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A special instrument for exact control of self-organized structures preparation in polymer layers

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ABSTRACT

Special micro-condensation drying system (MCDS) was constructed for precise preparation of self-organized polymeric layers by casting from solutions and dispersions at highly non-equilibrium conditions. There were respected the needs for exact regulation of temperature, temperature gradient and rate of solvent evaporation, vibration damping, dust-free design and wide variability in process parameters settings as well. MCDS allows to study a broad spectrum of effects determining creation of self-organized structures during the evaporation of solvent from the polymer solution (e.g. Rayleigh–Bénard or Bénard–Marangoni cells). As a model system for MCDS functionality testing aqueous solution of hydroxyethyl-cellulose was chosen, with respect to its excellent film-forming properties and the self-organization ability. All of this is observable in high number of cell patterns, originating according to the process parameters, in the given polymer-solution system.

1. Introduction

Understanding the laws which determine creation of self-organized structures has been the subject of interest for more than 100 years in biological, as well as physical systems. As an example can be mentioned the research dealing with the creation of Bénard cells in the liquid layer subjected to a temperature gradient [1–6]. Problems connected with such a complex system, non-linearity, non-reversibility and dynamic transport processes complication. All of them are very sensitive to external factors [7–10], are still relevant and pose questions. Their answers can only be found from results of precisely performed experiments. With the development of computers techniques and their application, it seems to be this field of science still interesting thanks to the possibilities to simulate structural evolution of highly complex systems [11–15]. Application of these results in many fields of science, especially in production of clean surfaces with exactly defined properties [16–18], nanostructured materials [19], etc. should be very profitable.

One of the classic examples of self-organized structures creation related to energy and mass transfer may be previously mentioned formation of Bénard cells as a result of thermo-capillary instability [1,4,20–23] occurring in a liquid layer with a free surface (phase boundary liquid/gas, liquid/liquid) and a thickness up to approx. 10 mm [3]. The driving force for such a structures production is the magnitude of thermal gradient and related non-homogeneity of the surface tension [6] as a result of thermal fluctuations at the phase boundary. The initiation and propagation of this process at ‘‘stationary conditions” is well described in the review work of Colinet [3], or Nepomnyashchy et al. [5] from the theoretical point of view. Nevertheless, the ways and conditions determining reproducibility of this phenomena during the solvent evaporation from the solution, are not well explained for a wide variability of physical properties of the systems and originating structures, as described in the monograph of Colinet [3] (a reference to the unpublished work of M. Schatz, on page 9) or in the work of Toussaint et al. [24].

According to the effort to understand the laws determining creation and fixation of self-organized structures in thin polymeric films, prepared from a solution, under the influence of different process parameters (temperature, temperature gradient, rated of drying, etc.) a special drying apparatus – Micro Condensation Drying System (MCDS) was designed, constructed and tested, Fig. 1. The main reason for the construction of this apparatus was the necessity to eliminate as many random effects which can influence the character of resulting structures, as possible. This effect was described as the ‘‘Butterfly wing effect”, by Lorenz in 1993 [9] for the reason, to express its extreme sensitivity to the initial setting of parameters. This means that the exact prediction of the systems development depends on the appropriate knowledge of initial and process parameters as well.

To achieve the maximum reproducibility of created structures it is necessary to prepare the set of experiments from one solution at the same time, especially in the case of non-stable biological systems. Therefore, eight sub-units of MCDS were constructed, each of them fulfilling the needs for work in vibration- and dust-free

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Nomenclature

a	relative acceleration (m s^{-2})
d	thickness of the liquid layer
dT	temperature gradient between the carrying plate with the sample and the air in the height of 20 mm above this plate
f	frequency
k	thermal conductivity
Ma	Marangoni number
MW	molecular weight, average (g mol^{-1})
T	temperature
w	mass fraction of polymer in the solution

Greek symbols

γ	surface tension
ΔT	temperature difference between the bottom layer and an open surface
ΔT_C	threshold value of temperature difference for the initiation of the BM-instability
ε	parameter determining transition from symmetric polygonal BM-cells to non-regular
μ	dynamic viscosity
κ	thermal diffusivity

environment, exact temperature regulation, rate of drying and the possibility to modify the magnitude of thermal gradient and the capacity of the system thanks to a variable setting of the posts for samples, heating units and thermal probes.

Hydroxyethyl-cellulose aqueous solution was taken as a model system, for its excellent film-forming properties and the tendency to self-aggregate during the solvent evaporation, accompanied with formation of hydrogel with a certain amount of incorporated water [25].

2. Experimental apparatus

Solidification of polymeric solution into thin film layer in the MCDS is based on the principle of condensation drying. The circulating gas (air) containing vapour of the liquid does not come into contact with surrounding atmosphere. The same principle is adopted in modern wood-dryers and in the other industrial applications [26]. The design and construction of the MCDS sub-unit was focused to high adaptability of the whole system, allowing wide spectrum of power output regimes and internal positioning for supporting systems.

The draft of one of the eight MCDS sub-units is given in Fig. 2. Condensation of evaporated liquid itself takes place in the ''head'' of the dryer, which is the most important part of the apparatus. From this place, the condensate is transferred via the system of channels and pipes to a specially designed collector. The outer frame of a MCDS sub-unit consists of 40 mm thick double-walled housing, with dimensions 550 × 250 × 250 mm, made of the stainless-steel (standard EN 10088-1, material designation 1.4301). This housing is filled with particles of siliceous sand SIOPOR (Nagara, Czech Republic), with thermal conductivity coefficient $k = 0.043$ W m^{-1} K^{-1} and thermal stability to 1000 K. Heating and cooling elements, excluding the condensation one in the ''head'' of the dryer, were made of copper ($k = 401$ W m^{-1} K^{-1}) or aluminium ($k = 235$ W m^{-1} K^{-1}) rather than stainless-steel ($k = 14$ W m^{-1} K^{-1}). Copper and aluminium were coated with a layer of silicon oil, to avoid corrosion. Casing, sealing and housing of wires connected to inner heating elements, thermal probes, and sealing components were constructed from silicon rubber. This determines the upper temperature limit of application in the MCDS to 473 K.

Heating and cooling inside the MCDS sub-unit is provided by three (or four) independent elements. Two of them are fixed and are situated in the upper and lower part of the dryer. They can be set to act as heating or cooling elements; simply by switching the polarity of Peltier couples (PT-couples) with the power output 25–55 W. PT-couples are cooled by massive aluminium coolers with independently controllable system of ventilators. Remaining internal elements were constructed as adjustable heating ones, consisting of a coil of electric resistor wire located inside a ceramic holder and encased altogether in a copper sheet. They were constructed to allow changes in sizes and outputs of active heating segments. The power output can range from 25 to 100 W.

Temperature and its gradient are measured by resistive (platinum) sensors Pt100 (RTDs) manufactured by National Instruments Co., with a measuring range 223–473 K and the accuracy 0.1 K in the range 253–293 K. Data acquisition and control of the whole system is provided by a PCI card NI PCI-4351, National Instruments Co. Special attention was given in the construction of the MCDS to ensure various possibilities in positioning the PT-100 sensors and

Fig. 1. MCDS apparatus consisting of eight sub-units and supporting systems.