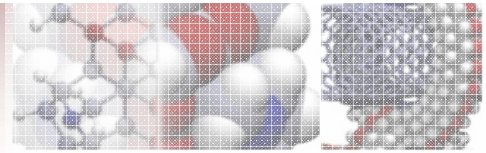

Filme de TiO_2 nanostructurate prin anodizarea Ti in electrolit pe baza de fluorura pentru aplicatii la celule solare

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IMT- Bucharest

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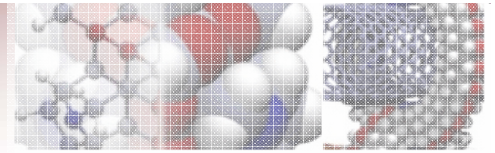
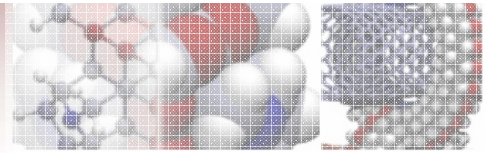


- **INTRODUCERE**
- **EXPERIMENT**
- **CARACTERIZARI**
- **CONCLUZII**



INTRODUCERE

TiO_2 este un material oxidic de tip $A_{II}B_{VI}$ cu banda interzisa larga (> 3 eV) si se prezinta in mai multe forme de cristalizare.

Poate fi obtinut cu proprietati adecvate pentru numeroase tipuri de aplicatii cum sunt in celule fotovoltaice, senzori de gaz, senzori biologici, acoperiri optice.

Metode de obtinere a straturilor de TiO_2

-Sol-gel;

-Anodizare strat metalic subtire de Ti depus pe diferite substrate;

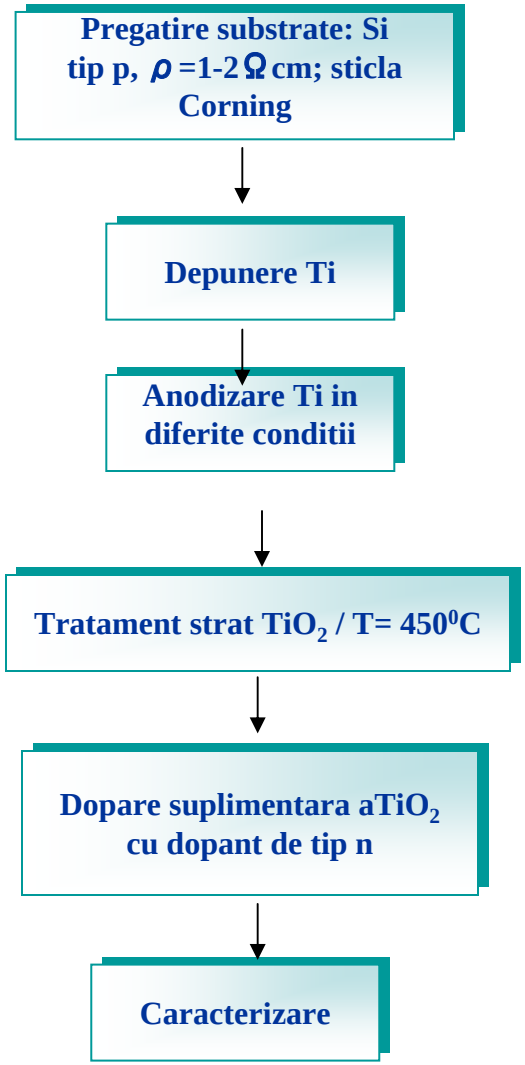
-Pyroliza – spray;

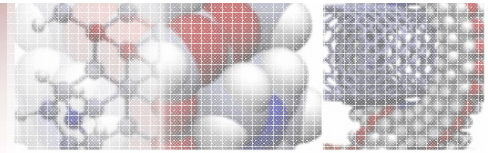
-Anodizare folie Ti pentru obtinerea de nanotuburi de TiO2.

Metoda de obtinere a TiO_2 prin anodizarea unui strat subtire de Ti este compatibila cu tehnologia celulelor fotovoltaice pe siliciu si permite controlarea nano structurarii.

Poate fi utilizata si la realizarea de celule solare pe baza de pigmenti, (DSC)

Principalele etape ale procesului de obtinere a straturilor de TiO_2 prin anodizarea titanului metalic.





DEPUNEREA FILMELOR DE TITAN

EXPERIMENT

-Tehnica utilizata: DC sputtering din tinta de Ti cu puritatea 99,9%.

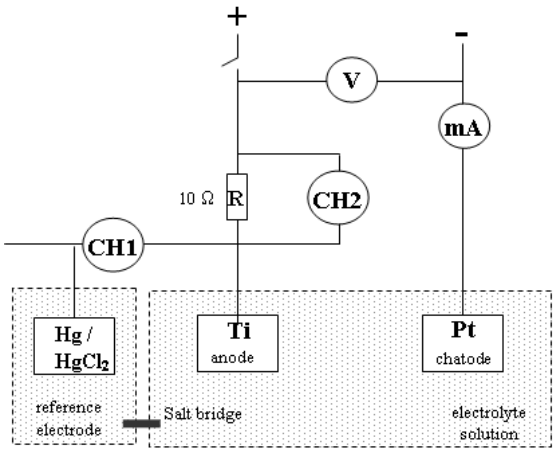
-Conditiile de depunere: presiunea in camera de depunere a fost 1.82 · 10-3 Pa utilizind un debit de argon de 2.5 sccm la o putere de 100W.

-Substraturile pe care s-au depus filme de Ti: plachete de siliciu de tip p, cu rezistivitatea de 1-2 Ω·cm, sticla Corning.

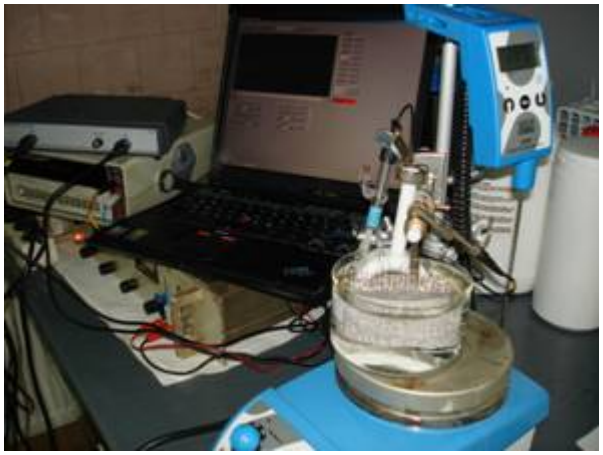
-Grosimea straturilor de Ti depuse: 90-200 nm

EXPERIMENT

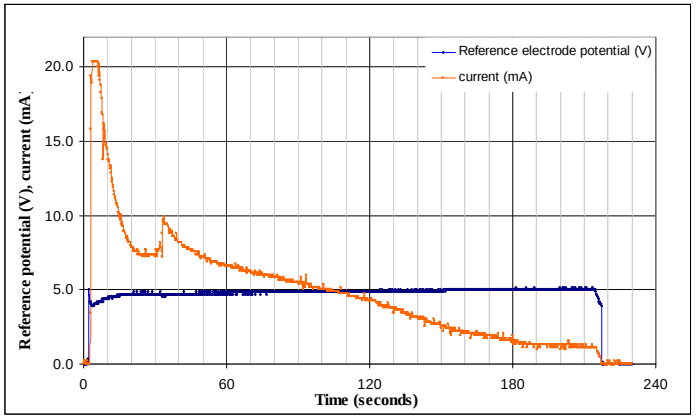
Instalatia de anodizare



CH1, CH2 - oscilloscope data acquisition channels



Dispozitiv pentru prinderea si realizarea contactului electric pe suprafata unui substrat.



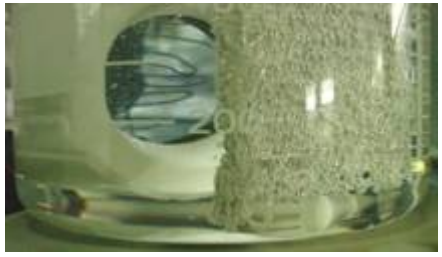
Urmarirea procesului de anodizare la V= const.

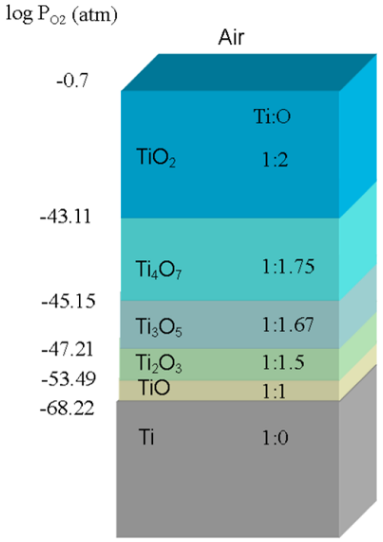
Observatii experimentale

EXPERIMENT

TiO

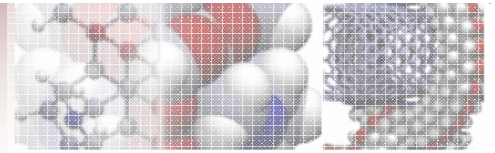
TiO_2





Ilustrarea schematica a interfetei metal-oxid la echilibru, functie de presiunea partiala a O_2 la interfata

Interfata	Reactiii
Aer/TiO_2	-
TiO_2/Ti_4O_7	$TiO_2 + 4Ti+5/2O_2 \rightarrow Ti_4O_7$
Ti_4O_7/Ti_3O_5	$Ti_4O_7 \rightarrow Ti_3O_5+Ti+O_2$
Ti_3O_5/Ti_2O_3	$Ti_3O_5 \rightarrow Ti_2O_3+ Ti+O_2$
Ti_2O_3/TiO	$Ti_2O_3 \rightarrow TiO+Ti+O_2$
TiO/Ti	$TiO \rightarrow Ti+1/2O_2$



EXPERIMENT

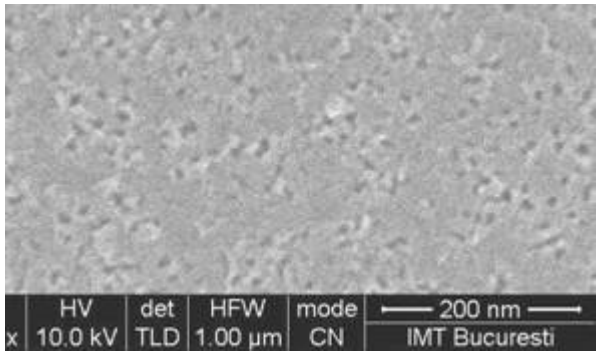
Electroliti / conditii de reactie

Nr. proba.	pH	Corectie pH	Solutie preparata	Temp (ºC)	U (V)	Timp (sec)	Tratament 450ºC
24		nu	NH_4F 0,5% sare	26,4	5	320	Da
22		nu	NH_4F 0,5% sare	26,4	10	200	Da
20		nu	NH_4F 0,5% sare	26,4	2,5	560	Da
19		nu	NH_4F 1% sare	26,4	5	130	Da
23		nu	NH_4F 1% sare	26,4	10	120	Da
25		nu	NH_4F 1% sare	26,4	2,5	340	Da
21		nu	NH_4F 2% sare	26,4	5	160	Da
1/16		nu	NH_4F 2% sare	26,4	10	40/90	Da
33	5,52	3,7% HF	NH_4F 10% sare	26,6	5	120	Da
29	4,46	3,7% HF	NH_4F 10% sare	26,6	5	120	Da
28	6,70	3,7% HF	NH_4F 10% sare	26,6	5	120	Da
31	8,42	10% NH_4OH	NH_4F 10% solutie	26,6	5 → 25	420	Da
34	6,40	3,7% HF	NH_4F 10% solutie	26,6	5	150	Da
26	6,02	3,7% HF	NH_4F 10% solutie	26,6	5	180	Da
30	7,82	10% NH_4OH	NH_4F 10% solutie	26,6	5 → 20	360	Da
41	7,08	nu	8,25%NH_4F, 30%H_2O	17	5		Da
39	6,9	nu	10%NH_4F, 52,5%H_20	20	5		Da
38	6,9	nu	10%NH_4F, 52,5%H_2O	28	5		Da
37	6,55	nu	1,44%NH_4F, 30%H_2O	26,8	5		Da
36	6,30	nu	1%NH_4F, 51,4%H_2O	26,8	5		Da

CARACTERIZAREA STRATURILOR NANOPOROASE DE TiO_2 OBTINUTE PRIN ANODIZARE PE SUBSTRATE DE SILICIU SI STICLA

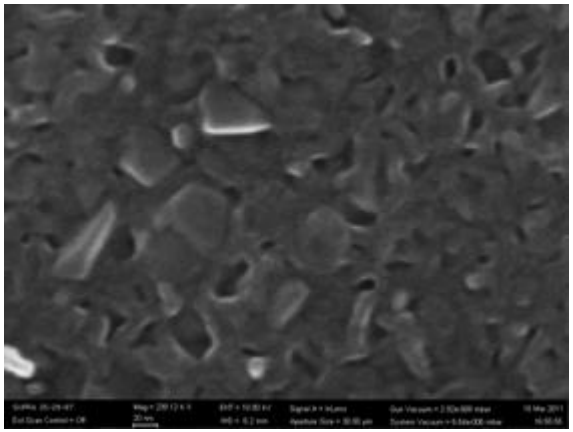
Morfologia suprafetei TiO_2 prin SEM

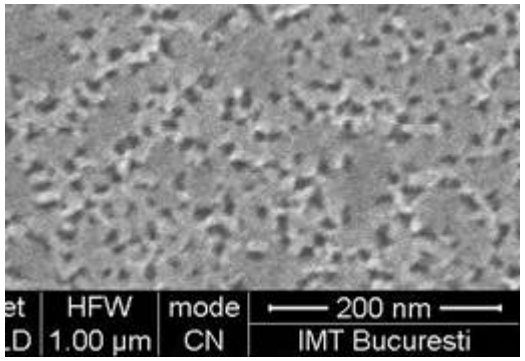
CARACTERIZARI



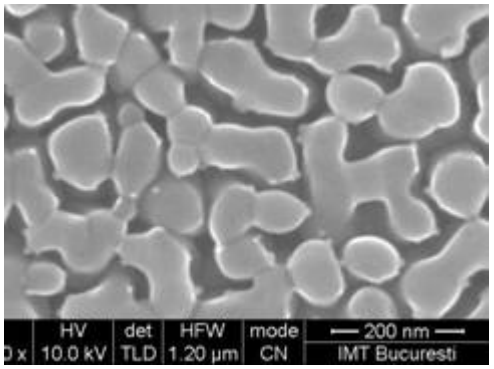
TiO_2 pe siliciu,

Solutie 10% NH4F in MEG,
V = 5V, pH = 6,4



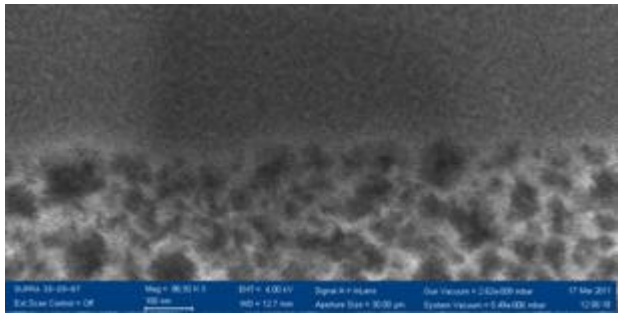


TiO_2 pe siliciu,
Solutie 10% NH4F in MEG, V = 5V, pH = 6,4 , tratat la 450 °C, (anatase)



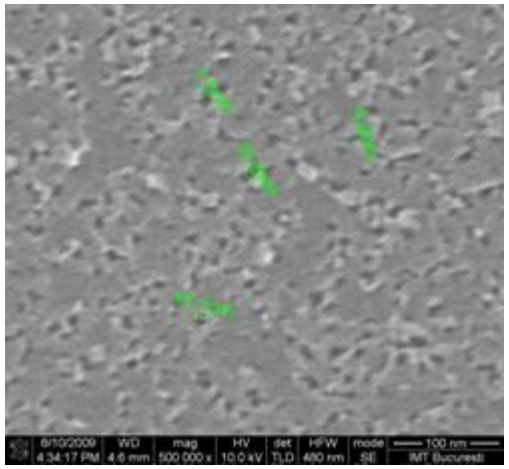
TiO_2 pe siliciu,
Solutie 10% NH4F in MEG, V = 5V, pH = 6,4 , tratat la 860 °C, (rutil)

TiO_2 pe sticla, tratat la 450 °C, (anatase)

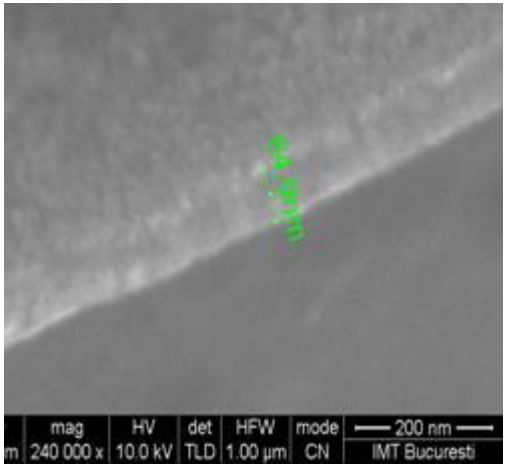


CARACTERIZARI

Dimensiunea porilor

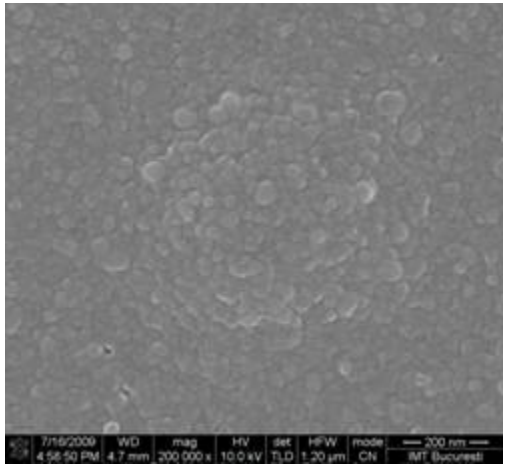


TiO_2 nedopat

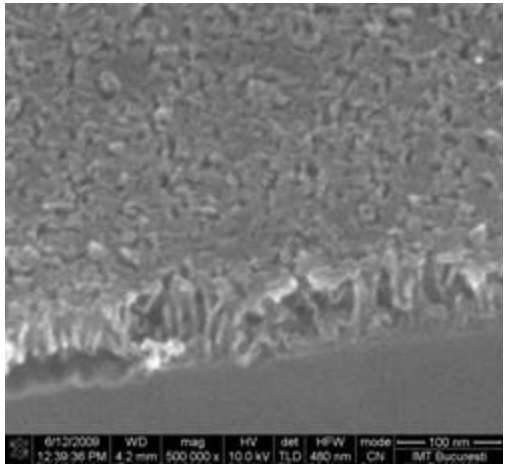


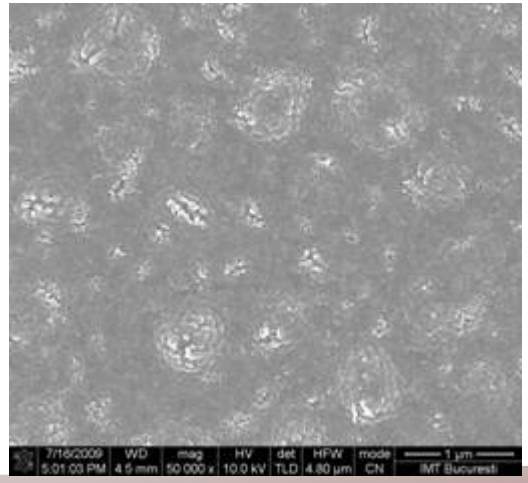
TiO_2 dopat cu fosfor din $POCl_3$ termic la 450 ^0C

Straturi de TiO_2 dopate



TiO_2 dopat cu fosfor din solutie de acid fosforic H_3PO_4 si acid oxalic$(COOH)_2$



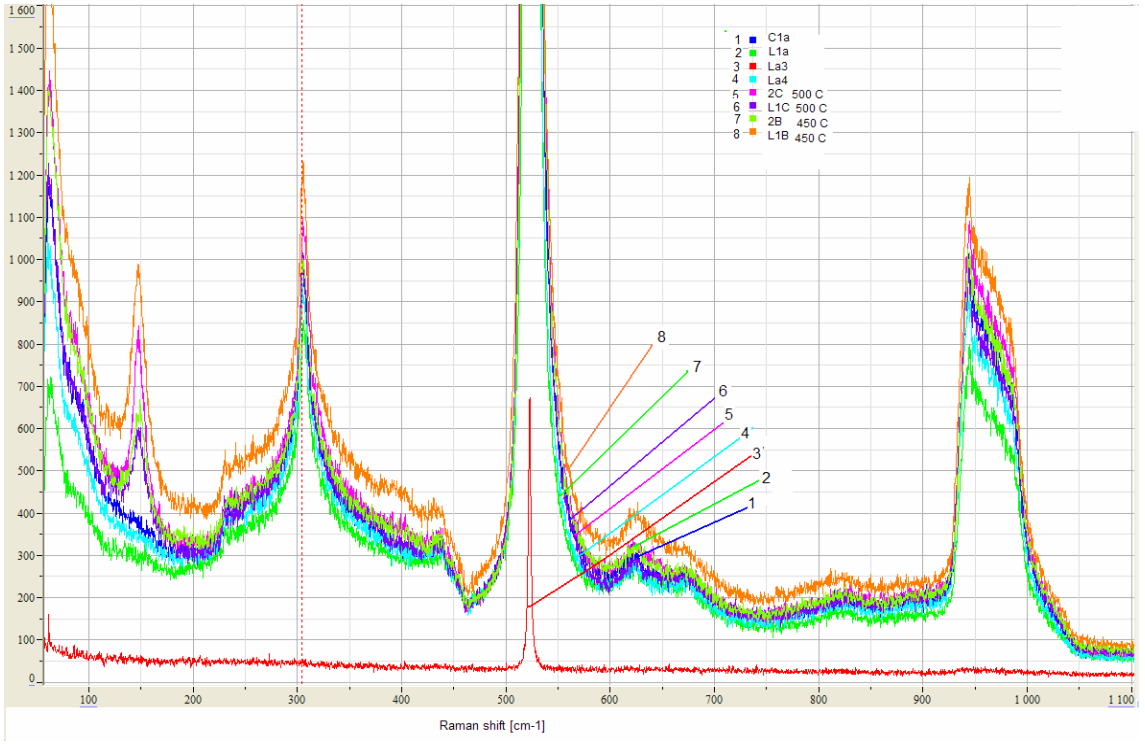


TiO_2 dopat cu Pd din solutie de $PdCl_2$

CARACTERIZARI STRUCTURALE

C A R A C T E R I Z A R I

Spectre Raman pentru probele de TiO_2 obtinute pe substrat de siliciu

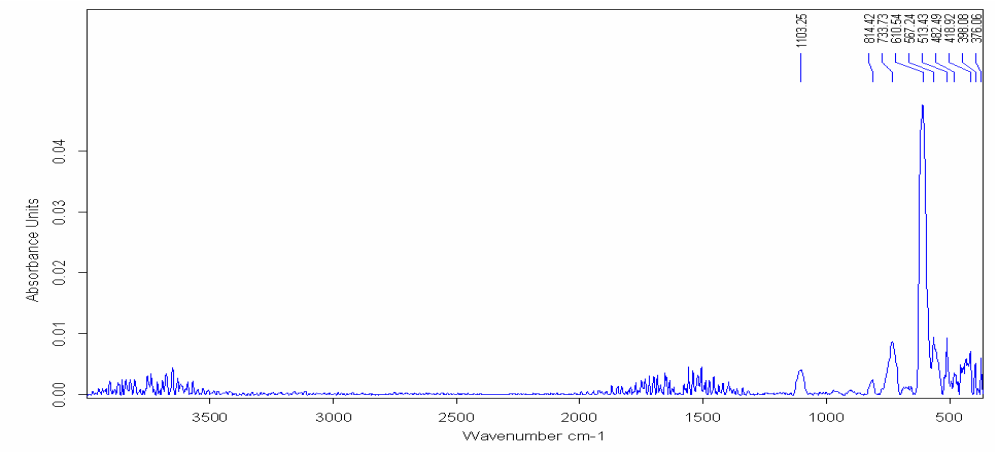


1-6 sunt anodizate toate la I=60 mA si
1- la 5V si 60 mA, 2 min. 26^0C ;
2- 5V, 5min., 26^0C ;
3 -8min, 7V si 3V, 26^0C ;
4- 5V, 6,5 min.,32^0C ; 5- 2C 8min, 7V, 26^0C tratat la 500^0C;

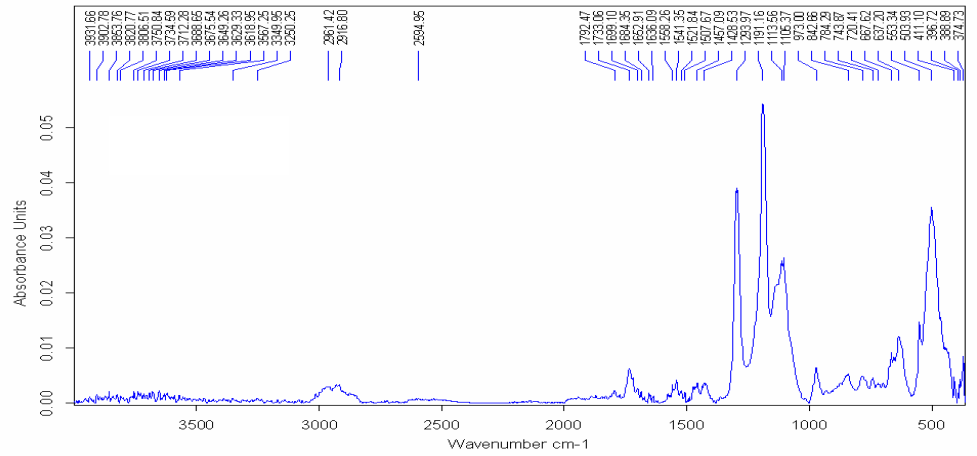
6- 5V, 5min., 260C , tratat la 500 °C;
7- 5V, 6min.,11 min, tratat la 450 °C;
8- 5V, 5min., 260C, tratat la 450 °C .

Conditiile optime pentru obtinere TiO_2-anatase prin anodizarea Ti, sunt: 5V, 60 mA, 450^0C, solutia acida 0,5%NH_4F[100%]; MEG; H_2O), ph=6,2.

Spectre FTIR relevante ale unor probe de oxid de titan obtinut prin anodizarea electrochimica a Ti



Spectrul probei de oxid de titan obtinut prin anodizarea intr-un electrolit format din 0,5%NH_4F[100%]: MEG:H_2O), ph=6,2 5V, 60 mA.



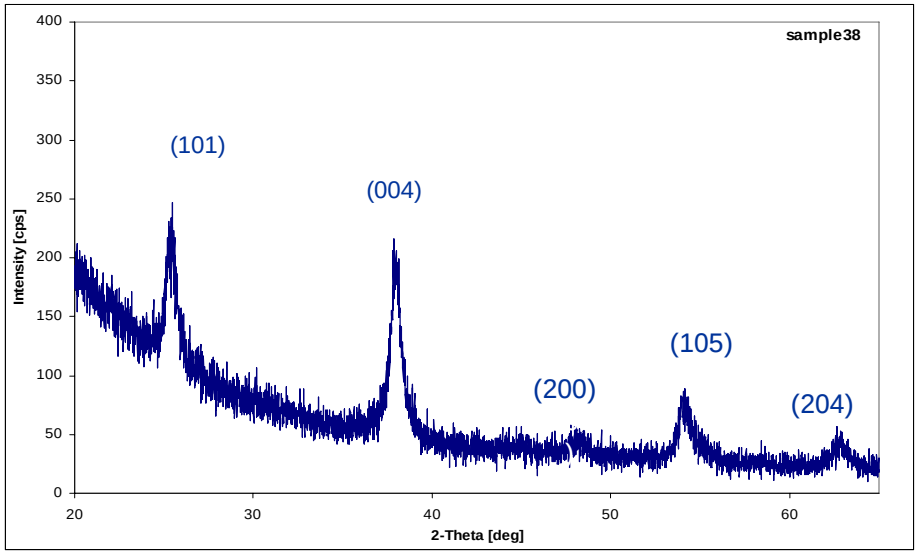
Spectrul probei de oxid de titan obtinut prin anodizarea intr-un electrolit format din din acid oxalic si acid orto fosforic

CARACTERIZARI

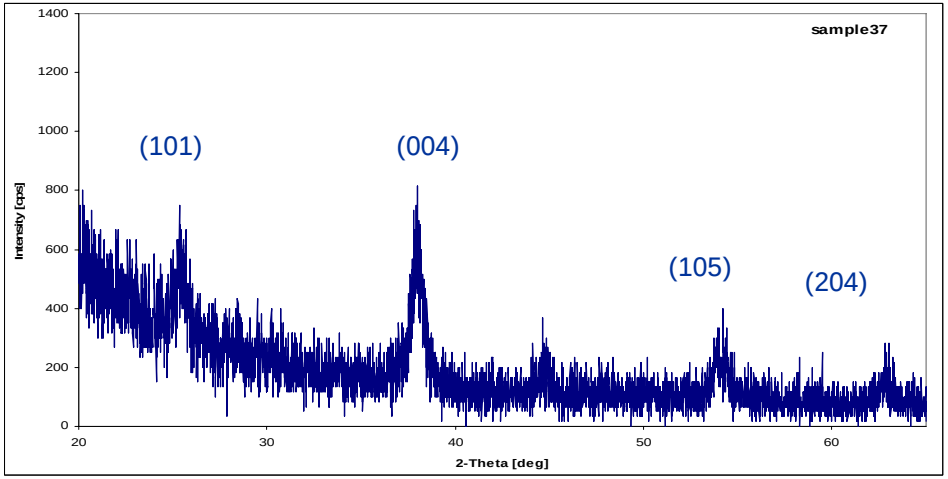
Spectre de raze X

Spectru X-ray al unei probe in care apar numai picurile caracteristice

pentru anatase

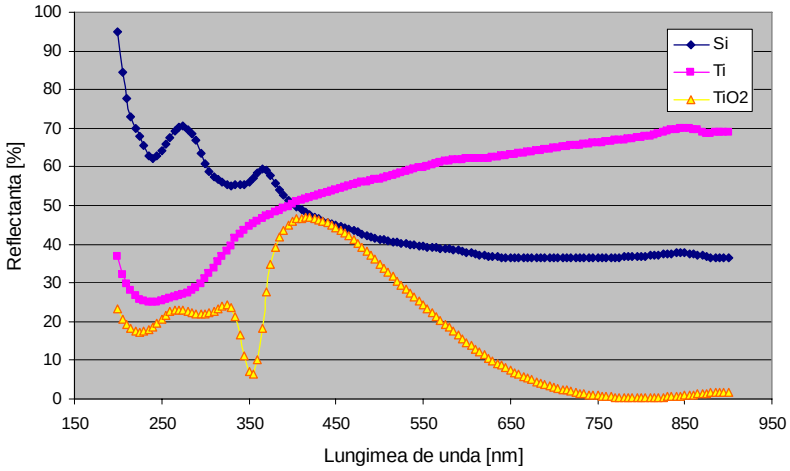


Spectru X-ray al unei probe in care apare si un pic caracteristic pentru ruthil, la 44^0

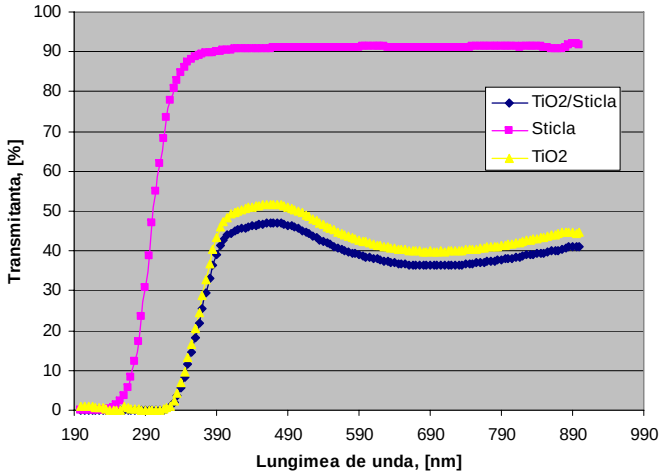


CARACTERIZARI OPTICE ALE FILMELOR DE TiO_2

CARACTERIZARI

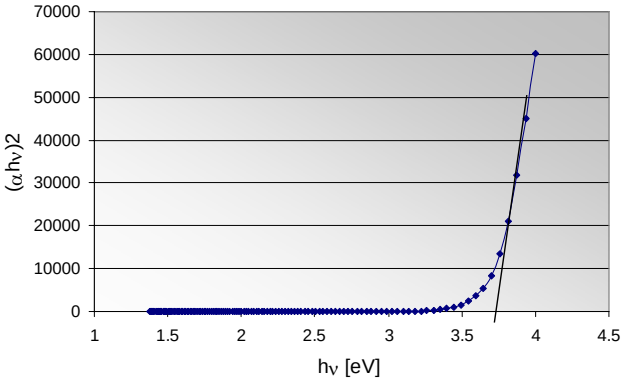


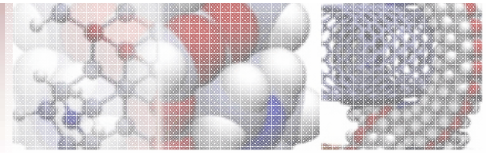
Dependenta spectrala a reflectantei pentru straturile de Ti, TiO_2 si pentru Si. Pentru TiO_2 reflectanta are valoare minima in domeniul λ>700nm.



Dependenta spectrala a transmitantei pentru straturile de TiO_2 si sticla si pentru TiO_2/sticla.

Determinarea benzii interzise pentru TiO_2





CONCLUZII

⇒ S-au obtinut filme subtiri de TiO_2 faza anatase prin anodizarea Ti metalic depus prin sputtering pe substrat de siliciu si de sticla.

⇒ Filmele obtinute sunt nano-poroase (3-10nm) in conditii de anodizare la tensiune constanta in domeniul 5-10 V.

⇒ S-a utilizat o solutie usor acida pe baza de fluorura de amoniu.

⇒ Experimental s-au determinat conditiile necesare pentru obtinerea porilor cu dimensiuni controlate.

⇒ Straturile nanoporoase de TiO_2 obtinute pot fi utilizate la realizarea de celule solare pe baza de pigmenti si la senzori bio si de gaze