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Note

Dynamic laser speckle pattern in monitoring of local deformation of tablet surface after compression

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Abstract

A method and a sensor device were developed to detect dynamic laser speckle pattern for monitoring of deformation of pharmaceutical tablets after compression. Temporal variations in optical signal revealed local surface deformation of the ibuprofen and the starch acetate tablets as well as anisotropy of tablet surfaces at the inspection location. © 2000 Elsevier Science B.V. All rights reserved.

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Pharmaceutical tablets are produced by compression of powder materials. The resulting porous tablets are expected to deform after compression due to relaxation processes in the tablet material. In other words the dimensions of the tablets can change as a function of the time. It is of importance for the tablets makers to gain information about such deformation because it will assist to optimise some further procedures that the tablet will undertake after compression. Unfortunately, the elastic or plastic deformation is usually rather small and therefore mechanical (destructive) measurement of the change of the dimensions of a tablet is a subject to some unreliability.

Optical metrology provides means for non-destructive real time inspection of the tablet's quality, such as surface roughness (Silvennoinen et al., 1999). Due to the surface roughness, scattering of light from the tablet's surface is always present. It means that diffuse reflection of light to the solid angle (half sphere) will occur. If the light source is

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a laser, which has properties such as low beam divergence and high coherence, then the scattering pattern at far-field region is a speckle pattern (Asakura, 1978). The generation of a speckle pattern is due to the coherent light scattering from a rough surface. Both vertical and lateral surface roughness can have an effect on the speckle pattern appearance. The spatial intensity of a speckle pattern at the detection point in the space depends not only on the resultant of the amplitudes of scattered light vector fields but also on their phases. This makes the speckle pattern sensitive to the surface roughness. A typical laser-induced speckle pattern from a tablet surface is shown, e.g. in Fig. 2(b) in (Silvennoinen et al., 1999). If a time-dependent deformation of a tablet's thickness or diameter is present, which can be equal or usually larger than one half of the wavelength of the coherent laser radiation, then dynamic speckle pattern (Okamoto and Asakura, 1995) will appear. This offers us a method to gain information in real time concerning the change of the tablet's local surface roughness, which may appear either in horizontal or lateral direction, e.g. as a result of plastic deformations. Indeed, it is a well-known fact that plastic deformations can make a surface rougher or smoother depending on the direction of the deformation.

As far as we know this is the first time that a speckle pattern technique is used for monitoring of the change of the surface roughness of a pharmaceutical tablet. The purpose of this paper is to give existence proof of the phenomenon. The information is qualitative hence we observe merely the time history of the change, but not the absolute value of the surface roughness (or its quantitative change) nor the direction of the change. Despite this shortcoming the time history of tablet's evolution has importance for tablet's makers.

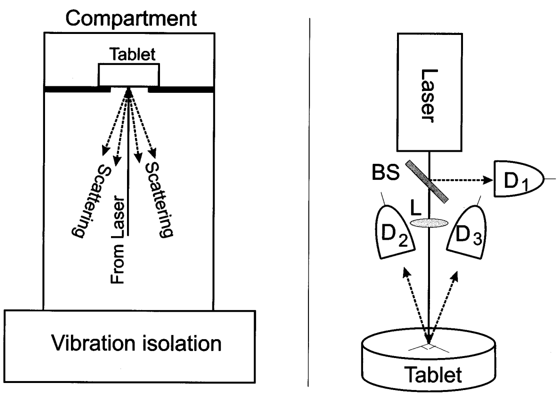

Fig. 1. Schematic diagram of the optical sensor. Left-hand side: the measurement compartment on optical bench; and right-hand side: optical transducer.

Ibuprofen (Ph. Eur. grade) and starch acetate (degree of substitution $ds = 2.9$, powder material was obtained from VTT Chemical Technology, Materials Technology, Rajamäki, Finland) tablets were compressed using a hydraulic compaction simulator (PuuMan Ltd., Finland) at the Department of Pharmaceutics at the University of Kuopio.

The optical sensor was fabricated in the Department of Physics at the University of Joensuu. Schematic diagram of the robust sensor is shown in Fig. 1. The laser source was a semiconductor laser (Meredith Instruments LDM-4d; wavelength, 635nm; and polarising degree, 1:100). The light (focused by lens L) is at normal incident on the tablet whereby fully developed speckle pattern appears in the far field region. The diameter of the laser beam waist on the tablet surface was 10 μm and the tablet diameter was 10 mm. The beam splitter (BS) is used in order to monitor possible intensity fluctuations of the laser by detector D1. The detectors D2 and D3, which both were 100 mm apart from the tablet surface, measure the variations of the local intensity of the corresponding speckle grain at far field region. The aperture diameter of the detectors D2 and D3 was 1 mm. The aperture of the detector is approximately one tenth of the diameter of single speckle grain. The speckle pattern is formed in the far-field region when light is scattered from a circular spot, which has a diameter of 10 μm. Whole circular area integrates the signal to be measured. If there is a change of surface roughness on that area then the speckle grain detected by the detectors D2 and D3 is expected to 'move' in detector planes. The apparent movement is due to the constructive or destructive interference of light field. This means that detectors will 'observe' a temporal change of light intensity due to dynamic speckle. These two

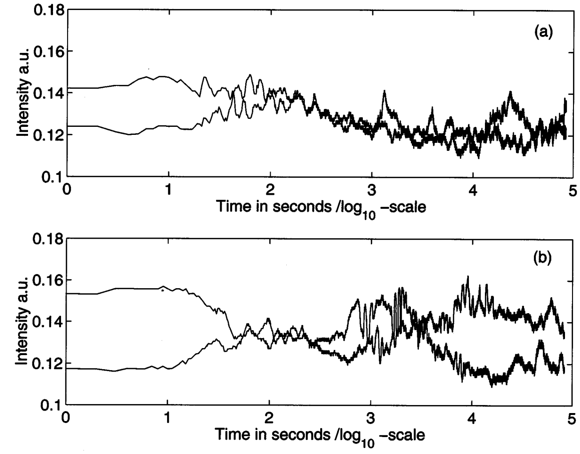

Fig. 2. Light intensities measured by detectors D2 and D3 for starch acetate tablets. (a) 8.01 kN, 300 mm/s; and (b) 8.69 kN, 100 mm/s.

detectors were installed in a rectangular configuration (the angle of the linearly polarised electric field vector of the laser beam halves the rectangle) for the purpose of monitoring possible anisotropy of the surface. Detectors D2 and D3 were small photodiodes (Texas Instruments TSL light-to-voltage optical sensor).

The tablet was transferred immediately from the compaction simulator inside a compartment of the optical sensor, which was on a vibration isolated optical bench. The compartment was used in order to block stray light. The tablet was put over a thin hole inside the compartment. The laser beam is incident on the lower surface of the tablet via the hole. The air humidity inside the compartment was not expected to change during the course of the measurements. The measurements started 10 s after compression.

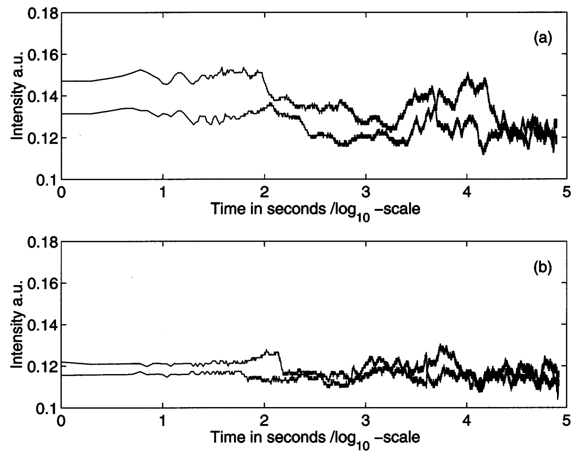

Fig. 3. Light intensities measured by detectors D2 and D3 for ibuprofen tablets. (a) 10.86 kN, 300 mm/s; and (b) 11. 82 kN, 100 mm/s.

As an example we show data for two ibuprofen tablets using compression loads and constant compression speeds equal to (11.82 kN, 100 mm/s) and (10.85kN, 3 00 mm/s) and for two starch acetate tablets (8.69 kN, 100 mm/s) and (8.01 kN, 300 mm/s).

In Figs. 2 and 3 we show the signals for the two tablets recorder by the two detectors. The signals have been normalised using the reference signal (D1). Such a procedure cancels possible fluctuations in the laser intensity. In Fig. 2(a) and (b) the signals for starch acetate are shown. The levels of light intensities of detectors D2 and D3 are rather stable but differ relatively much during the first 10 s in Fig. 2(a) (8.01 kN, 300 mm/s). The difference of the levels of light intensities is believed to be due to the anisotropy of the surface. After about 10 s deformation of the tablet occurs. Relatively quick deformation appears within 10–100 s (note the logarithmic time scale). At later instances deformation can be observed but the time responses are longer. Nevertheless, prolonged deformation is present. In Fig. 2(b) (8.69 kN, 100 mm/s) the signal levels differ more, during the 10 first s, if we compare them with those of Fig. 2(a). It has been also observed for starch acetate tablets that the higher the speed of compression, the faster the elastic deformation and vice versa (Raatikainen et al., 1999). Thus, for the 300 mm/s compression the deformation of tablet might be past, and for the 100 mm/s slower elastic deformation predominates. We suggest that the monitored surface in Fig. 2(b) is more anisotropic than the surface in the case of the Fig. 2(a). Nevertheless, in Fig. 2(b) relatively fast tablet deformation appears also in the time interval 10–100 s. Similar time responses in Fig. 2(a and b) are suggested to result from approximately equal compression forces.

As concerns the ibuprofen the tablet (10.86 kN, 300 mm/s) is subject to deformation after 10 s (Fig. 3a), whereas the tablet (11.82kN, 100 mm/s) is rather stable during the first minute (Fig. 3b). Both tablets have been deforming at later instances but in a manner that the tablet of Fig. 3(b) is more isotropic than the tablet of Fig. 3(a), i.e. there is a larger departure of the intensity levels in Fig. 3(a) than in Fig. 3(b). Deformation induced by a higher compression speed clearly makes the tablet surface less stable.

As a conclusion we may state that local inspection of tablet by speckle pattern technique may indicate temporal changes either in local or global surface/volume of the tablet. Short-term plastic deformation of a tablet is usually expected after compression. Our measurements show that also longterm local deformation is possible. The clarification of the origin of the latter phenomenon requires investigations.

It was observed that applying approximately same compression forces resulted to rather similar time responses of the tablet's deformation. However, the role of the combination of applied force and compression speed is not straightforward factor as concerns the anisotropy of the surfaces, and therefore further investigations are needed. A corroborating method to investigate tablets' deformation could be based on the use of a piezo crystal as a transducer for plastic deformation induced acoustic waves. However, the piezo crystal has to be in contact with the tablet (attached by glue). Therefore, the crystal would certainly disturb the tablet's proper state and the sensitivity of transducer would be rather poor. Another method, which might be useful is the photo-acoustic spectroscopy (PAS). Unfortunately, such a (commercial) spectral device is rather expensive if compared with the apparatus for speckle pattern technique. Furthermore, PAS needs extremely stable laboratory conditions for reliable measurements and the thermal waves generated by the apparatus disturb proper state of the inspected tablet.

The average signal levels depend on the reflectance of the tablets and also on their surface roughness. In principle it is possible to estimate also the surface roughness time-dependence by monitoring dynamic speckles. In that case so-called speckle correlation (Asakura, 1978) provides the measure for surface roughness. Then one usually has to assume that the surface height profile obeys normal distribution function. The experimental set-up for detection of speckle correlation is also different. That is to say either two laser beams with very small angle separation are simultaneously incident on the tablet, or one beam is used but the tablet is rotated slightly. After recording such data, e.g. by CCD-carnera it is possible to estimate surface roughness approximately in the range of 1–20 μm. Finally we remark that speckle pattern method can be applied to study deformation of any kind of tablet shape.

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