ISSN 1330–0008
CODEN FIZAE4

GRAZING-INCIDENCE SMALL-ANGLE SCATTERING OF SYNCHROTRON RADIATION ON NANOSIZED TiO_2 THIN FILMS OBTAINED BY CHEMICAL VAPOUR DEPOSITION AND SPRAY METHOD

MAGDY LUČIĆ-LAVČEVIĆ[1], ALEKSANDRA TURKOVIĆ[2], DAMIR ŠOKČEVIĆ[2], PAVAO DUBČEK[2,4], OGNJEN MILAT[3], BOŽIDAR ETLINGER[1], PETER LAGGNER[5] and HEINZ AMENITSCH[4,5]

[1]*Department of Physics, Faculty of Technology, University of Split, Teslina 10/V,*
21000 Split, Croatia

[2]*Institute "Rudjer Bošković", P. O. Box 1016, 10000 Zagreb, Croatia*

[3]*Institute of Physics, University of Zagreb, P. O. Box 304, 10000 Zagreb, Croatia*

[4]*Sincrotrone Trieste S.c.p.A., ss. 14, km 163,5 Basovizza, 34012 Trieste, Italy*

[5]*Institute for Biophysics and X-ray Structure Research, Austrian Academy of Sciences, Steyrerg 17,*
8010 Graz, Austria

Received 16 February 1998; revised manuscript received 17 September 1998
Accepted 21 September 1998

Nanosized TiO_2, 1 μm thick, films on glass substrate were prepared using chemical vapour deposition (CVD) and spray method procedure. The average grain size $<R>$, obtained by grazing-incidence small-angle scattering of synchrotron radiation (GISAXS) varied with the annealing temperature (500 to 900 °C in H_2, O_2 and N_2) for the CVD prepared samples. For TiO_2 films obtained by the spray method, $<R>$ increased with annealing temperature from 4.2 to 7.2 nm. Specific surface areas of both films were also determined and varied from $0.18 \cdot 10^7$ to $3.2 \cdot 10^7$ cm^{-1}.

PACS numbers: 68.55.-a, 68.15.Ef, 68.15.Rs UDC 538.975

Keywords: TiO_2 thin films, chemical vapour deposition, deposition by spraying, annealing, size of grains, GISAXS

1. Introduction

At the conference MatTech'90-Finland, we presented an Ag/AgI/TiO_2,SnO_2 rechargeable, photosensitive, electrochromic galvanic cell [1]. Its performance is based on photon or electrically induced Ag^+ ion diffusion in TiO_2. Our research, which was actually a search for a rechargeable battery for solar-cells back-up and resulted with a solar battery, was a small part of the world-wide quest for the clean and renewable electrical energy sources. This ecological idea has encouraged large research activities and development in the field of photovoltaic solar cells in the last twenty years [2]. Photosensitive cathode in our cell was constructed of TiO_2 thin film on the glass support obtained by chemical vapour deposition (CVD) and spray methods [3,4].

In 1991, Grätzel and co-workers (see e.g. Ref. 5) made a breakthrough in preparing an efficient dye-sensitized cell, which was prepared of relatively unclean material using a cheap preparation procedure, and reached the energy-conversion efficiency ranging from 7 to 12%. For us, it was a great satisfaction to learn that TiO_2 semiconductor electrodes used in the cell were the same material that we had used in our photo-sensitive galvanic cell [1,3]. Also, the application of AgI [6] as the dye [5], to TiO_2 has produced a shift of the absorption maximum to the visible region of the solar spectrum. The performance of the cell also depends on porosity of nanosized TiO_2 electrode, which produces a 2000-fold increase of the surface area [5].

As the construction of the solar cell requires a solid film formed on glass substrate, it is imperative to investigate morphology of the material prepared in the thin film form as it might differ in some properties from the powder form, which was previously studied by different experimental methods [7]. Also, in order to obtain an efficient solar cell, the TiO_2 thin film on the glass substrate should have a high surface area [8].

In order to determine the grain size and specific surface area of TiO_2 thin films on glass substrates, prepared by the chemical vapour deposition [3] and by the spray method [4], we have performed grazing-incidence small-angle X-ray scattering (GISAXS) measurements using synchrotron radiation at ELETTRA, Trieste, which has a thousand times higher intensity than a laboratory X-ray tube. The results of these measurements are presented in this work.

2. Experimental

The experimental procedures of the preparation of TiO_2 thin films by CVD and spray methods were described in previous publications [3,4]. Thin films were formed with tape casting method on glass substrates with spherical curvature. The surface area of thus obtained films was of about 1 cm^2.

GISAXS measurements were performed at the new SAXS beamline at the synchrotron ELETTRA, Trieste [9,10]. The beamline was built by the researchers from the Institute for Biophysics and X-ray structure Research (IBR), Austrian Academy of Science. Its characteristics are: photon-beam wavelength of $\lambda = 1.55 \cdot 10^{-8}$ cm, energy resolution $\Delta E/E \leq 2.5 \cdot 10^{-3}$, focal spot size FWHM 1.2×0.6 mm^2, spot at the sample 5.4×1.8

mm^2 and flux at the sample of 5×10^{12} photons/s (electron beam energy 2 GeV, beam current 200 mA and X-ray photon energy of 8 keV).

GISAXS is the experimental technique that combines both grazing incidence and scattering at small angles. As a new tool, it was first proposed by the Northwestern University group of professor J. Cohen [11]. This technique has been developed and applied to morphological characterization of aggregates deposited or gathered on a flat substrate and of nanostructure of thin films [12–14]. We have applied GISAXS for measuring nanosized TiO_2 thin films on glass substrate [15,16]. In the first position, the incoming X-ray beam was barely touching the surface i.e. it was tangential to the surface of the film. The position of the sample was gradually rised until the film surface was 0.02 mm inside the beam. In this geometry, the path of X-rays through the thin film is much longer than for a standard transmission geometry, and the substrate signal is weak. However, in this geometry, the totally reflected power is superimposed on the scattering signal at higher values of $q = 4\pi\Theta/\lambda$ [15].

The GISAXS intensity, recorded by a position sensitive detector, is given as a function of q in the interval from $q = 1/25$ nm^{-1} to $q = 1/1.25$ nm^{-1}. The scale q(nm^{-1}) versus channel number is determined by the rat-tail standard [9].

3. Results and discussion

3.1. GISAXS in TiO_2 thin films on the glass substrate

Thin TiO_2 film on the glass substrate can be considered as an aggregate containing TiO_2 nanosize grains and nanosize pores [17]. In this case, the SAXS is caused by the difference of electron density within and around the nanosize grains. Using the Guinier approximation [18], the grain size can be readily determined. The limiting angle Θ_m of the small-angle diffuse scattering is approximately $\Theta_m = \lambda/(2D)$ where D is the largest grain diameter. Porod approximation [19,20] is suitable to determine specific surface area of nanosized thin films. In our previous measurements [17] with a laboratory X-ray source, the intensity of the recorded signal was to small to get the part of curve relevant for Porod approximation. With the high intensity synchrotron radiation sources, the scattered intensity is so high that we can apply both approximations and obtain both relevant parameters, i.e., the grain size and the specific surface of TiO_2 thin films.

Figure 1 represents the data in a $\log I$ vs. Θ^2 plot as a test whether one can apply the above mentioned Guinier law: $I(q) = (\Delta\rho)^2 \exp(-R_g^2 q^2/3)$ for small q, where $q = 4\pi\Theta/\lambda$ and $\Delta\rho$ is difference of electron density between grain and surrounding medium. The average grain radius can be estimated from the radius of gyration R_g in the Guinier formula; this was calculated from the slope of the linear fit of $\log I$ vs. Θ^2 for different annealing temperatures and in different atmospheres. From these fitting lines, we have obtained R_g and the average grain radius $<R>$ using $<R>= \sqrt{5/3}R_g$ (for spherical shape). It can be seen from Fig. 1 that the Gaussian form is valid up to $(2\Theta)^2 = 5 \cdot 10^{-5}$ rad^2, corresponding approximatively to $q = 0.3$ nm^{-1}. The approximation is very good (within 5%) up to $qR_g = 2\pi \times 0.4$ for grains of spherical shape.

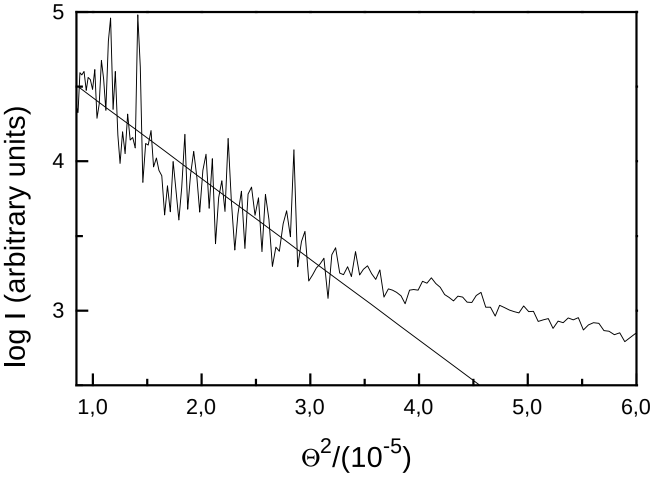

Fig. 1. Linear fit $\log I = f(\Theta^2)$ *to GISAXS data of the CVD-prepared sample, annealed in* N_2*, giving:* $\log I = -54140\ \Theta^2 + 4.97$ *and* $R = 9.7$ nm.

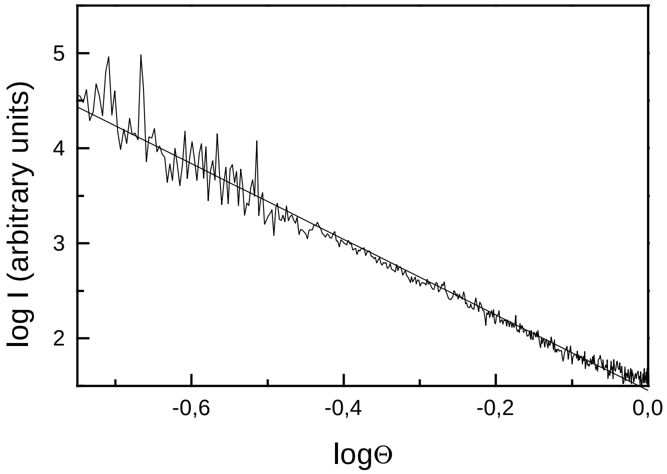

Fig. 2. Representation of the data as $\log I = f(\log\Theta)$ *as a test whether we can apply the Porod's formula.*

The fitting line in Fig. 2 with a slope of -4 indicates that the Porod law $I(q) = 2\pi(\Delta\rho)^2 S/q^4$ can be applied for larger values of q. In the case of samples obtained by CVD and spray methods, it is a great certainty that a size distribution of grains gives the log-log plot with a slope of -4. Our assumption is based on our previous measurements of TEM on sol-gel obtained TiO_2 thin films [7], revealing the Gaussian distribution of spherical grains. Taking that into account, the “specific surface area” S_s can be calculated from the Mittelbach-Porod formula [19,20]:

$$S_s = S/V = \pi \lim_{q\to\infty} \left(q^4 I(q)\right)/Q, \qquad (1)$$

where Q is the invariant integral

$$\int_0^\infty q^2 I(q)\mathrm{d}q,$$

which we have approximated by the value Q_{max} defined as

$$Q_{max} = \int_{q_{min}}^{q_{max}} q^2 I(q)\mathrm{d}q,$$

where $q_{max} = 10^7$ cm^{-1}.

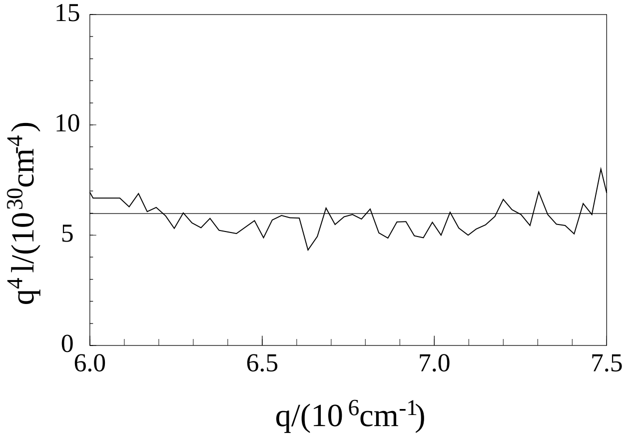

Fig. 3. Representation of data for large values of q ($q = 4\pi\Theta/\lambda$). *In this region, asymptotically* $q^4I = 2\pi q^2 S$. *The value of* q^4I *derived from our graph is* $6.2 \cdot 10^{30}$ cm^{-4}.

Figure 3 presents the data in the $q^4I(q)$ vs. q plot for large values of q. For $q \geq 6 \cdot 10^6$ cm^{-1}, a constant value of $q^4I = 2\pi\rho^2 S$ is used to calculate the specific surface area from formula (1) for each film.

3.2. GISAXS in thermally annealed TiO_2 *thin films obtained by chemical vapour deposition*

Among a number of available methods for depositing TiO_2 thin films, chemical vapour deposition (CVD) is promising since high deposition rates can be obtained at low substrate temperatures. In this work, we use simple CVD method for fabrication of TiO_2 thin films. Air was used as the carrier gas. Its flow through a pipe was at a rate of 10 ml/min. The $TiCl_4$ vapour passed through another pipe to a clean substrate (SiO_2) placed on a plate mantained at 370 °C. At the substrate surface, $TiCl_4$ molecules received enough energy to interact with the moisture from the air:

$$TiCl_4 + 2H_2O = TiO_2 + 4HCl\,. \qquad (2)$$

The growth rate of TiO_2 thin films is typically 5 nm/s. The film thickness of the films was of the order of 1 μm and was determined using a Tencor-alpha-step instrument. Since the samples have been produced in atmospheric conditions, they have been transported without any special treatment and inserted in the SAXS set-up.

In the literature [21], there is the evidence of an intermediate phase occuring prior to the phase transition temperature in solids. In nanosized materials, such an intermediate phase tends to be in the amorphous state [21]. The phase transition temperature gets lower with smaller sizes of nanograins [21]. In our previous work [3,4] on Raman spectra of CVD-prepared samples annealed in oxidizing and non-oxidizing atmospheres, we have observed both effects: the occurrence of an amorphous phase, and the difference in phase transition temperature regarding the atmosphere in which sample was annealed. Raman spectra are shown in Fig. 4. In the low temperature anatase phase, the strongest characteristic Raman line is positioned at 144 cm^{-1}. Above the phase transition temperature of 830 °C, for sample annealed in hydrogen, and 900 °C for samples annealed in non-oxydizing atmospheres, the high temperature rutile phase is characterized with a 443 cm^{-1} strong peak. At 800 °C, an amorphous phase

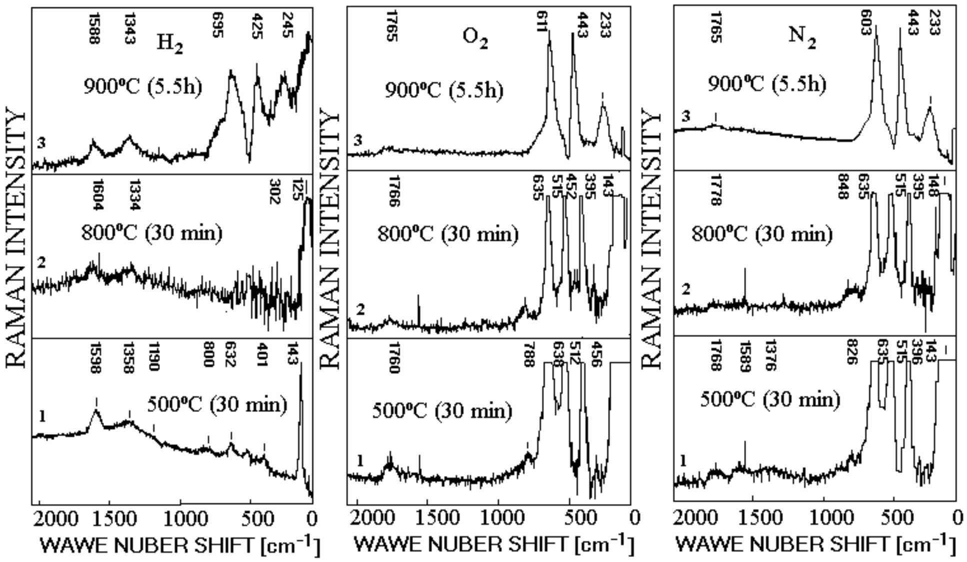

Fig. 4. Raman spectra of CVD-obtained TiO_2 *films annealed for 0.5 or 5.5 hours in: a) hydrogen, b) oxygen and c) nitrogen atmospheres [3].*

occurs in the sample annealed in hydrogen. All these facts have indicated that we have nanosized TiO_2 thin films with different grain sizes for different annealing atmosphere. In order to prove that, we have performed GISAXS measurements of grain sizes and specific surfaces on the materials.

TABLE 1. CVD-samples annealed in hydrogen, oxygen and nitrogen of thickness $d =$ 1 μm, annealing temperature, annealing time, "average grain radius" $<R>$ obtained from the linear fit with an accuracy of about 0.15 nm *and "specific surface area" S_s, as determined from GISAXS.*

Sample	Annealing temperature (°C)	Annealing time (hours)	SAXS $<R>$ (nm)	S_s (10^7 cm^{-1})
H_2J3	500	0.5	5.2	0.57
H_2J4	800	0.5	5.7	0.29
H_2J6	830	0.5	7.3	0.34
H_2J7	900	5.5	5.2	1.0
O_2K1	500	0.5	8.8	0.17
O_2K2	800	0.5	7.1	0.34
O_2K4	900	5.5	3.5	0.15
N_2L1	500	0.5	9.7	0.31
N_2L2	800	0.5	8.1	0.22
N_2L3	900	5.5	5.9	0.34

Comparison of values of S_s and $<R>$ obtained by GISAXS for various annealing temperatures in hydrogen, nitrogen and oxygen atmosphere of the TiO_2 films prepared by CVD procedure is given in Table 1. In hydrogen atmosphere, we can see two trends, the first of increasing of the grain size up to the annealing temperature of 830 °C, and the second of decreasing after the anatase-rutile phase transition took place (temperature 900 °C, annealing time 5.5 hours). The reason for a longer annealing time was the fact that we had still a small amount of anatase after an annealing time of 30 min at this temperature. After a longer time of annealing, this part of the sample has transformed to the rutile phase, which was evident by the disappearance of the 144 cm^{-1} line. Such a behaviour is characteristic of thin films, as in the bulk solids phase transition occurs in a short time interval, i.e., in TiO_2 powder obtained by the sol-gel method, the phase transition recorded by XRD occurs at 850 °C [22]. $<R>$ varies from 5.2 nm to 7.3 nm and then to 5.2 nm. This correlation of grain sizes to annealing temperatures (see Table 1) is given in Fig. 5a (curve 2). In the same figure, we can observe an opposite trend for surface area (curve 1). It has a minimum at 800 °C. This behaviuor can be compared with

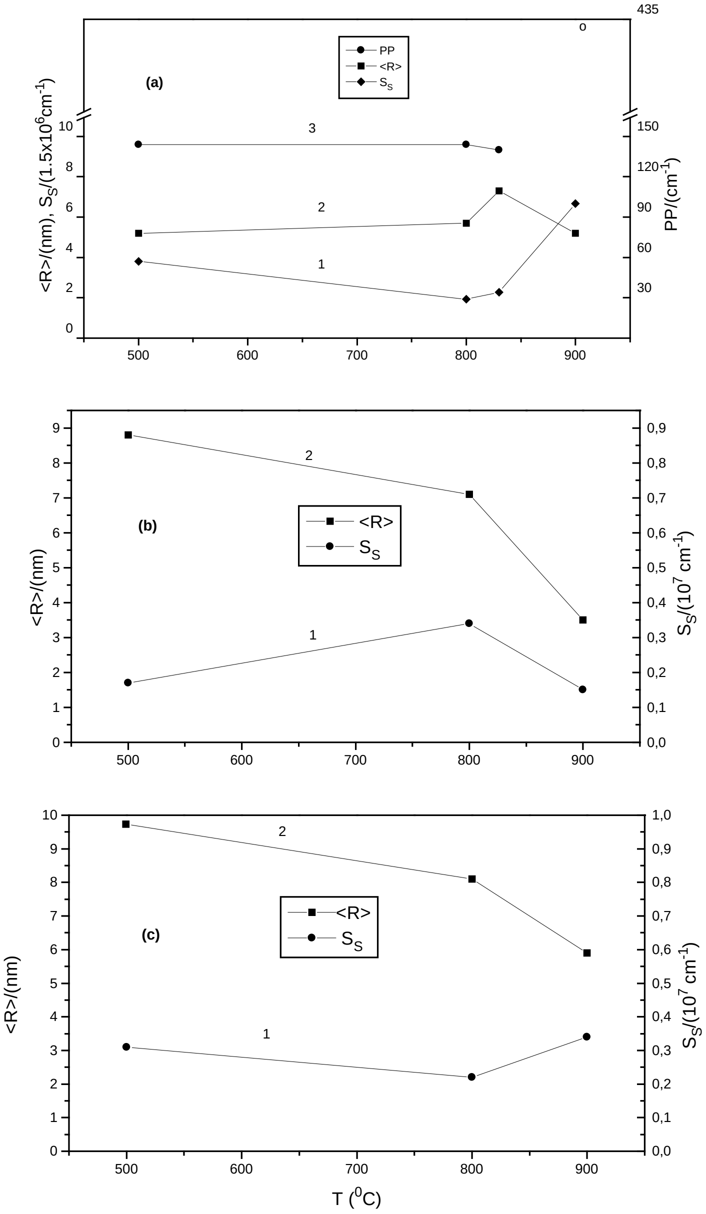

Fig. 5. (1) Specific surface area, (2) average grain size and (3) "144 cm^{-1}*" (black circles) and "443* cm^{-1}*" (white circle) peak positions, PP, of Raman lines as a function of temperature for CVD-prepared samples annealed in (a) hydrogen, (b) oxygen and (c) nitrogen atmosphere.*

our previous Raman measurements [3] of the TiO_2 thin films annealed under the same conditions, as is shown in Fig. 4. Thin films annealed in hydrogen atmosphere, different from the films annealed in other atmospheres, show the amorphous phase at 800 °C. At this temperature, in Fig. 5a "the average grain radius" $<R>$ shows the beginning of a pronounced peak, which does not occur during annealing in oxygen and nitrogen atmospheres (Figs. 5b and c). Also, the grain sizes in the samples annealed in oxygen and nitrogen are bigger than of the samples annealed in hydrogen. This actually accounts for the higher phase transition temperature in these samples. Specific surface curve can be correlated with the peak position, (PP) of "144 cm^{-1}" and "443 cm^{-1}" Raman lines, shown in Fig. 5a as a function of temperature, which were published in our previous work [4]. It is shown by the curve 3 in Fig. 5a and represents the non-stoichiometry induced by hydrogen treatment. The first part of the curve denoted by black circles is anatase phase of TiO_2 and above 900 °C white circle is denoting rutile phase. As the trend in curve 3 can be correlated to curve 1 (S_s), we can conclude that the specific surface reflects the non-stoiochiometry induced by the hydrogen treatment. Specific surface in hydrogen-annealed samples is higher than in the samples annealed in oxygen and nitrogen. By the process of annealing in hydrogen, oxygen atoms were extracted from TiO_2, inducing non-stoichiometry, lowering grain sizes and increasing specific surface i.e. porosity of material.

3.3. GISAXS in TiO_2 *thin films obtained by spray method*

Thin films of TiO_2 have been prepared by the spray method at the substrate temperature of 370 °C from a commercial (Merck) solution of 15% $TiCl_3$ in 15% HCl (Merck) with 20 vol.% of ethyl alcohol.

TABLE 2. Samples obtained by the spray method, annealed in hydrogen, of thicknesses $d = 1$ μm*, annealing temperature, annealing time, "average grain radius"* $<R>$ *obtained from linear fit with an accuracy of about 0.15* nm *and "specific surface area"* S_s*, as determined from GISAXS.*

Sample	Annealing temperature (°C)	Annealing time (hours)	SAXS $<R>$ (nm)	S_s 10^7 cm^{-1}
S_pI1	100	0.5	4.7	2.0
S_pI2	200	0.5	4.2	2.5
S_pI3	500	0.5	5.0	3.0
S_pI4	700	0.5	5.0	2.6
S_pI5	720	0.5	5.6	2.4
S_pI6	740	0.5	5.2	2.8
S_pI7	790	0.5	5.4	2.8
S_pI8	800	0.5	7.2	0.80
S_pI9	900	0.5	6.6	0.88
S_pI10	900	5.5	4.4	3.2

In our previous work [4], we have measured Raman spectra of thermally annealed TiO_2 films obtained by the spray method in a non-oxidizing atmosphere of H_2. We have shown that these films have a higher non-stoichiometry than CVD-prepared samples, which can be confirmed by our GISAXS measurements showing higher specific surface area for spray-prepared samples (Table 2). Our correlation curves of $<R>$ and S_s are shown in Fig. 6.

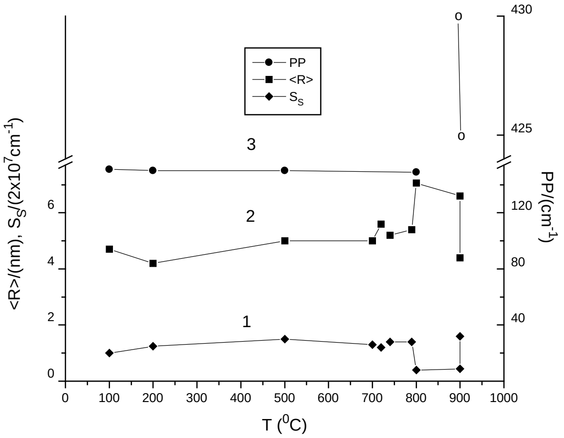

Fig. 6. (1) Specific surface area, (2) average grain size and (3) "144 cm^{-1}*" (black circles) and "443* cm^{-1}*" (white circles) peak positions, PP, of Raman lines as a function of temperature for the spray-method-prepared samples annealed in hydrogen atmosphere.*

Comparison of S_s and $<R>$ values for various annealing temperatures in hydrogen atmosphere of the TiO_2 films prepared by spray method is given in Table 2. We can see two trends, the first of increasing of the grain size, $<R>$, for annealing temperatures up to 800 °C, and the second of decreasing of the size to a lower value after 5.5 hours of annealing at 900 °C. The size varies from 4.7 nm to 7.2 nm and back to 4.4 nm. This correlation of grain sizes to annealing temperatures is shown in Fig. 6. In the figure, we can observe an opposite trend to the specific surface area. This behaviour can be interpreted in terms of the induced non-stoichiometry with the reaction:

$$2TiO_2 + H_2 = Ti_2O_3 + H_2O\,, \tag{3}$$

which we had proposed in our previous work [3]. At 800 °C, the phase transition of anatase to rutile takes place; it was observed by Raman spectroscopy and XRD in our previous work [4]. Raman spectra of the samples obtained by the spray method are shown in Fig. 7. Two of the titanium dioxide phases, anatase and rutile, have been observed in the thin film samples heated from 100 to 900 °C in H_2. The spectra show that thin films of TiO_2 were in the anatase form for temperatures of 100 to 800 °C. We were in search for the feature in the Raman spectrum of TiO_2 heated at 800 °C. In analogy with Raman spectrum of CVD-prepared thin films annealed in hydrogen, we expected formless features which actually resemble the spectrum of the amorphous phase which was observed by Bobovich and Center [21] as a general process near the phase transition for nanosized thin films. We have even searched for the intermediate amorphous phase in the range from 700 to 790 °C in small temperature steps of 20 to 30 °C, but amorphous phase was not observed. Our assumption was that different morphologies of the samples obtained by the two methods account for the existence or lack of the amorphous phase. After 5.5 h of annealing at 900

°C, the anatase-rutile phase transition was completed.

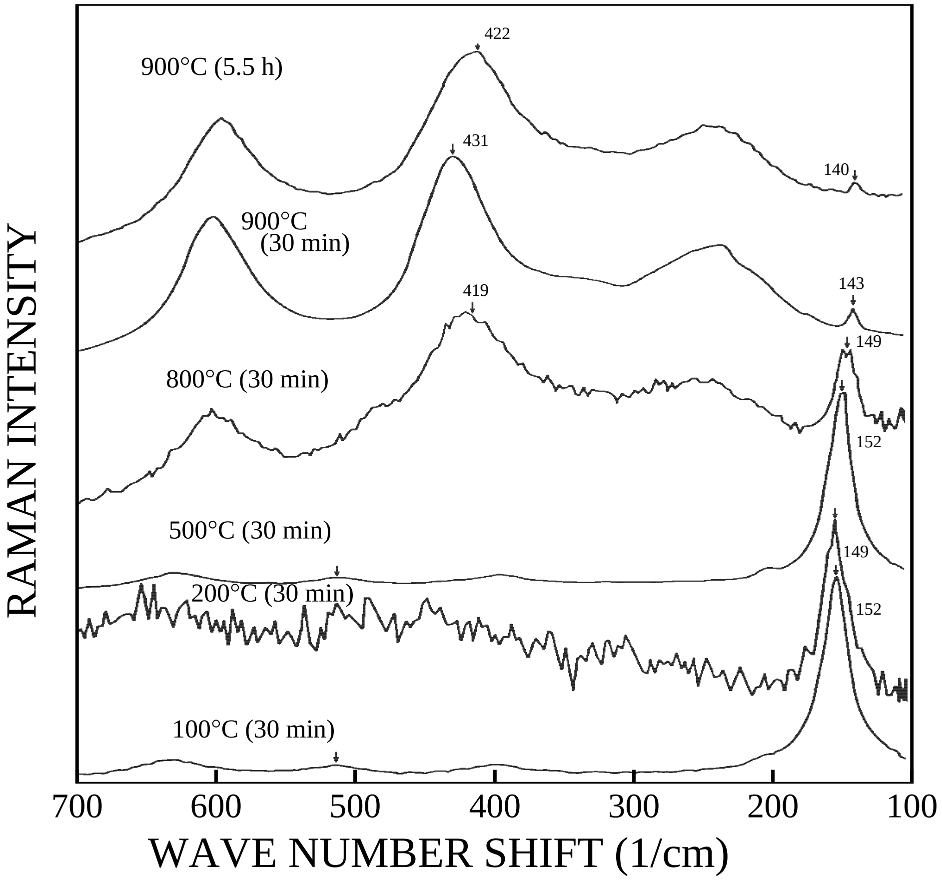

Fig. 7. Raman spectra as a function of temperature for spray-prepared samples annealed for 0.5 or 5.5 hours in hydrogen atmosphere [4].

The specific surface area S_s had the highest value for the film annealed at 500 °C. For the whole range of annealing temperatures, S_s varied from $2.0 \cdot 10^7$ cm^{-1} to $3.2 \cdot 10^7$ cm^{-1}. This shows that TiO_2 films obtained by the spray method have a higher surface area than the previously discussed CVD-prepared material. This is in agreement with the above discussion about the difference in morphology between these two films [4]. Also, the grain sizes in the samples obtained by the spray method are lower than in the

CVD-obtained samples and, accordingly, the phase-transition temperature is lower. It is 800 °C, and for the CVD-obtained samples it is 830 °C.

The specific surface S_s and grain size $<R>$ behaviour below 800 °C are resembling those of CVD-obtained samples. They indicate the possibility of amorphous phase in the region from 700 to 800 °C. We can not draw this conclusion solely from the GISAXS data. Wide-angle X-ray scattering (WAXS) measurements at ELETTRA will be performed with the aim to find the solution of this puzzle.

4. Conclusion

The present study showed that GISAXS measurements at ELETTRA light source can be applied for the grain-size determination on nanosized thin films of TiO_2 on glass substrate. Compared to other methods [17] for grain size determination, GISAXS is suitable for specific surface determination, which is the crucial parameter for efficient solar cells. GISAXS measurements have also confirmed our previous Raman findings [4] about different morphologies in TiO_2 films obtained by CVD and by spray methods of preparation. Also, we have shown that films obtained by both ways are nanosized films, with a high surface area characteristic for porous materials [5,8].

References

1) A. Turković and V. Vraneša, Int. J. of Mat. Prod. Techn. **47** (1992) 51;
2) K. Zweibel, *Harnessing Solar Power; The Photovoltaic Challenge*, Plenum Press, New York, 1990;
3) A. Turković, M. Ivanda, A. Drašner, V. Vraneša and M. Peršin, Thin Solid Films **198** (1991) 199;
4) A. Turković, M. Ivanda, V. Vraneša and A. Drašner, Vacuum **43** (1992) 471;
5) B. O'Regan and M. Grätzel, Nature **353** (1991) 737;
6) A. Turković, N. Radić and D. Šokčević, Mat. Sci. Eng. **B 23** (1994) 41;
7) A. Turković, A. M. Tonejc, S. Popović, P. Dubček M. Ivanda, S. Musić and M. Gotić, Fizika **A 6** (1997) 77;
8) A. Hagfeldt and M. Grätzel, Chem. Rev. **95** (1995) 49;
9) H. Amenitsch, S. Bernstorff and P. Laggner, Rev. Sci. Instrum. **66** (1995) 1624;
10) H. Amenitsch, M. Rappolt, M. Kriechbaum, H. Mio, P. Laggner and S. Bernstorff, *Small Angle X-ray Scattering - Beamline at ELETTRA: A New Powerful Station for Structural Investigations with Synchrotron Radiation*, Book of abstracts: *Sixth Croatian-Slovenian Crystallographic Meeting* 56, Umag, Croatia, June 19-21, 1997;
11) J. R. Levine, J. B. Cohen, Y. W. Chung and P. Georgopoulos, J. Appl. Cryst. **22** (1989) 528;
12) A. Naudon and D. Thiaudiere, Surface and Coat. Tech. **79** (1-3) (1996) 103;
13) D. Thiaudiere and A. Naudon, J. de Physique **IV, 6 (C4)** (1996) 553;

14) A. Naudon and D. Thiaudiere, J. Appl. Cryst. **30** (2, Part 5) (1997) 822;

15) P. Dubček, O. Milat, M. Lučić-Lavčević, A. Turković, B. Etlinger and H. Amenitsch, *Small Angle X-ray Scattering and X-Ray Reflection on Thin Films*, Book of Abstracts: *Sixth Croatian-Slovenian Crystallographic Meeting*, Umag, Croatia, June 19-21, 1997;

16) M. Lučić-Lavčević P. Dubček, O. Milat, B. Etlinger, A. Turković and H. Amenitsch, *Small Angle Scattering of Synchrotron Light on Nanophase* TiO_2 *Measured at ELETTRA*, Book of Abstracts: *Sixth Croatian-Slovenian Crystallographic Meeting*, Umag, Croatia, June 19-21, 1997;

17) A. Turković, M. Ivanda, S. Popović, A. M. Tonejc, M. Gotić, P. Dubček and S. Music, J. of Mol. Structure **410-411** (1997) 271;

18) A. Guinier, Ann. Phys. (Paris) **12** (1939) 161;

19) G. Porod, Kolloid Z. **124** (1951) 83;

20) G. Porod, Kolloid Z. **125** (1952) 108; **133** (1953) 51;

21) J. S. Bobovich and M. J. Center, Opt. Spektrosk. **62** (4) (1987) 946;

22) S. Musić, M. Gotić, M. Ivanda, S. Popović, A. Turković, R. Trojko, A. Sekulić and K. Furić, Mater. Sci. Eng. **B47** (1997) 33.

RASPRŠENJE SINHROTRONSKOG ZRAČENJA NA MALIM KUTOVIMA PRIKLONA I RASPRŠENJA U TANKIM SLOJEVIMA PRAHA TiO_2 PRIPREMLJENIM POLAGANJEM IZ PARA I RASPRAŠIVANJEM

Sloj TiO_2 nanometarske veličine zrna, debljine 1 μm, polagao se je na staklene podloge metodama kemijskog polaganja iz para i raspraševanjem. Prosječna veličina zrna, $<R>$, određena mjerenjem raspršenja sinhrotronskog zračenja na malim kutovima priklona i raspršenja, mijenjala se je s temperaturom toplinskog otpuštanja (500 do 900 °C u H_2, O_2 i N_2) za uzorke pripremljene iz para. Za uzorke TiO_2 pripremljene raspraševanjem, $<R>$ se je povećao s temperaturom otpuštanja od 4.2 do 7.2 nm. Specifična površina obje vrste tankih slojeva se je također određivala i mijenjala od $0.18 \cdot 10^7$ do $3.2 \cdot 10^7$ cm^{-1}.

æ