

# Tin dioxide sol–gel derived thin films deposited on porous silicon

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## Abstract

Undoped and Sb-doped SnO<sub>2</sub> sol–gel derived thin films have been prepared for the first time from tin (IV) ethoxide precursor and SbCl<sub>3</sub> in order to be utilised for gas sensing applications where porous silicon is used as a substrate. Transparent, crack-free and adherent layers were obtained on different types of substrates (Si, SiO<sub>2</sub>/Si). The evolution of the Sn–O chemical bonds in the SnO<sub>2</sub> during film consolidation treatments was monitored by infrared spectroscopy. By energy dispersive X-ray spectroscopy performed on the cross section of the porosified silicon coupled with transmission electron microscopy, the penetration of the SnO<sub>2</sub> sol–gel derived films in the nanometric pores of the porous silicon has been experimentally proved. © 1997 Elsevier Science S.A.

**Keywords:** Tin dioxide; Sol–gel; Porous silicon

## 1. Introduction

Tin oxide semiconducting layers incorporating small amounts of noble metal (Pt, Pd) and metal oxides dopants (Sb, In, Bi, etc.), prepared by thick and thin film technology have been widely used for gas sensing applications where conductivity change is used to detect reducing gases [1]. Thick film technology for SnO<sub>2</sub> preparation (up to 1 µm), mainly based on hot pressing at high pressures and sintering at high temperatures (around 1200°C) is practically excluded from integration in the fabrication of intelligent microsystems, not only due to processing incompatibility but the high power consumption for gas sensing applications. Thin film technologies (below 1 µm) based on physical methods like vacuum evaporation or sputtering for SnO<sub>2</sub> deposition, combined with ion implantation for film doping with metal ions are very expensive and may suffer of composition inhomogeneity and poor capabilities to provide a good step coverage for topographies with very high aspect ratios.

Recently [2–4], an increasing interest towards sol-gel synthesis of the SnO<sub>2</sub> thin films has been observed due to the method advantages: increased capability to tailor complex compositions, simple and cheap technological equipments, low thermal budget necessary for film consolidation and specific controlled porosity properties [5]. This last property, determining the high specific area of the SnO<sub>2</sub> sensing layer seems to be quite attractive for promoting increased sensing properties of the SnO<sub>2</sub> based sensors [6]. An alternative approach for an increased sensing area was the deposition of thin SnO<sub>2</sub> films in the porous silicon (with very high specific area) by atomic layer deposition [7].

It is the final target of our research to investigate the capabilities of the sol–gel method to obtain very thin SnO<sub>2</sub> layers in the nanometric pores of the porous silicon (PS) and to further enhance the sensing area due to controlled porosities of SnO<sub>2</sub>, as mentioned above. Moreover, it would be interesting to observe if there is any influence on the SnO<sub>2</sub> sensing properties, themselves, due to a porous silicon substrate.

As an initial step, it is necessary to study the capability of the sol–gel method to generate a SnO<sub>2</sub> (liquid) sol phase able to penetrate in the pores of the PS and

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then to obtain a SnO<sub>2</sub> solid film on the walls of the PS by subsequent annealing. For this purpose, the basic aspects of the sol–gel method for the synthesis of the SnO<sub>2</sub> sol compatible to this challenging task will be briefly discussed.

It is the purpose of this paper to prove by coupled measurements of cross-section transmission electron microscopy (XTEM) and energy dispersive X-ray spectroscopy (EDXS) performed on porous silicon that the tin oxide is present inside the pores of PS and to show thus the feasibility of sol–gel method for this task. Further technological improvements concerning increased wettability of the solution in conjunction with surface substrate preparation have to be investigated in order to enhance the penetrability of the SnO<sub>2</sub> in the pores.

## 2. Basic aspects of sol–gel method for the synthesis of SnO<sub>2</sub> sols compatible with deposition in the porous silicon

The deposition of SnO<sub>2</sub> films in the pores of the nanometric PS determines the selection of organic precursors (metal alkoxide, carboxylate,  $\beta$ -diketonates, citrates) as a starting point for a polymeric sol generally consisting of ‘particles’ not larger than 1 nm [5], best defined as polymeric molecules formed in nonaqueous solutions.

Under these circumstances, the possible limitations in the pores filling with SnO<sub>2</sub> liquid precursor might arrive from the surface tension phenomena rather than a larger diameter of the ‘particle’ with respect to the pore size. A possible solution is the use of different surfactants introduced in the hydrolysed sol or applied on the substrate in order to increase the wettability of pore walls.

The following chemical principles should be considered in order to prepare homogeneous polymeric sol with particle size of 1 nm. Because the metal alkoxide precursors should preserve their solubility while polycondensation takes place, an optimum water/alkoxide molar ratio, known as hydrolysis ratio (HR) should be chosen. Therefore, alkoxides with greater covalent character should be selected [8].

The hydrolysis reaction (replacement of the organic alkoxy groups by OH groups) should be catalysed by an acid (electrophilic mechanism), whenever is the case in order to obtain linear macromolecule, clear gel and dense film. The amount of water is essential for the thin film synthesis. In fact, the effect of water depends on the type of catalyst. Thus, at basic pH and very high hydrolysis ratios (equal to 1833), SnO<sub>2</sub> particles with diameter in the range 100–250 nm are obtained from tin ethoxide with ammonia as a catalyst [9]. At HR of 73 and basic hydrolysis, monodisperse 70 nm SnO<sub>2</sub>

spheres are obtained. Therefore, under acidic catalyst and very low HR, molecular dimensions of the SnO<sub>2</sub> particles are expected.

The solvent choice is determined by cost, safety, vapor pressure and precursor complexity. Thus, more complex compositions are easier to mix in longer-chain alcohols (parental alcohols are welcomed). In alcoholic solutions, at low pH, depolymerization rate is very low and a Sn–O–Sn bond once formed cannot be broken resulting thus in a three-dimensional network structure for the polymeric gel.

For the case of preparation of the doped SnO<sub>2</sub> films with a noble metal, the principles of multicomponent sol synthesis should be considered. In this case the miscibility of the precursors in the common solvent and the hydrolysis rate of each component should be taken into account. Very slow addition of water, partial hydrolysis of the less reactive alkoxide or chelation of the cations (complexing by ligands which donate a pair of electrons to the cation) are methods to be used in order to avoid precipitation of the faster hydrolysable components in the multicomponent sol.

## 3. Experimental procedure

Based on the above principles we have started the synthesis of the undoped and Sb-doped SnO<sub>2</sub> sol phase consisting of the following precursors: tin (IV) ethoxide, laboratory prepared, SbCl<sub>3</sub> (J.T. Baker), while absolute ethyl (Merck) alcohol was used as solvent.

As an initial step before sol synthesis, the precursor in the form of powder was analysed by differential thermal analysis (DTA), thermogravimetric analysis (TGA), infrared spectroscopy (IR) and X-ray diffraction (XRD).

The DTA experiments have shown that endothermic effects occurred at 180 and 230°C while exothermic effects appeared at 310 and 430°C. Below 180°C, the solvent and the water are removed (50% weight loss was obtained from TGA measurements) while at higher temperatures, the decomposition of tin ethoxide (at 230°C), burning of organic residues (at 310°C) and crystalline ordering (at 430°C) occur. From TGA it was obtained that additional 24.45% weight loss took place in the temperature range 230–430°C. An important conclusion from these experiments is that after thermal annealing at 450°C, the SnO<sub>2</sub> obtained from this precursor may be in the crystalline state.

The IR study (in the range 400–4000 cm<sup>-1</sup>) of the precursor has revealed the specific absorption band of the Sn–O bond from the wave number 540 cm<sup>-1</sup> and also the presence of water and hydroxyl groups in the range 3200–3600 cm<sup>-1</sup>.

The XRD analysis of the (powder) precursor calcinated at 400 and 1000°C confirmed that at low temper-

Table 1

Evolution of pH, viscosity and long-term stability of the undoped and Sb doped SnO<sub>2</sub> sol as a function of solution concentration and hydrolysis ratio for tin (IV) ethoxide and SbCl<sub>3</sub> precursors diluted in ethanol (a common solvent)

No.	Solution concentration (M)	HR	pH	Viscosity (cP)	Temperature (°C)	Order of adding <sup>a</sup>	Stirring time (min)	Doping	Sol stability
1.	0.26	0	1.41	1.4	20	A+B	30	No	10 h
2.	0.09	0	1.4	1.37	20	A+B	30	No	48 h
3.	0.075	0	1.4	1.3	20	A+B	30	No	48 h
4.	0.066	0	1.4	1.25	20	A+B	30	No	20 days
5.	0.046	0	1.4	1.2	20	A+B	30	No	30 days
6.	0.046	15	1.4	1.2	20	A+B+C	30	No	No
7.	0.046	10	1.4	1.2	20	A+B+C	30	No	10 days
8.	0.066	0	0.8	1.2	20	A+B+D	30	Sb 10% M	30 days
9.	0.046	0	0.8	1.2	20	A+B+D	30	Sb 10% M	30 days
10.	0.046	6	0.8	1.2	20	[(A+B)+C]+D	30	Sb 10% M	30 days

<sup>a</sup> A, tin IV ethoxide; B, ethanol; C, deionized water; D, SbCl<sub>3</sub>.

ature the SnO<sub>2</sub> cassiterite crystalline structure is present (ASTM No. 21-1250). The XRD investigation of the precursor before any thermal annealing provided a diffraction pattern with few weak diffraction peaks, characteristic for the microcrystalline samples.

The tin ethoxide was dissolved in different amounts of ethanol and the following sol concentrations were obtained 0.08, 0.1124 and 0.32 M.

For the initial stage no water was added into the sol. This means that only residual water from ambient, precursor and solvent will be present in the sol phase.

For these three sol concentrations, a spin coating method was used to deposit layers of tin oxide on the silicon wafers.

The parameters of the process for the spin coating film preparation were as follows:

- wafer rotation was equal to 4000 rpm for 30 s,
  - wafer drying after spinning was performed at 120°C for 15 min,
  - thermal annealing was made at 500°C for 30 min.
- The films prepared with the sol of 0.32 M concentration presented cracks after the drying process.

The next experiments were carried out with the sols of lower concentrations.

Multiple spin coating depositions were performed directly on the silicon substrate. The thermal annealing at 500°C was made after each spin coating.

This preliminary set of experiments was the driving force for a systematic investigation of the role of tin concentration and hydrolysis ratio on the pH, viscosity and stability of the undoped and Sb-doped SnO<sub>2</sub> hydrolysed sol. The results of this experimental study are given in Table 1.

According to this table, optimum solution concentration with respect to tin ethoxide is in the range from 0.046 to 0.066 M where the sol stability is over 30 days. As presented above, the hydrolysis ratio plays an essential role in the sol stability. Values of HR up to ten can be used for the hydrolysis but the sol stability with time

is lowered. The viscosity of the sol has slowly increased with sol concentration remaining in the range 1.2–1.41 cP when the sol concentration increased from 0.046 to 0.26 M. These relatively low values of the sol viscosity are encouraging for the penetration of the sol in the pores of porous silicon according to our gas sensing application. The pH of the sol was low indicating an acidic character (1.4) and therefore no acidic catalyst was further added. The addition of the SbCl<sub>3</sub> dopant to the sol has further decreased the pH of the solution to about 0.8. Clear, transparent and stable sols were obtained for a hydrolysis ratio equal to zero and sol concentrations of 0.046–0.066 M in the case of undoped and Sb doped 'hydrolysed' sol. In fact, in this case, residual water from solvent and ambient was present in the sol.

Based on the above experiments, uniform, transparent and crack-free SnO<sub>2</sub> thin solid films were obtained by spin casting of a 0.2 µm filtered solution on freshly cleaned Si, SiO<sub>2</sub>/Si and PS/Si substrates. The thermal sequence of film consolidation consisted of drying (150°C), firing (400°C) and crystallization (500–850°C) in oxygen ambient. The evolution of the film composition and its chemical bonds during SnO<sub>2</sub> consolidation was monitored by IR spectroscopy performed on a IR-80/C Zeiss spectrophotometer in the 200–2000 cm<sup>-1</sup> range.

The porous silicon substrates (about 2 µm in depth) were obtained by standard anodization process on highly boron doped silicon substrates with resistivity of about 1 mΩ-cm and (100) crystallographic orientation. For the cross-section transmission electron microscopy coupled with X-ray detector investigations of the SnO<sub>2</sub> penetration in the pores of PS, two small chips of the sample have been cut and embedded face to face into a special Ti supporting grid. After mechanical grinding (down to 50 µm) and polishing, the sample was thinned using Ar ions at 10 keV in an IV3 type ion miller. The sample preparation and the TEM analyses were per-

formed at RITP Budapest on a Philips CM 20 TEM instrument equipped with NORAN EDS X-ray detector for composition investigation on the cross section of the PS. Electron diffraction patterns were also recorded, in order to investigate the structural changes in porous layer as a function of technological processing and SnO<sub>2</sub> film deposition.

#### 4. Results and discussion

Starting from tin (IV) ethoxide, up to ten consecutive undoped SnO<sub>2</sub> layers were successively deposited on Si substrates. Each layer was dried and thermally annealed separately in order to avoid cracking. The thickness of each SnO<sub>2</sub> spin coated layer was about 15–20 nm while the refractive index was 2.1 in agreement with the literature data [4]. The Sb-doped SnO<sub>2</sub> films obtained from tin (IV) ethoxide and SbCl<sub>3</sub> presented a lower thickness per spin casting, of about 10–15 nm at a similar solution concentration.

The IR analysis of the SnO<sub>2</sub> layers (from tin ethoxide) has been performed at different stages of the layer consolidation as shown in Fig. 1. In Fig. 1(a) we show the IR spectrum of the layer after air drying at 150°C for 30 min. A well configured band at 320 cm<sup>-1</sup> (not described in the literature), as well as a broad band at about 550 cm<sup>-1</sup> (assigned to Sn–OH, according to Ref. [10]) and a shoulder at about 650 cm<sup>-1</sup> can be seen.

After firing treatment at 500°C in air, the IR analysis of the SnO<sub>2</sub> densified films (Fig. 1(b)) has shown an interesting evolution of the absorption bands specific to the film composition and interface processes as follows. The absorption band from 320 cm<sup>-1</sup> has decreased in intensity. The absorption band from the wavenumber 550 cm<sup>-1</sup> and the shoulder from 650 cm<sup>-1</sup> have transformed together into the well resolved absorption band from 620 cm<sup>-1</sup> which is associated to Sn–O–Sn covalent bonds vibration [10]. Therefore, after this treatment, the polymerisation reactions (Sn–O–Sn bonds formation) have become dominant, explaining thus, the decrease in intensity for the bands from 550 cm<sup>-1</sup> (SnOH) or 320 cm<sup>-1</sup>. In addition, in Fig. 1(b), two new, poor resolved absorption bands appeared at about 800 cm<sup>-1</sup> and 1100 cm<sup>-1</sup>. These are the well-known absorption bands of the SiO<sub>2</sub> network formed by surface oxidation of silicon substrate. In this case, these new bands appeared due to the annealing in air of the SnO<sub>2</sub> film deposited on monocrystalline silicon. Therefore, the IR spectroscopy has revealed the onset of the thermal oxidation process of silicon in addition to native thermal oxide. The oxygen from the air has diffused through the loose structure of the SnO<sub>2</sub> layer to the interface and oxidised the silicon surface creating a very thin layer (a few nanometers, including the

native oxide) of SiO<sub>2</sub> as an interface layer between the SnO<sub>2</sub> and Si substrate.

After a thermal treatment at 850°C in air, all the absorption band structure (Fig. 1(c)) has changed according to the trends seen after the 500°C annealing, as follows. The 320 cm<sup>-1</sup> band has decreased very much and the absorption band from 620 cm<sup>-1</sup> became narrower, proving thus the end of condensation reaction. Taking into consideration the increased intensity of the 320 cm<sup>-1</sup> band after drying treatment at 120°C and its gradual decrease with SnO<sub>2</sub> film annealing temperature, this band might be associated to the vibration of a Sn–O bond of the alkoxide precursor, itself. In addition, this 320 cm<sup>-1</sup> absorption band has been revealed by us in a commercial SnO<sub>2</sub> powder even after annealing at 1000°C in air, proving thus its assignation to a certain vibration of Sn–O bond present mainly in precursor, SnO<sub>2</sub> powder and less densified SnO<sub>2</sub> layers.

The 850°C annealing in air has revealed a new absorption band at 460 cm<sup>-1</sup> which is also specific to

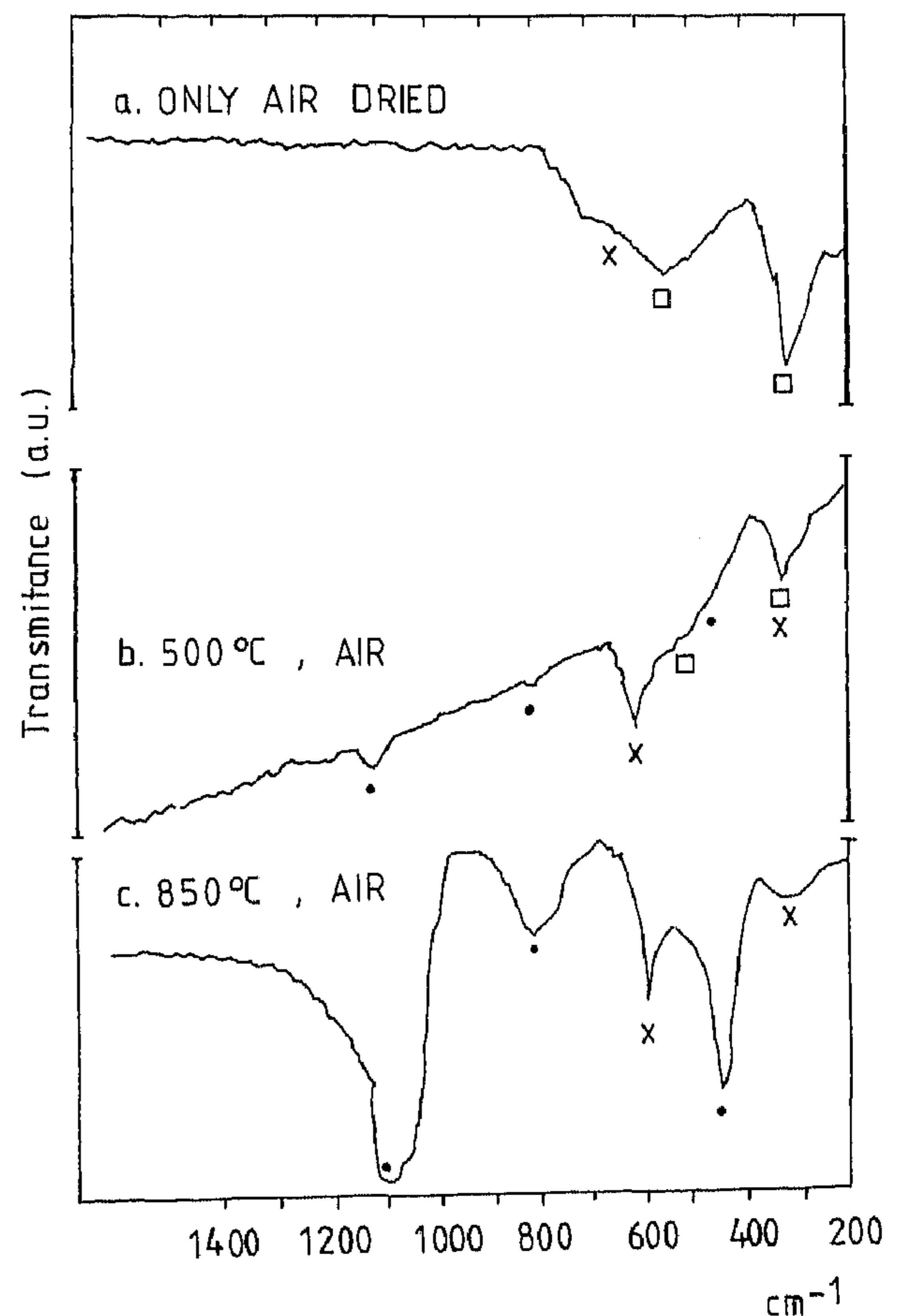


Fig. 1. Temperature evolution of infrared absorption spectra of the sol-gel SnO<sub>2</sub> films deposited on monocrystalline silicon. <sup>a</sup> □, Sn–O bond vibrations; <sup>b</sup> ×, Sn–O–Sn bond vibrations; <sup>c</sup> •, Si–O–Si bond vibrations.

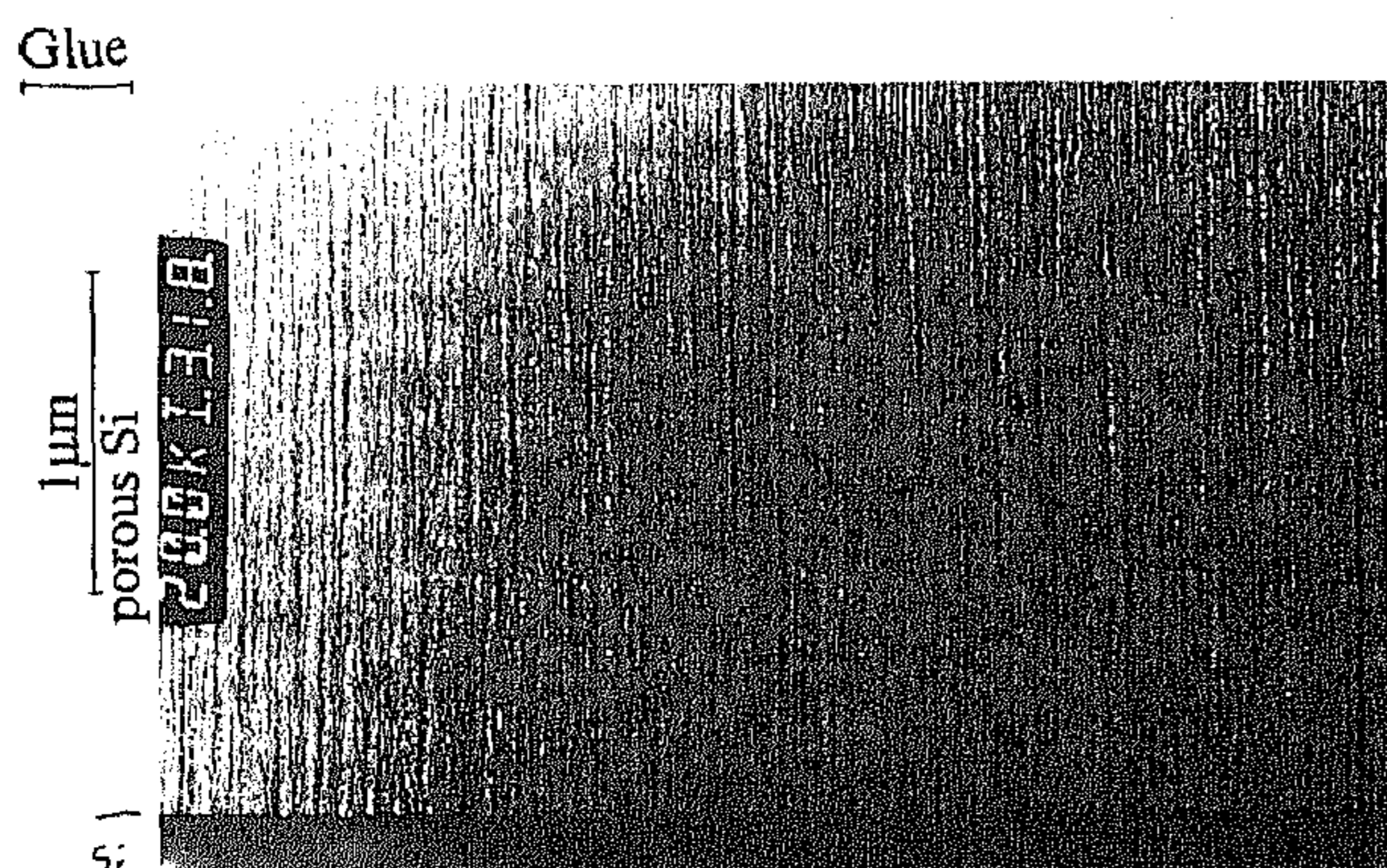


Fig. 2. XTEM image of the porous silicon after  $\text{SnO}_2$  film sol-gel deposition. The thickness of the PS is about  $2.2 \mu\text{m}$ .

$\text{SiO}_2$  films. Therefore, the IR spectroscopy has shown very clearly the evolution of the  $\text{SiO}_2$  layer that appears at interface between  $\text{SnO}_2$  and silicon during the thermal annealing of  $\text{SnO}_2$  film consolidation.

The XTEM image (40 K magnification, obtained by two-fold photo magnification of the 20 K value given by TEM apparatus) of the porous silicon after  $\text{SnO}_2$  sol-gel deposition is presented in Fig. 2. According to this picture, the pores structure is mainly columnar and oriented on the  $\langle 100 \rangle$  crystalline direction, while the thickness of the porous layer is about  $2.2 \mu\text{m}$ . The contrast between the porosified silicon (upper side) and monocrystalline silicon (dark, at the bottom of Fig. 2) is quite evident. The very small dimension of the pores is associated with the high boron doping level of the silicon wafer and low anodisation current. At this level of pore dimensions, it is not possible to 'see' by direct observation (by means of the differences in the image contrast) the  $\text{SnO}_2$  film on the walls of the PS.

The XRD analysis indicated that the crystallization of  $\text{SnO}_2$  started at annealing temperature of  $450^\circ\text{C}$ , the crystalline structure being of rutile type after a thermal annealing of  $850^\circ\text{C}$  for 30 min. Electron diffraction (ED) pattern of the  $\text{SnO}_2$  deposited on PS/Si substrates and annealed at  $500^\circ\text{C}$  is presented in Fig. 3. Practically, the same image was obtained for PS/Si substrate before  $\text{SnO}_2$  deposition indicating that either the  $\text{SnO}_2$  inside the pores is too thin or the layer crystallization is incipient, or both of them.

In order to analytically investigate the presence of the Sn atoms in the pores of the PS/Si substrates on which  $\text{SnO}_2$  from tin (IV) ethoxide was deposited, the energy dispersive X-ray spectroscopy was performed on the cross section of the porosified silicon with a small dimension beam focused in the middle side of the porosified silicon (about  $1 \mu\text{m}$  below the surface level). The EDX spectrum is presented in Fig. 4. The diagram shows the presence of two peaks associated to the Sn atoms whose concentration appear to be higher than 1

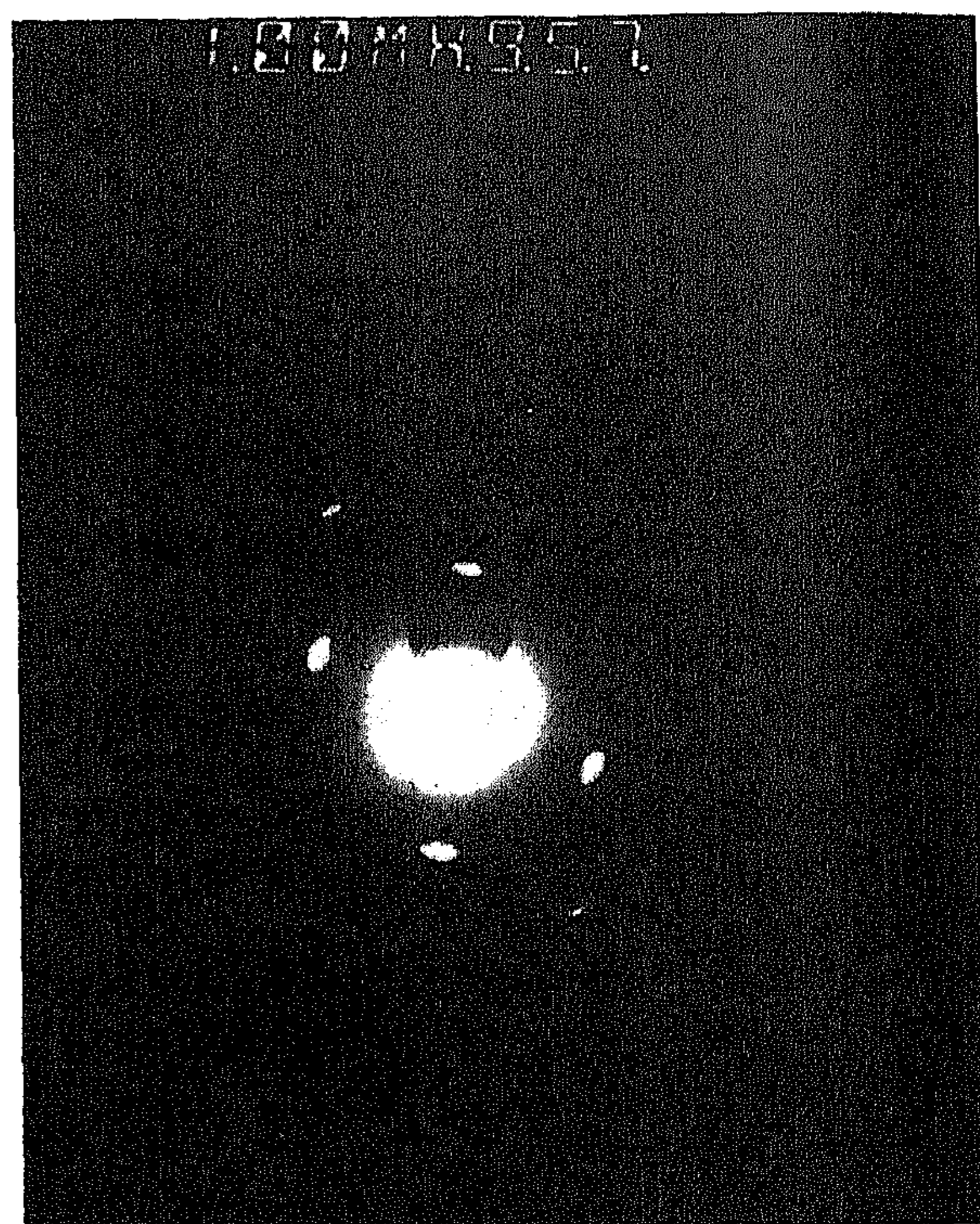


Fig. 3. Electron diffraction pattern of the porous silicon after  $\text{SnO}_2$  film sol-gel deposition.

at.% and which are well above the detection limit of the equipment. The oxygen present in the spectrum can be assigned to both thermal silicon dioxide and tin dioxide existing in the pores according to Sn detected. In fact, the increased oxygen quantity found by EDXS is a

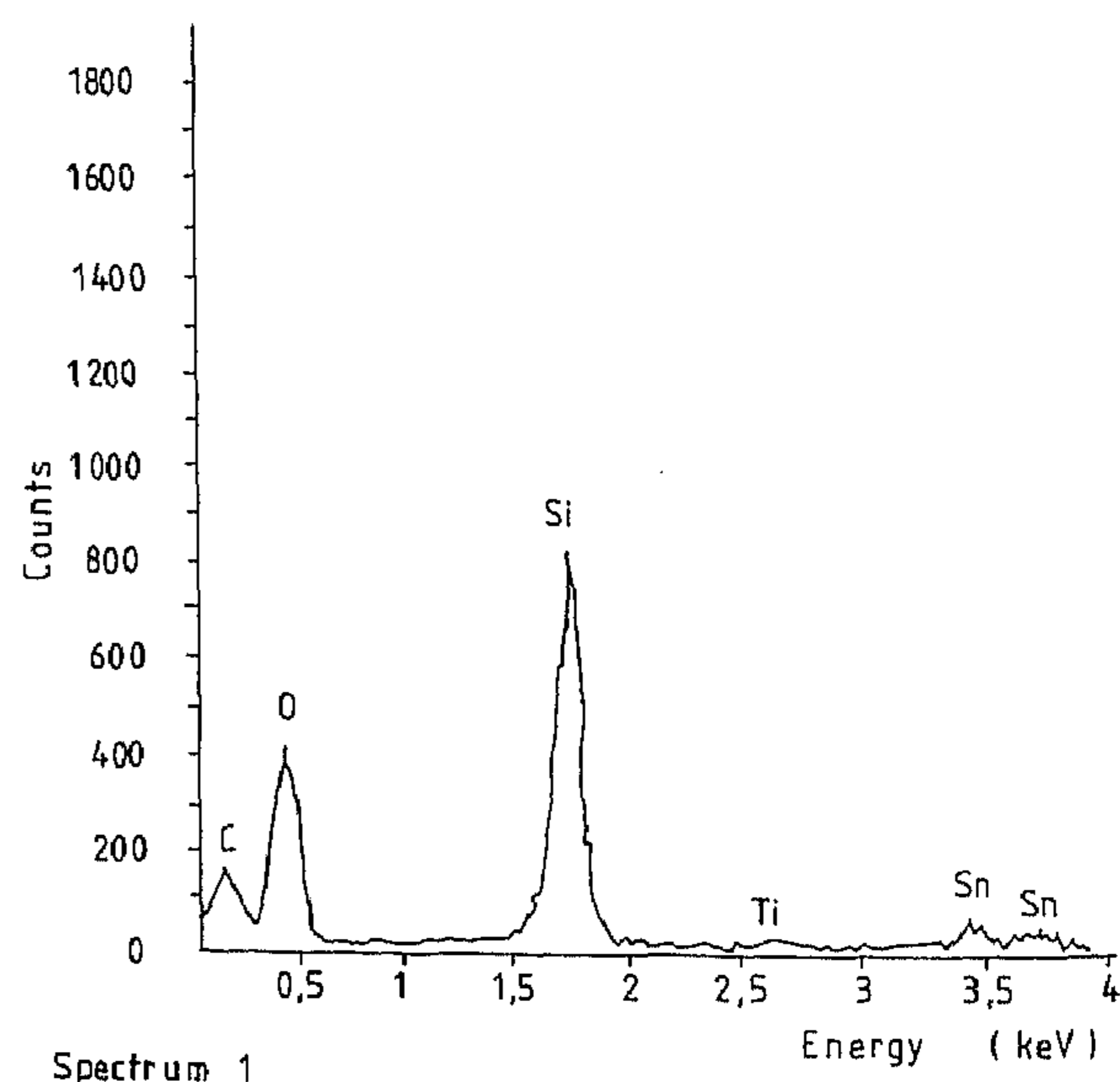


Fig. 4. EDX spectrum of the porous silicon after  $\text{SnO}_2$  sol-gel deposition proving the presence of the Sn atoms in the middle part of the PS.

basic feature of the porous silicon layers exposed to oxidant ambient. In this case, the consolidation annealings (in oxygen ambient) of the SnO<sub>2</sub> film deposited on porous silicon can explain the enhanced thermal oxidation at the interface between PS and SnO<sub>2</sub>. The carbon peak from about 0.3 keV is an indication of the carbon introduced by EDXS chamber. The oxidation could take place especially in the first stages of SnO<sub>2</sub> densification when its solid matrix is also very porous. These results are in agreement with the IR results described in Fig. 1. These initial results confirm the capability of the sol-gel method to synthesise (from tin (IV) ethoxide) polymeric sol with solid 'particles' of molecular dimensions able to penetrate in the nanometric size pores of PS/Si.

## 5. Conclusions

Transparent, crack free, undoped and Sb-doped SnO<sub>2</sub> films were prepared by sol-gel method from tin (IV) ethoxide (SnO<sub>2</sub> synthesis from this precursor was not reported before) with SbCl<sub>3</sub> as a doping source. The layers were deposited on Si, SiO<sub>2</sub>/Si and porous silicon/Si substrates. Based on TEM/EDXS analysis, the capability of the SnO<sub>2</sub> sol phase (from tin (IV) ethoxide) to penetrate (as deep as 1 μm) in the nanometric pores of the PS has been evidenced.

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## Biographies

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*Dan Dascalu* received the M.Sc. and Ph.D. degrees in electronics from Technical University of Bucharest in 1965 and 1970, respectively. From 1965 he served the Department of Electronics and Telecommunications from Technical University of Bucharest, where he has been a full professor from 1990. From 1968 to 1969, he was a research fellow at the University of Birmingham, UK. The industrial and academic activity of Professor Dascalu covers the semiconductor electron devices: technological research of metal–semiconductor contacts in microelectronics, prototyping of microwave digital radios, microwave generating diodes, space-charge-limited devices, physics and technology of microsystem. He is the author of three scientific monographs and of over 150 papers published or communicated at international conferences. Since 1991, Professor Dascalu has served also as general manager of the Institute of Microtechnology (former Center of Microtechnology), coordinating the linkage between microelectronics and microtechnology. From 1993, he has been a full member of Romanian Academy of Science. Professor Dascalu is coordinating different joint European projects within TEMPUS, PECO, NEXUSEAST, NEXUSPAN and COPERNICUS programmes. He is a senior member of IEEE. His present research interests focus on microelectronics, microsensors, microsystems for biomedical applications.

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