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ORIGINAL PAPER

Computer-aided experiment of using real-time small angle light scattering image processing technique for visual characterization flow field of polymer melts

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Abstract The phenomenon of small angle light scattering (SALS) has been applied to the actual extrusion molding process. The current study utilizes a self-designed mold with built-in windows for observation of polymer melts within a slit die. A high-speed charge-coupled-device (CCD) camera is used to record the SALS images in real-time with different process conditions for subsequent analysis. Modification algorithm has been proposed to eliminate the effect of multiple scattering. Flow behavior of polymer melts is simulated and analyzed by real-time SALS image processing technique. Visualization is performed via a high-performance computer-aided analysis software which allows on-line data acquisition and characterization the flow field of polymer melts.

Keywords Small angle light scattering · CCD · Visualization

Introduction

On-line, non-intrusive characterization of the flow field of polymer melts is an essential step towards an implementation of a structural control system that can regulate the structure development during polymer processing. In this study, we present a real-time and non-intrusive, flow field of polymer melts characterization technique that can estimate the variation of the flow field of polymer melts while the process conditions of material is changing.

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The light scattering method is valid for giving information about overall structures, but is not applicable to a structure of large scale compared with the wavelength of the light and is difficult to use for extracting local information on morphology. Recently, a digital image processing technique has shown its utility in analyzing the pattern formation in polymer systems [1].

Light scattering techniques have developed extensively. Small angle light scattering (SALS) [2–6] study provides information on changes of morphology [7]. A study was undertaken to determine the tissue structural information from 2D SALS data [8]. SALS has been used to study micron-sized liquid crystalline droplets [9]. The scatting noise in the reconstructed image can be reduced by phase-conjugated [10]. Both the real and the conoscopic SALS images are used to obtain information on the increase of the band spacing as a function of relaxation time [9]. The quantitative analysis software system for conforms to the digital imaging and communications standard and can be integrated into the picture archiving and communication system [11].

Studies are recommended on how to integrate charge-coupled-device (CCD) camera, digital video processing system, and image analysis and computation software application to build a compact and hand-held instrument [12]. The combination of advances in CCD fabrication, camera design, digital interface technology, and software development has enabled scientific-imaging-device manufacturers to overcome the challenges created by the wide range of requirements [13]. The software kits, which include PCI Device Driver and Image Processing Package, are developed with Visual C++ Language based on Windows OS [14].

In our experiments, device performs real-time image analysis of the evolving light scattering pattern during extrusion. Such an approach provides a means of characterization of the flow field of polymer melts, allowing calculation of light intensity in relation to the molecular orientation of polymer. The experimental device incorporates He–Ne laser generator, optics, a visual slit die, an extruder, a CCD camera, and a personal computer as its major hardware components. Software designed specifically for this application performs real-time analysis of the light scattering pattern. Intensities at various scattering and azimuthal angles are plotted at each time [15].

The purpose of the present work is to apply the optical image technique to the characterization the flow field of polymer melts. In particular, we attempt to on-line analysis the light intensity in relation to molecular orientation of polymer.

Theoretical background

Analysis of variation of polymer melts in different processing conditions effects on light intensity matrix

When a light beam passes through a diffusion surface, the variation of propagation direction of the beam can not be determined by the principle of geometrical optics because of scattering function of light beams on diffusion surface [16].

A transparent fluid is an optical phase object. In the experimental set-up for measuring flow fields in fluid flows by using speckle interferometry, the part of the arrangement for the object light beam is just like a subjective photographic system. Therefore, in general, speckle displacements are generated. The speckle displacements can change the intensity distribution of spatial speckle fields. As a result, the intensity distribution of a speckle interferogram is also changed. In this paper the effect of variation of the intensity is analyzed and discussed in detail. Experimental results are shown. Methods for elimination of the multiple scattering effect are provided. This is advantageous to improve the quality of the speckle interferogram [17].

Small angle light scattering (SALS)

Figure 1 shows a typical set-up for SALS measurement device. A laser light passes through polymer melts in the visual slit die. In order to capture the scattered light, a polarizer and an analyzer are placed before and after the polymer melts. The laser light first passes the polarizer, which removes one orthogonal component of the light [18]. The other component of light passes through the polymer melts with resulting scattering due to the orientation of molecular chain. The analyzer removes the second component since it is placed 90° out of phase with respect to the analyzer. Therefore, any light that comes out of the analyzer is entirely due to the scattering within the polymer melts. In our experiments, the depolarized intensity of light that passes through the polarizer, polymer melts and analyzer is recorded and related to the orientation of polymer melts. A CCD camera captures the image and the total intensity of the image is determined in every 15 s. The total intensity is assumed proportional to orientation of molecular chain.

We assume that each column of the following matrix represents the intensities of one observed Raman spectrum at the selected waveshifts,

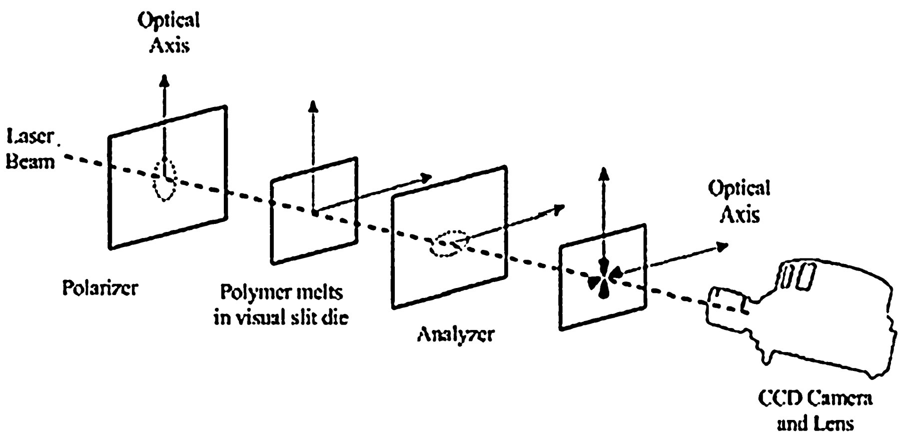

Fig. 1 A typical set-up for SALS

$$D = \begin{bmatrix} d_{1,1} & d_{1,2} & . & d_{1,n} \\ d_{2,1} & d_{2,2} & . & d_{2,n} \\ . & . & . & . \\ d_{m,1} & d_{m,2} & . & d_{m,n} \end{bmatrix}; D \in R^{m \times m} \tag{1}$$

Therefore, each spectrum is represented by (m) number of spectral intensities and a total of (n) spectrum exist. The dispersion matrix Z that represents the variation in the data is computed as [18]

$$Z = D^{\mathrm{T}} D; Z \in R^{m \times m} \tag{2}$$

Modification algorithm for multiple scattering

The diagram of multiple scattering is shown in Fig. 2. According to the effect of the sample on the incident light, the sample can be divided into a surface layer, a first scattering layer, a second scattering layer, etc. Because the incident light first impinges on the surface of the sample the first scattering layer (random reflection) of the medium. The second one comes from the first scattering layer (because of the internal heterogeneity) and third in turn [19].

Because of multiple light scattering caused by the thickness of the sample, the light scattering images will dispersion and distortion. A distortion model is constructed, and a correcting factor is introduced. Computer simulation verifies that, under some factual circumstance. Introducing of the correcting factor improves the precision and the reliability.

The measurable intensity of scattered light $I_s^{'}$ and the factual intensity of scatted light I_s have the relationship as follow.

$$I_s = K_f I_s^{\prime} \tag{3}$$

where K_f is the correcting factor can be written as

$$K_f = e^{\left(\frac{\tau d}{\cos\varphi}\right)} \tau d\left(\cos^{-1}\varphi - 1\right)\left\{e^{\left[\tau d\left(\cos^{-1}\varphi - 1\right) - 1\right]}\right\}^{-1} \tag{4}$$

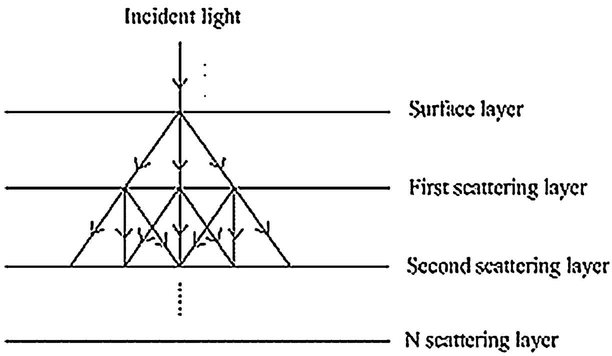

Fig. 2 Diagram of multiple scattering

where φ is the scattering angle, τ is the turbidity of the sample, and d is the thickness of the sample [20, 21].

Experimental set-up

Visual die used

The channel of slit die used for the experiment is two dimensional as shown in Fig. 3. It is characterized by its length W, gap 2H and depth L ($L/W \gg 10$). The channel surfaces were smooth steel surfaces. Observations and light scattering measurements were performed through quartz glass windows.

Transparent fluids used

The results reported in this study were obtained with high-density polyethylene (HDPE) and polystyrene (PS). They are both transparent, which is necessary to perform visualization experiments. As they are commercial polymers that melt at high temperatures, they enabled the study to be performed under quasi-industrial conditions.

Optic

A He–Ne laser is used as an incident light. Optical system and the polarization analyzer is detected by a CCD connected to a computer.

Experimental procedures

Table 1 shows the experimental conditions. We use two kind of materials (PS and HDPE), three kind of rotate speed (20, 24, and 32 rpm), five kind of vibration amplitude (0.04, 0.08, 0.12, 0.16, and 0.20 mm), and five kind of vibration

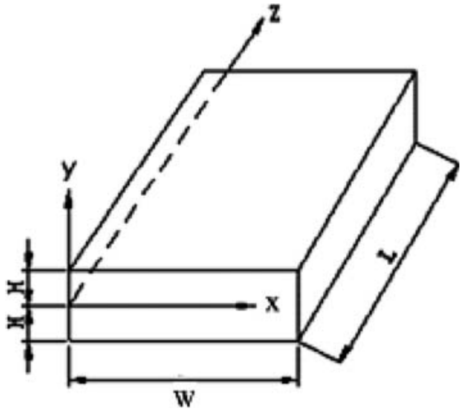

Fig. 3 Channel of slit die

Table 1 Experimental conditions

Material	Rotate speed (rpm)	Vibration amplitude (mm)	Vibration frequency (Hz)
PS	20	0.04	5
HDPE	24	0.08	8
	32	0.12	10
		0.16	12
		0.20	15

frequency (5, 8, 10, 12, and 15 Hz). In the experiments, first the screw of the extruder rotates at constant speed. Second, we change the amplitude and frequency of the screw, respectively. At the same time, a CCD camera captures the light scattering image of each processing condition. Finally, the digital image analysis software real-time characterizes the flow field of polymer melts.

Experimental characterization flow field of polymer melts

On-line data acquisition

This paper presents an effective method of recording the light scattering image and spatial shape of light scattering intensity. In this method,the light scattering images are acquired with a CCD.

After digital image processing, the spatial curves representation the light intensity distribution can be drawn by the real-time light scattering analysis software. In order to improve automatic degree of data acquisition, the real-time communication between computer and high-performance CCD can be realized with the method and functions under Delphi7.0. RS-232 standard protocol is used in the communication between computer and high-performance CCD, and automatic data acquisition is accomplished by computer and CCD.

Figure 4a shows light scattering image of HDPE at 24 rpm screw rotate speed without vibration. Figure 4b shows light scattering image of HDPE at the same rotate speed with vibration frequency 10 Hz and vibration amplitude 0.20 mm. Their 3D light intensity images are shown in Fig. 5a and b. Form Fig. 4a and b, we can see that light scattering image with vibration is brighter than image with out vibration. In comparison with 3D light image without vibration, 3D image with vibration has stronger light intensity. This is the reason why the light image become brighter as shown in Fig. 4a and b. It is also illustrated that the orientation of molecular chain increases because light intensity is proportional to orientation of molecular chain.

On-line characterization and analysis flow field of polymer melts

On-line characterization the variation of flow field of PS with the same vibration amplitude and different frequency.

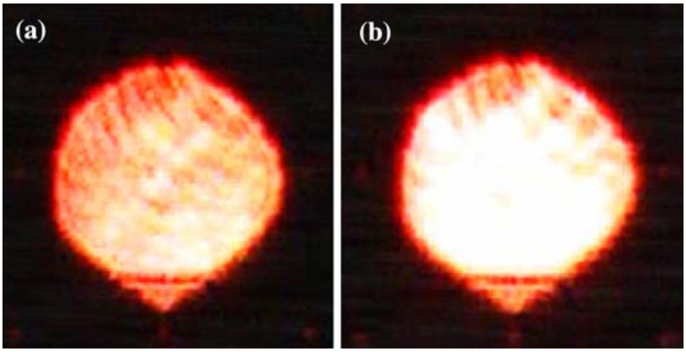

Fig. 4 The light scattering images of HDPE at 24 rpm screw rotate speed. **a** Without vibration, and **b** with vibration frequency 10 Hz and vibration amplitude 0.20 mm

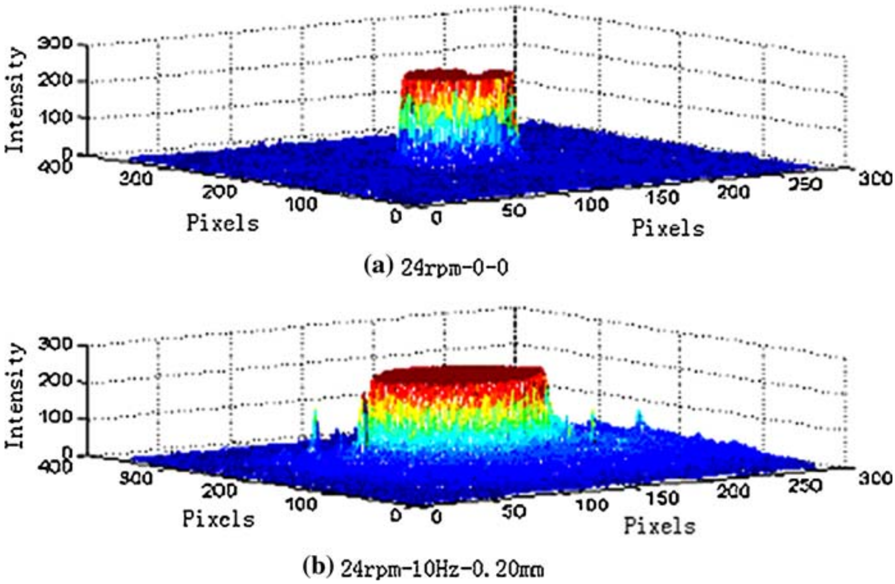

Fig. 5 3D light intensity compare images of HDPE at 24 rpm screw rotate speed. **a** Without vibration, and **b** with vibration frequency 10 Hz and vibration amplitude 0.20 mm

Figure 6 shows the maximum intensity projection area compare images of PS at 18 rpm screw rotate speed. Figure 6a shows the image without vibration. Figure 6b–f show the images of at the same vibration amplitude of 0.16 mm and different vibration frequency of 5, 8, 10, 12 and 15 Hz, respectively. Figure 7 shows the variation trend of maximum intensity projection area with the increase of vibration frequency. As shown in Figs. 6 and 7, with the increase of vibration frequency, the maximum intensity projection area becomes larger. This is because with the increase of vibration frequency, the molecular orientation of polymer melts

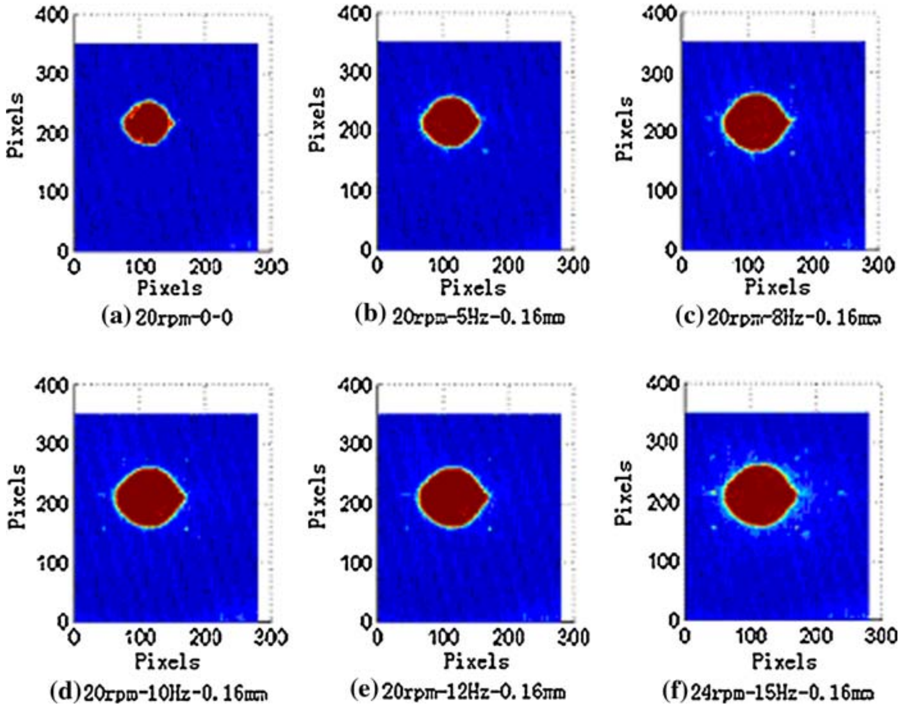

Fig. 6 Maximum intensity projection area compare images of PS at 20 rpm screw rotate speed. **a** Without vibration. With the same vibration amplitude of 0.16 mm and different vibration frequency: **b** 5 Hz, **c** 8 Hz, **d** 10 Hz, **d** 12 Hz, and **f** 15 Hz

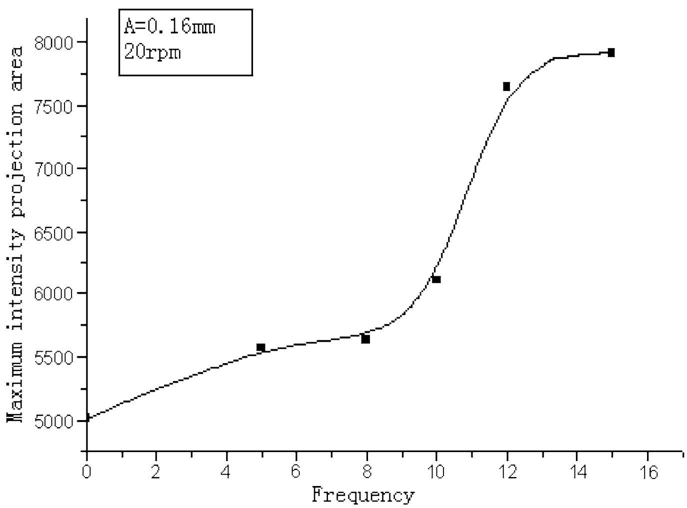

Fig. 7 The relationship between maximum intensity projection area and vibration frequency of PS (rotate speed: 20 rpm, vibration amplitude: 0.16 mm, different vibration frequency)

also increases. As a consequence, the light intensity becomes stronger and the light scatting image becomes brighter.

Conclusions

We presented a new technique that can be used to characterize the flow field of polymer melts on-line and non-intrusively while the material is processing with different conditions. The technique is based on SALS and digital image on-line analysis software. It is shown that the proposed technique is easy to implement and provides more flexibility approximating the non-linear relation between the maximum intensity area and the corresponding vibration intensity (frequency or amplitude). This approach was validated in the case of transparent polymer melts in the visual die. Applying this method to character the flow field of polymer melts will be of great interest, since it will contribute information on optical prosperities have been proven to be useful for obtaining deep insights into the molecular and structural parameters of polymers.

The results can be summarized as follows

a. The scattered laser light can be used to characterize the flow field of the polymer melts in the multiple scattering regime.
b. The optical image processing technique to characterization the variation of polymer melts with the increase of vibration intensity (including frequency and amplitude) can be shown as: the light scattering image becomes brighter, the light intensity matrix become stronger, and the maximum intensity become larger.
c. The experimentally obtained datum are in good accord with the theory of electromagnetism dynamic extrusion [22, 23]. The increase of orientation of polymer melts can change the flow behavior. Through the experimental research it was proved that inducing of vibration field increased the orientation of polymer melts, because the light intensity is proportional to orientation of molecular chain.

In conclusion, this technique is believed to be important and promising to on-line characterize the flow field of polymer melts.

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