beam splitter divided up the collected radiation into the two detectors. In order to select the proper wavelength pair needed for polyester, narrow-band filters at 7.95 ± 0.10 and 9.01 ± 0.10 μm, respectively, were incorporated in front of the appropriate detectors. A mechanical chopper is suitable for getting rid of the background noise.

To calibrate the thermometer and investigate its thermal resolution on a controlled-temperature fibre, we simulated the fibre with a 200 μm diameter pinhole located in front of a Mikron M315 blackbody source. According to the optical design employed, the pinhole was equivalent to a 10 μm diameter fibre located at the centre of the measuring focal spot of the pyrometer. The pinhole was used for three purposes: firstly, to analyse the signal-to-noise ratio for both detector channels, particularly in the low-temperature range; secondly,

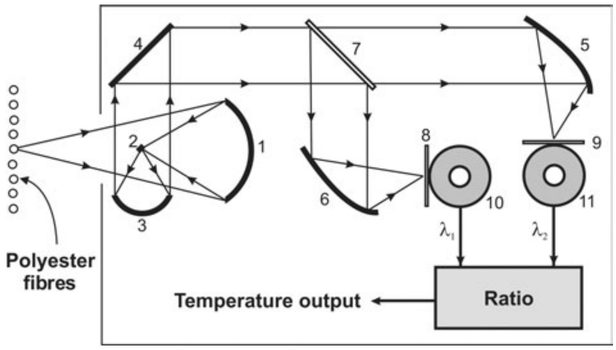

Figure 4. Schematic of the optical system where 1–6 are the optical reflectors, 7 is the dichroic beam splitter, 8 and 9 the narrow-band-pass filters, and 10 and 11 are the infrared detectors.

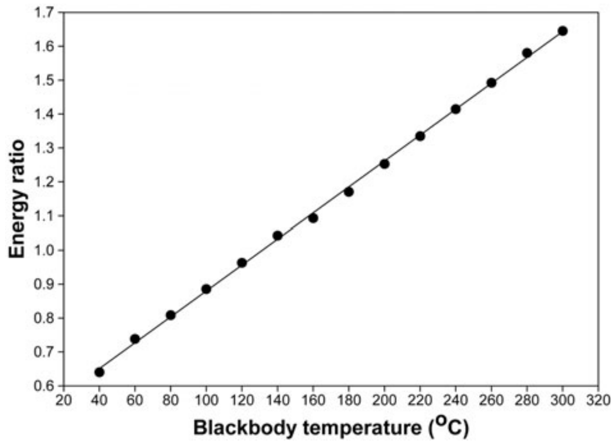

Figure 5. Plot of the pyrometer ratio output versus a blackbody source temperature. The circles represent the measured data, while the solid line shows the calibration curve.

to find out the suitable gain adjustment for the detectors and following electronics; and thirdly, to determine below what critical target temperature the background radiation could not be neglected. The last procedure consisted simply in comparing for each blackbody temperature the energy ratio computed when the pinhole was employed in the calibration to the energy ratio obtained without the pinhole. The critical target temperature below which a temperature averaging effect would occur was estimated when the two energy ratio versus temperature curves diverged. The blackbody source temperature could be set at any temperature between 10 and 500 °C. Once set, the source accuracy was within ±0.25% of the Celsius temperature reading. A resonant fork chopper was installed in front of the pinhole to overcome the background noise and to simulate the oscillating motion of the fibres in the real process. The experimental results showed that small changes in temperature resulted in quite large changes in the energy ratio; the temperature resolution was good. The energy ratio was defined as the energy detected at the 7.95 ± 0.10 μm wavelength band divided by the energy detected at the 9.01 ± 0.10 μm wavelength band. However, we observed important chromatic aberrations caused by the lens optics when the pinhole was moved slightly away from the focal plane [18, 31]. In other words, when the pinhole was moved out of focus, energy ratio changes unrelated to a true temperature change occurred. This was fairly problematical since polyester fibres in the extrusion-blowing process vibrate back and forth across the focal plane. Chromatic aberrations are due to the fact that the refractive indices of materials used for lenses (in this case ZnS) are spectrally dependent. This means that the different wavelength components of the energy emitted by the hot target will be focused differently by lenses. In turn, the target size imaged onto a radiation detector using lens optics will be different for different wavelengths. For two-colour thermometry, this will result in a change in the ratio when the target size changes or moves along the optical field of view of the pyrometer.

The solution to the chromatic aberrations issue is to make sure that the optics employed are achromatic, i.e. not wavelength dependent. Achromatic lenses can be constructed but these lenses are perfectly achromatic for only a few narrow bands. Thus, for ratio thermometry, each time the use of a different pair of wavelengths is desired, a new lens system must be chosen and only narrow wavelength bands may be used. To provide some flexibility in the choice of the wavelength pair that may be required for polymers other than polyester, the obvious and simple solution is to use mirror optics since they are inherently achromatic.

A new optical design of the pyrometer was performed this time by using spherical concave, off-axis paraboloidal and flat aluminium mirrors. Figure 4 shows the new thermometer architecture and the details of the mirror optics design. The optical assembly had a focal plane located at a working distance of 180 mm from the end of the pyrometer metallic casing and a numerical aperture of 0.12. The size of the measuring focal spot at the latter working distance was ∼3 mm in diameter. To protect the optics and the electronic components from the overheating caused by the convergent hot air streams launched from the linear die, a flow of cold compressed air was injected inside the pyrometer casing. To feed the detectors with liquid nitrogen during operation, circular windows were machined on the top of the pyrometer casing above each detector. Once the filling of liquid nitrogen was completed, metallic caps were used to close the access to the detector Dewars (figure 1).

The newly designed thermometer was then calibrated according to the same procedure used for the lens optics system. Figure 5 shows the calibration curve, which is the evolution of the energy ratio versus blackbody temperature. The blackbody temperature was changed from 30 up to 300 °C, which was the interval of concern for the polyester fibres in the extrusion-blowing process. A comparison of calibration curves obtained with and without the insertion of the pinhole into the optical path of the pyrometer was carried out. It revealed that the critical target temperature below which the instrument would indicate an incorrect temperature reading was around 50 °C. Below this temperature an averaging effect would occur resulting in underestimated temperatures. Nevertheless, it is worth noting that the temperature range that is of interest is beyond the glass transition temperature which is about 73 °C for polyester [33]. From this point of view, although the developed pyrometer was not so effective below 50 °C, this would not affect its usefulness for the extrusion-blowing process.

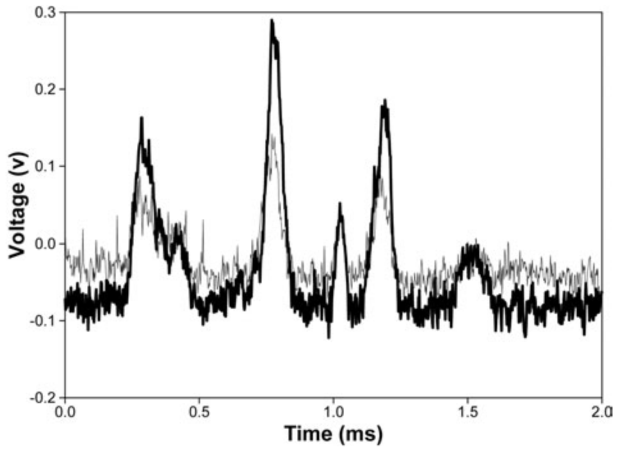

Figure 6. Typical infrared raw signals acquired at the two wavelengths at a distance of 340 mm from the die. The thin solid curve corresponds to the 7.95 μm wavelength, and the thick solid curve to the 9.01 μm wavelength. The ratio of the RMS values of the two signals is 0.69, which according to the calibration curve reported in figure 5 leads to a fibre temperature of $\sim$50 °C.

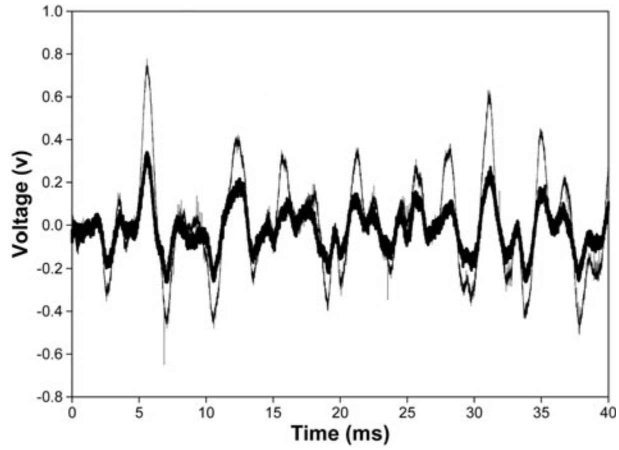

Figure 7. Infrared raw signals acquired at the two wavelengths at a distance of 5 mm from the die. Similarly to figure 5, this time the calculated ratio is 1.68, which leads to a fibre temperature of 308 °C.

3. Experiments and results

After the successful experiments carried out to investigate the temperature resolution of the newly devised pyrometer, on-line trials on an industrial-scale extrusion-blowing machine were undertaken to prove the pyrometer in real operating conditions. The experiments were performed with a 150-hole vertical die nosepiece. The holes were 0.4 mm in diameter and spaced at 1 mm. The polymer melt was extruded from these holes to form filament stands that were subsequently attenuated by high-velocity hot air, forming very fine fibres. The polymer used in these experiments was WTE E-842 polyester. The melt temperature inside the extruder was 310 °C while its pressure was 2.65 MPa. After exiting the extruder, the polymer was fed to a metering pump that in turn fed the die nosepiece at a controlled flow rate of 6 kg h^{-1}, which resulted in a flow rate per hole of 0.5 g min^{-1}. During processing, the whole die assembly was heated section-wise using cartridges to attain a desired processing temperature of 310 °C. The high-velocity air was generated using an air compressor. The compressed air was passed through a heat exchanger to heat the air to the desired processing temperature of 310 °C. The heated air was then conveyed to the air manifolds and exited from the sides of the die through narrow air gaps, as shown in figure 1. The hot air velocity was 25 m s^{-1} at the die exit. Air temperature and air velocity were measured on-line near the die exit with a multifunction instrument at centreline locations where air temperature and air velocity were maximum.

The infrared pyrometer was mounted on a traverse system that permitted measurements over a range of positions along the threadline. Unlike the calibration procedure, this time the fork chopper, which was employed to overcome the background noise, was removed since the fibres themselves were naturally vibrating across the field of view of the detector. The oscillating thermal radiation emitted from the fibres was collected with the two detectors, then transformed by low-noise preamplifiers into alternative voltage signals. The latter were subsequently recorded via a Nicolet 440 electronic scope at an acquisition rate of 0.5 MHz. Figure 6 shows typical infrared signals captured by the detectors at a height of 340 mm from the die exit. The thin solid curve refers to the signal collected in the 7.95 $\pm$ 0.10 μm wavelength band, while the thick solid curve refers to the signal collected in the 9.01 $\pm$ 0.10 μm wavelength band. The minimum level of the oscillating curves corresponds to the radiation emitted by the ambient environment, while the maximum level represents the radiation emitted by the fibres at the time they crossed the field of view of the detectors. Figure 7 illustrates the equivalent signals that were monitored near the exit of the die at a height of 5 mm. We can clearly observe the lower average magnitude of the signals when compared individually to the previously described signals monitored at 340 mm from the die exit. This was predictable since near the die exit the fibres were hotter and larger than far from the die exit. The reader may also notice the difference between the shapes of the signals monitored at both heights, which reveals the different vibrating behaviour of the fibres near and far from the die exit. The larger the distance from the die exit, the higher is the vibration frequency of the fibres. Similar observations have already been reported elsewhere on research analyses devoted to fibre vibrations and distributions in extrusion-blowing [34–36]. On the other hand, figures 6 and 7 show that the two signals were perfectly synchronized in time, which was an indication that the optical chromatic aberrations problem had been completely solved. The latter observation is also an indication that the fibres were imaged onto both detectors' planes at almost the same locations within sensitive areas having very similar sizes. In other words, the fields of view of both detectors were well superimposed. This is an important feature that radiation ratio thermometry of small moving targets must meet as much as possible. Otherwise, when the target image crosses the edges of the sensitive areas, the signal change rate will not be the same for both detector channels, yielding incorrect energy ratio calculations.

During the experimental trials, measurements were repeated at several other heights in order to obtain a vertical temperature profile. For each height, the root-mean-square (RMS) values of the two signals delivered by the two detectors were calculated over an acquisition period of 1 s.

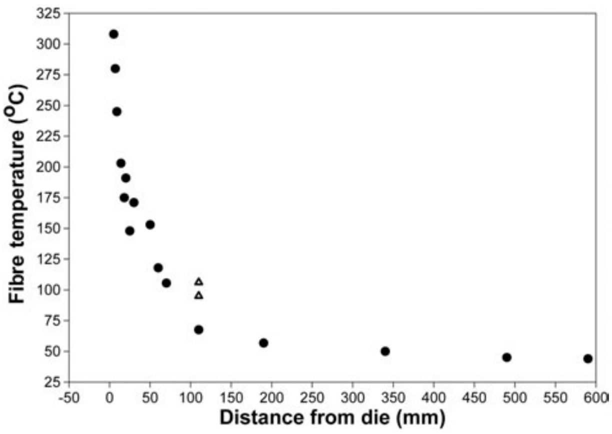

Figure 8. Fibre temperature decay along the vertical centreline (●), and two extra temperatures measured in the x-direction at a distance of 90 mm on both sides of the vertical centreline (△). As indicated in the plot, the latter measurements were performed at a distance of ~100 mm from the die.

Subsequently, the division of the two processed RMS values allowed the determination of the energy ratio. The latter was then introduced into the calibration function to estimate the temperature of the fibres. The circles in figure 8 show the temperature profile; the zero on the abscissa axis refers to the height at the die exit. The reported fibre temperatures are an average of eight different measurements at each particular position. The standard deviation in temperature measurements was found to be 3 °C for measurements close to the die ($z \leqslant 250$ mm) and about 5 °C for measurements away from the die ($z > 250$ mm). The cooling profile evolution when the distance from the die exit was changed from 5 to 500 mm exhibited a very reasonable shape (figure 8). The temperature obtained at a distance of 5 mm beneath the die was very close to the polymer melt temperature, i.e. 308 °C. Far from the die, at a distance of ~500 mm from the die, the fibre temperature caught up the ambient temperature beneath the extrusion-blowing threadline, which was ~35 °C. Moreover, the temperature dropped drastically in the first 80–100 mm beneath the die exit, then the cooling rate became very slow.

After the monitoring of the vertical temperature profile (z-direction in figure 1), two additional measurements were performed to investigate the horizontal profile (x-direction in figure 1) at a height of ~100 mm from the die exit. The two measurements were carried out beneath the extreme sides of the die at a distance of 100 mm on either side of the vertical centreline of the threadline. The two monitored temperatures are reported in figure 8 by the triangles together with the vertical temperature profile previously measured along the centreline of the threadline. The data revealed that the temperature on the extreme sides was higher than in the centre and the horizontal temperature profile (x-direction) was not necessarily symmetrical. This might be the effect of a non-uniform temperature distribution within the die or just the result of chaotic air turbulence along the threadline.

4. Conclusion

An original low-temperature two-colour pyrometer has been developed for on-line temperature monitoring of polyester fibres in an extrusion-blowing process. The combination of the semi-transparency of the polymer and especially the small size of the fibres and their vibration during the process make the use of single-band thermometers or infrared cameras inappropriate. Two-colour thermometry overcomes these latter two problems. The technique uses the ratio of the energies monitored at two distinct wavelengths. The energy ratio is independent of the size and position of the fibres within the pyrometer's field of view. Polyester has only a few absorption wavelength bands that can be used in radiation thermometry. Those wavelengths are not affected by the extrusion-blowing process conditions. Most of them are located in the far-infrared spectral region, which is, according to the Planck law [30], very convenient for low-temperature radiometry applications. This is the case here for extrusion-blowing where fibre temperatures are typically within the 30–300 °C range. To design a two-colour instrument, three absorption wavelength bands were selected in the far-infrared region, namely the 5.80, 7.95 and 9.01 μm wavelengths. The first wavelength, i.e. 5.80 μm, was rejected because it was superposed with a water absorption band whose effect on temperature reading will vary with variations in relative humidity and air-path length. As a consequence, the remaining 7.95–9.01 μm wavelength pair was kept for the final design of the pyrometer. The fact that the two latter wavelengths were quite far from one another (a bandwidth larger than 1 μm separated the two wavelengths) helped to achieve a good temperature resolution for the new instrument. But at the same time, the large bandwidth between the two wavelengths led to drastic optical chromatic aberrations that were observed in a first pyrometer assembled using lens optics. The chromatic aberrations issue was solved by using reflecting mirror optics instead of lens optics. The new pyrometer was first calibrated using a blackbody source and then tested on-line on an industrial extrusion-blowing die. The monitored temperature profiles exhibited reasonable evolutions that were in good agreement with the operating conditions and the scientific literature on extrusion-blowing.

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