

Preparation of SnO₂ thin films for photovoltaic electrode materials using sol-gel method

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Introduction

Surface modification of glass by various methods is currently one of the most popular ways to improve the performance of glass products and extend its functionality. The most common way is to modify the surface by applying the thin layers of different composition and properties. This can be a layer of metal oxides or metal, as well as amorphous inorganic coating or modified with organic compounds. The surface layers typically have different chemical composition than the substrate. To make the resulting glass-layer system stable, the coating must be characterized by good adhesion to the substrate and their presence may not cause stress debilitating to mechanical properties of the product. The issue of adhesion is dependent on the chemical composition of the coating, the imposition of conditions and the properties of the surface of the glass itself. The best adhesion is obtained when a layer between the substrate forms chemical bonds, however physical bond is in many cases sufficient to obtain the required adhesion. Another solution may be to produce additional thin layer of intermediate binding glass to resistive or conductive layer.

Layers transparent to the visible light having the electrical conductivity are based on compounds of indium, tin, and zinc oxide doped with tin (IV) in the case of In₂O₃, In₂O₃ or SnO₂ as a matrix. The quantity of dopant is usually not exceeding 10%. The lowest resistance is achieved in the case of oxide layers of indium oxide doped with tin, and these are the most common, but due to the high price of indium it is important to search for less expensive, equally effective alternatives. There are commercially available conductive glass with a resistance of less 10 Ω/m or less at 75–85% visible light transmittance and high homogeneity of the surface conductivity. These types of glass are used mainly in electronic devices, as part of all types of displays, screens, electrodes for defrosting windows of vehicles, antennas, electrostatic screens [1] and gas sensors [2 ÷ 3]. Another no less important area of application are photovoltaic cells [4]. Area of application for electroconductive glasses of high transparency increases every year. The relationship between the transmittance and the conductance is closely correlated. In the above-described applications the high conductivity of the coating is important. Due to the increase in light absorption it can be increased by simply increasing the thickness of the layer both for reasons of cell efficiency decrease and economic factors [4]. Therefore, the search for new materials, which can be used as a transmission electrode has a very great practical significance.

Among conductive oxide materials tin dioxide in the form of thin films is widely used due to its good performance, high mechanical strength, chemical and environmental stability, and low cost. Tin dioxide is a tetrahedral crystal structure semiconductor. Its semiconductive properties are determined by a wide energy gap (3.5 – 4 eV) [5 ÷ 7]. Tin oxide itself a relatively poor conductor of electricity so in the practical solutions tin oxide doped with fluorine or antimony is used. Fluorine doping leads to substitution of the oxygen O²⁻ ions with monovalent F fluorine. This results in a deficiency of

electrons, and the material has *p*-type hole conductivity [8]. The modification by Sb³⁺ antimony ions by partial tin Sn⁴⁺ ions substitution, results in the excess of electrons in an oxide network and the material exhibits *n*-type electron conductivity [9 ÷ 10]. SnO₂ is relatively non-toxic and inexpensive, and the layers obtained have a very good adhesion to glass and high mechanical strength after heat treatment.

One of the types of photoelectrochemical cells are dye-sensitized solar cells (DSSC). In these structures the photovoltaic phenomenon does not occur in the traditional sense. Two functions of semiconductor such as silicon that are normally common here are separated. Photoelectrons are provided by an organic dye molecules and the semiconductor layer or the nanoparticles are used for the separation of charges. In a typical dye cell an illuminated electrode is a conductive glass with a layer of a broadband semiconductor nanoparticles and adsorbed organic dye molecules on its surface, which acts as a sensitizer. There is widespread use of titanium dioxide, TiO₂, which is a semiconductor with a wide energy gap 3.2 eV, highly absorbs ultraviolet light, but only a few percent of visible light. Illuminated electrode building materials are deposited on the glass, which is transparent to the incident sunlight and has a layer of conductive ITO (indium tin oxide) or a cheaper and more stable FTO (fluorine tin oxide). The counterelectrode is also usually made of glass with a layer of FTO, coated with platinum, on which the reduction reaction of the electrolyte occurs – see Figure 1.

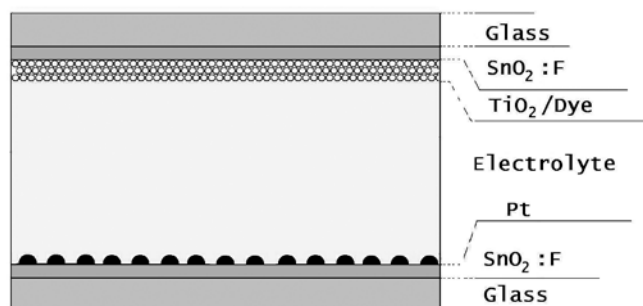


Fig. 1. Schematic of the Dye Sensitized Solar Cell

As a precursor of tin (IV) oxide in thin film technology both inorganic compounds – mainly chloride, tin (II) chloride, tin (IV) [11 ÷ 12] and tin metal (in the vapor deposition techniques, combined with the simultaneous oxidation of the layer) and a series of organic compounds are used. For synthesis using the sol-gel technique, sols are prepared from alkoxyates of tin (IV), such as ethoxide [13 ÷ 15], *n*-propylane [16 ÷ 17], tin butoxide [18] or more complex systems [19 ÷ 20]. SnO₂ layers can be prepared many ways, which often results in the production of coatings with different utility values. The goal is to develop a methodology for obtaining and regulating the parameters of tin oxide thin films with optimum electrical and mechanical properties based on the sol-gel process using tin (IV) acetate as a precursor of tin (IV) oxide. Currently,

inorganic tin compounds used as precursors of SnO₂ can replace an organic derivative of tin, eliminating the emissions of hydrogen chloride resulting from the deposition of SnO₂ layers using SnCl₂ and SnCl₄. Furthermore, tin (IV) acetate of high purity [21 ÷ 22], in contrast to the other of the metal precursors used do not contain chlorine or fluorine atoms, both in the structure of the molecule, or as impurities after the synthesis, as well as other undesirable from the standpoint of chemical dopant material (cationic), eg, sodium, potassium. In this way it becomes possible to eliminate the influence of these elements in the results interpretation of the thin films properties such as electrical conductivity. This allows for the elimination of inorganic anions in the synthesis of the layers and makes it possible to obtain thin films of high mechanical quality, high transmittance in the UV-Vis and to reduce the cost of production elements using the tin (IV) oxide.

To obtain a thin layer on the glass dip coating technique was used which allows to obtain a uniform layer but requires repeated application, as a single layer is relatively thin and is not continuous over the whole surface, which usually leads to a lack of conductivity. The solution of the problem was obtaining the tin oxide by sol-gel technique, producing high-tenacity and continuity layers containing small defects. Deposition of a liquid phase coating by the sol-gel technology in comparison to the CVD and PVD requires far less complicated equipment, is less costly and it gives greater availability of the precursors and the possibility of modification, the ability to control the reactivity of the precursors. The obtained materials are non-toxic and the resulting coatings have good mechanical properties and thermal stability. The process is low-temperature and gives the ability to mix the organic and inorganic components [23].

Experimental

Preparation

Preparation of SnO₂ by means of the sol-gel technique

The precursor for the tin(IV) oxide obtained by sol-gel synthesis was a tin (IV) acetate. To 150 cm³ of methanol containing 0.25% (v/v) water and 0.1% (v/v) ethylene glycol 3 g of tin (IV) acetate was added and heated for 4 hours under reflux at 50 °C. The resulting opalescent sol was used for application to glass substrates. SnO₂ gel for thermogravimetric analysis and TEM studies was prepared by drying 10 cm³ of the sol described above in a petri dish at room temperature for 2 days.

Preparation of the glass substrate

As the substrate for the SnO₂ layer soda-lime glass with dimensions of 25 x 76 mm and a thickness of 1.5 mm was used. The plates were cleaned using the following organic solvents: methanol, hexane, acetone. The plates were placed in a glass vessel in an ultrasound bath for 5 minutes, in above mentioned solvents, followed by drying in a stream of nitrogen. In order to activate the glass surface (to obtain Si-OH silanol groups) they were subjected to the following procedure: after placing them in a 3:1 solution of sulfuric acid and hydrogen peroxide (60 cm³ H₂SO₄ : 20 cm³ 30% H₂O₂) in a Teflon vessel, heated at 80 °C for 2 hours. After that plates were washed with redistilled water and acetone and dried under a nitrogen stream.

Preparation of SnO₂ layer on a glass substrate

The prepared plates with a hydrophilic surface were loaded by immersion in SnO₂ sol. Layering technique consisted of the following steps: dipping (1), ascending (2), the beginning of the run-off (layer formation) (3), evaporation of the solvent (4). After application and drying of the gel calcining the substrate coated with

a layer at a temperature of 500 °C in air for 10 minutes was carried out. A series of samples containing from 1 to 12 layers of the applied SnO₂.

In a separate experiment the drying process by means of techniques of FT-IR spectroscopy was examined, using a snap-ATR (SPECAC). On the surface of the diamond crystal methanol SnO₂ sol was applied. Recording of absorption spectra were carried out every 5 seconds (total time of recording of one spectrum in the 4000–600 cm⁻¹).

Physicochemical characteristics

Kinetic studies of drying the film consisting of SnO₂ sol in methanol was carried out using FT-IR spectrometer (Bruker, TENSOR 27) from ATR accessory (SPECAC). Measurement resolution was 4cm⁻¹.

The phase identification of the layer applied on the glass substrate and the influence of the crystallinity of calcined SnO₂ phase on the glass substrate was performed using X-ray diffraction (XRD) powder diffractometer (Philips, PW 1050) using CuK α lamp radiation and Ni filter. X-ray spectra were recorded in the angular range 15–60 [2 θ]. The measurements were carried out for a series of systems containing from 1 to 12 layers.

Imaging the surface of the layers was performed with the help of SEM electron microscopy (Philips 515 SEM).

The identification of the elemental composition and location for the layers on the glass substrate was performed using SEM-EDS with (OES) GDS500A LECO, Neophot 32.

SnO₂ gel thermal analysis was performed using Differential Thermal Analyzer (Netzsch STA 409C 3F) under a nitrogen atmosphere (15 cm³/min) and air (15 cm³/min).

UV-Vis measurements were performed on Merck (SPECTROQUANT Pharo 300 model) spectrophotometer.

Discussion

The study of time-resolved techniques using FT-IR (ATR) allowed the determination of the drying time and film forming at room temperature. Figure 2 shows the absorption spectra of forming layers. The characteristic bands for the methanol (the main component of sol) can be observed – broadband 3319 cm⁻¹ OH vibration, the C-H band 2943, 2831 cm⁻¹ and the strong at 1022 cm⁻¹. Moreover, there are observed much less intense bands characteristic for the acetic acid – 1249 cm⁻¹, 1730 cm⁻¹ and 1748 cm⁻¹ derived from acetic acid, the C=O carbonyl group in the spectrum.

Reflectance technique is particularly useful [24] in the study of the layers formation, so that the interfacial area is being observed (contact of the surface and forming a crystal layer). This makes possible to determine which components of the matrix diffuse to the surface of the evaporating film and which are moving to the bottom. An additional advantage of this technique is precise determination of the end point of the drying and forming a layer on the substrate (in this case it was after about 370 seconds).

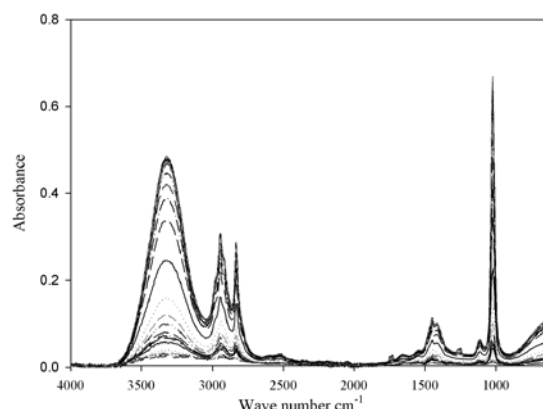


Fig. 2. Time-resolved studies of the process of drying the applied layer of SnO₂ measured by FT-IR technique (ATR)

This effect has been described in detail in comparison of the individual bands of acetic acid and methanol (the main components of the precursor layer) in Figure 3. Acetic acid (bands D, E, F) formed during the hydrolysis of the tin (IV) acetate diffuses into the surface layer film in the first phase of the drying process. A significant decrease of its concentration from 0 to 150 seconds the process is observed. Approximately in 335th seconds of the process one can observe a sharp decrease in intensity of all bands, associated with the final evaporation of methanol (band A, B, C) and the formation of a gel film. It was also found that some of the acetic acid is not evaporated, but is still in the gel structure.

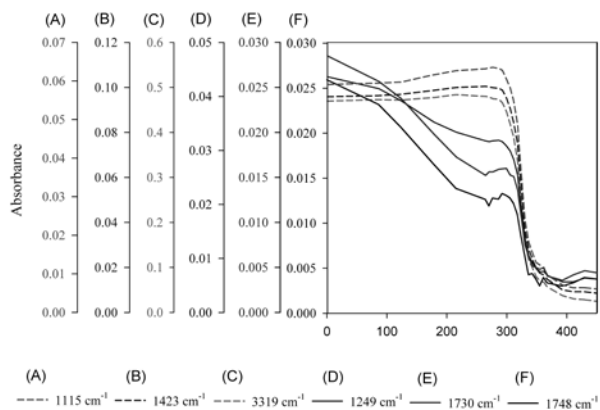


Fig. 3. Kinetics of loss of the methanol (A, B, C) and acetic acid (D, E, F) characteristic bands in a forming gel SnO₂ layer

The presence of organic residue in the structure of the materials obtained by the sol-gel process is well known and well documented in the literature [21]. To illustrate the process of its removing during the thermal forming of SnO₂ layer on the surface of the substrate, thermogravimetric studies were performed under oxidizing and inert (nitrogen) conditions (air) (Fig.4). In the course of material processing in the oxidizing conditions initially (up to 480K) the process of removing physical (weakly) associated solvent molecules is observed. At a later stage (above 600K) a rapid loss of weight with a maximum process speed at 625K was observed. The second change, most likely mainly of an exothermic reaction is bound to an oxidation of organic compounds originating from the tin(IV) precursor [25].

The process of removing the organic solvent from the gel network run under an inert atmosphere is similar in the first stage but at higher temperatures (above 600K) is much more gentle, it is not such a rapid sequence. Treatment under inert conditions also requires a higher temperature to completely remove the organic residues.

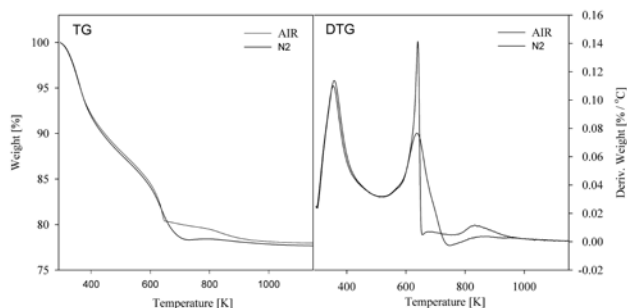


Fig. 4. Thermogravimetric analysis SnO₂ gel

Structural studies of the obtained gel lead to the conclusion that the SnO₂ after drying at room temperature is amorphous (Fig. 5). High-temperature treatment (773K, 30 minutes) changes the diffraction pattern, which indicates an appearance of a small crystalline structure proportion in the resulting tin gel. The broad low-intensity reflections located at 2θ=26° and 33° (Fig. 5) correspond to the tin (IV) oxide (cassiterite) characteristic reflections [26].

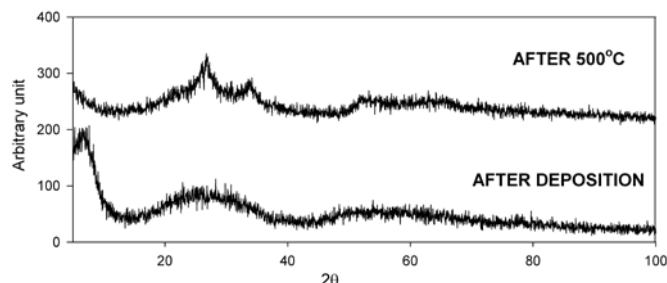


Fig. 5. SnO₂ gel diffraction patterns before and after calcination

The process of successive applying SnO₂ layers on a glass substrate was also illustrated using X-ray analysis – see Figure 6. The clearly highlighted presence of SnO₂ reflections the can be observed from 4th layers of oxide. Layers one to three give picture of amorphous phase. As expected, with increasing number of deposited layers the intensity of the bands increases, although it not is a regular tendency.

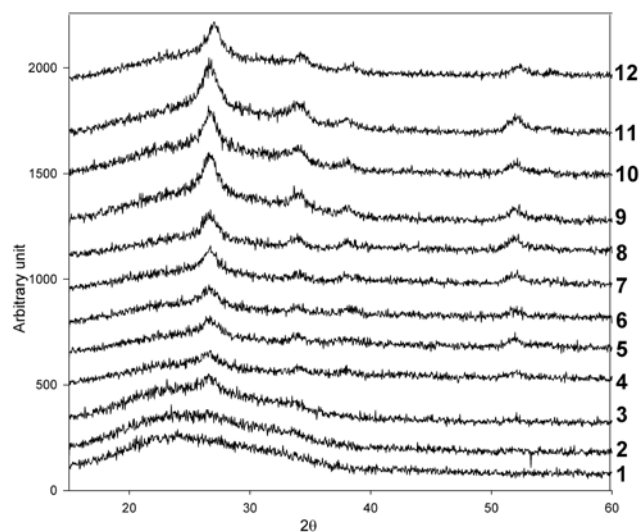


Fig. 6. Summary of diffraction patterns obtained from the SnO₂ on glass surface containing one to twelve layers, after calcination in air

The proposed method allows to obtain uniform continuous layer with low imperfections. First, the continuity and composition of the resulting layers was analyzed by SEM-EDS technique. The tested sample consisted of three tin gel layers deposited on the glass plate after calcination at 500°C in air. Elemental composition of the layers on the glass was confirmed – see Figure 7. A main component is SnO₂. Si signal comes from the glass substrate.

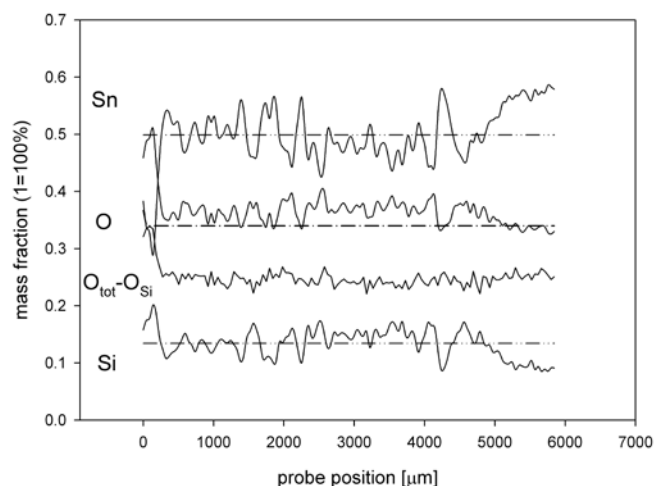


Fig. 7. Elements distribution profile on the surface of the obtained layer (based on SEM EDS)

SEM images are shown in Figures 8a, b, c. Pictures show defects on the glass surface layer caused by the method of the tin gel drying. The gel formed under ambient conditions is dried and then annealed to a 500°C temperature. During the thermal treatment the polycondensation reaction is continued. Also the release of water, the solvent and the oxidation of organic thickening gel occurs. Heat treatment should ensure that despite the huge contraction of the heated gel it has not been fractured because of the forces of the surface tension acting inside the pores. As can be readily seen, this step needs to be developed.

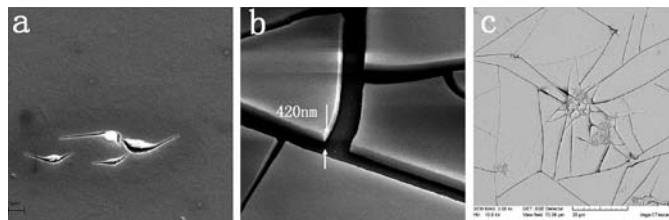


Fig. 8. Microscopic photographs of a SnO₂ single layer on the surface of the glass (SEM – a, b, c)

Dip-coating technique allows to obtain a uniform layer but requires repeated application, since the single layer is relatively thin and is not continuous over the whole surface, which usually leads to a lack of conductivity. The total film thickness rises with the increasing number of deposited layers but the resistance decreases. Each application of a coating however, involves the firing in temperature 400–550°C, which significantly increases production costs of this type of coatings [1]. In addition, unfavorable phenomenon appearing with increasing number of layers is imposed by the deterioration of the optical parameters of the resulting electrode. This is particularly undesirable from the standpoint of the applicability of SnO₂ layer on the glass substrate for photovoltaics. In the proposed method electrode material is obtained with a high optical quality. This has been confirmed by spectroscopic studies (Fig. 9.). Systems containing 6 to 12 layers of tin(IV) oxide had a 90% transmissivity.

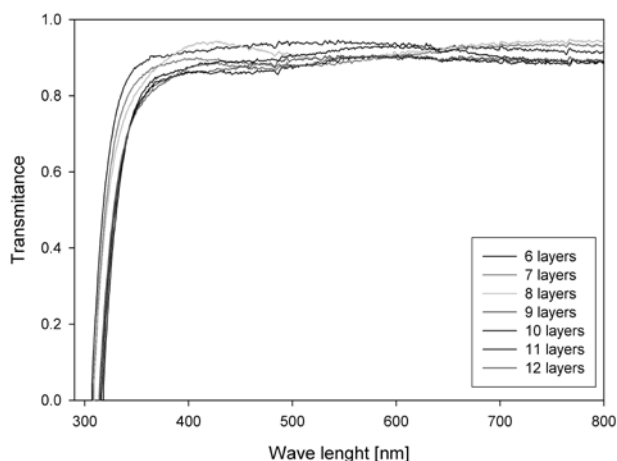


Fig. 9. Transmission spectrum of UV-Vis glass substrates containing six to twelve SnO₂ layers

It appears that the main reason for such a good performance is the homogeneity of the layer construction, including its uniform surface [27].

Results and summary

The proposed preparation method of the tin(IV) oxide thin films allows to obtain a homogeneous material with a high surface area. Optimization of layer drying conditions will allow obtain a low defects material and thus high optical performance. FT-IR measurement techniques of layer drying kinetics were shown to be useful in particular

to determine the end point of the layer formation. A further step in research work would be to introduce fluorine ions into the structure obtained layers to increase their conductivity. The high attractiveness of the proposed method for the preparation of tin(IV) oxide layers is acknowledged by cooperation with the Innosil Company in research and development at the field of obtaining conductive glass substrates for optoelectronics. At the same time we look for partners from other research units in order to extend the application study of the obtained material to other areas of science and technology.

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NOWE PRODUKTY

Basell Orlen z nowym PP

Moplen RP2802, z najnowszej oferty Basell Orlen Polyolefins Sprzedaż Sp z o.o. (BOPS), stanowi nowy wzorzec osiągnięć dla kopolimeru randomicznego w zastosowaniach wytwarzanych metodą formowania wtryskowego, łącząc bardzo wysoką płynność polimeru w stanie stopionym z jego doskonałą przezroczystością. Typowe zastosowania dla gatunku Moplen RP2802, to cienkościennie pojemniki o dużej pojemności, które są stosowane w gospodarstwach domowych oraz pojemniki o wyjątkowych właściwościach estetycznych i dobrych osiągnięciach mechanicznych. Testy przeprowadzone przez polskich klientów potwierdzają, że wysoka płynność, w połączeniu z najnowszą generacją środkiem nukleującym, w przypadku gatunku Moplen RP2802 umożliwi poprawę warunków przetwórstwa i redukcję czasu cyklu w szybkim wtrysku, co może przyczynić się do podwyższenia produktywności. (kk)

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Nowy rozpuszczalnik organiczny w ofercie Brenntag Polska

TOU (2,5,7,10-tetraoksaundekan) to innowacyjny, skuteczny i przyjazny środowisku naturalnemu rozpuszczalnik acetalowy o bardzo wysokiej – porównywalnej z rozpuszczalnikami chlorowcopo-

chodnymi, czy NMP – efektywności, który charakteryzuje też doskonałą mieszalność z wodą i innymi rozpuszczalnikami organicznymi oraz wysoka kompatybilność ze środkami powierzchniowo-czynnymi. TOU świetnie sprawdza się jako tzw. stripper, czyli środek do usuwania starych powłok malarskich, a także w zastosowaniach przemysłowych, np. przy produkcji farb i lakierów, klejów, polimerów i ekologicznych środków czyszczących. (kk)

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NOWE INWESTYCJE

Nowy oddział produkcyjny Polpharmy w Nowej Dębie

Spółka Sanfarm w Tarnobrzесьkiej Strefie Ekonomicznej stała się częścią Zakładów Farmaceutycznych Polpharma SA. W wyniku połączenia obu spółek powołano Oddział Produkcyjny Polpharmy w Nowej Dębie, który będzie realizował zadania w zakresie produkcji farmaceutycznej, zarówno dla firm należących do Grupy Polpharma, jak też podmiotów zewnętrznych. Wszyscy pracownicy Sanfarmu zostali zatrudnieni w nowo utworzonym Oddziale, a dotychczasowy prezes spółki Ryszard Latawiec objął stanowisko Dyrektora Oddziału. Formalne połączenie przyczyni się do integracji działalności spółek i przyniesie wymierne korzyści dzięki efektowi synergii, m.in. w procesach zakupu surowców, materiałów opakowaniowych czy mediów. (kk)

(<http://www.polpharma.pl>, 19.11.2013)

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