

Metode Moderne de Analiza si Control Nedistructiv cu Radiatii X:

Conditii, Limite, Perspective

Aplicatii in studiul materialelor nanostructurate si

Metrologia Nanotehnologiilor Aplicate

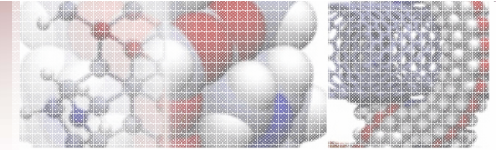
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Metode de Analiza Nedestructiva cu Radiatii X:



A. Microstructura fizica (faze cristaline) – XRD (WA, IP, GI) ->

Transmisie +/Reflexie, Topografie

A1. Materiale Policristaline

Filme subtiri, pulberi, suspensii, probe de volum

Nanomateriale (faze cristaline slab difractante, cantitati mici)

A2. Materiale Monocristaline

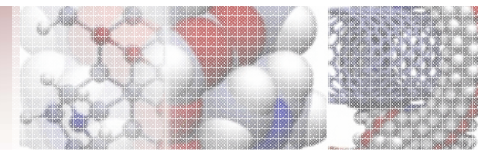
Plachete- probe de volum/grosime mare, filme epitaxiale, QD&(M)QW

Straturi poroase PS-Si

B. Determinarea grosimii, rugozitatii, densitate, largimea interfetei filmelor subtiri depuse pe substrat – XRR (Reflectivitate de raze X)

(Indiferent de tipul materialului probei/compozitia de faze – cristalin si/sau amorf)

Conditii / Limitari / Perspective impuse/determinate de:



1. Caracteristicile fascicolului de raze X incident pe proba:

- lungimea de unda/energia fotonului X $1\text{\AA}/1\text{-}15\text{ keV}$,
- fluenta si intensitatea fascicolului pe proba $1\text{-}10^6\text{ -}10^9\text{ cps}$,
- durata pulsului,
- tipul fascicolului: paralel sau divergent,
- divergenta fascicolului.

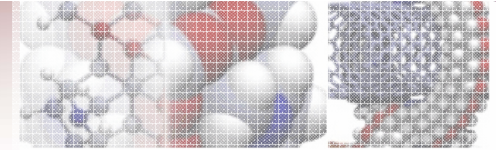
2. Caracteristicile goniometrului & sistemului de detectie:

- numarul de axe independente de rotatie ale probei – uniax (pulberi & policristale),
- uniax, axa dubla sau tripla.

3. Caracteristicile probei

- Faza slab difractantata – volum/cantitate mica, amestec faza cristalina/amorfa
- Nanomateriale: dimensiunea medie de cristalit $1\text{nm} < D < 5\text{nm}$;
- Limita pentru faza cristalina este de 1nm!

Aplicatii:



1. Difracție pe pulberi (policristale) > X-Ray Powder Diffraction

- Compoziție de faze
- % cristalinitate (analiza cantitativă&calitativă)
- Dimensiune de cristalit
- Incidență razantă GIXRD- grazing incident small angle X-ray diffraction
- Rafinarea parametrului de retea cristalina
- Deformari si tensiuni reziduale
- Microdifracție & Mapare parametri fizici pe proba
- Textură

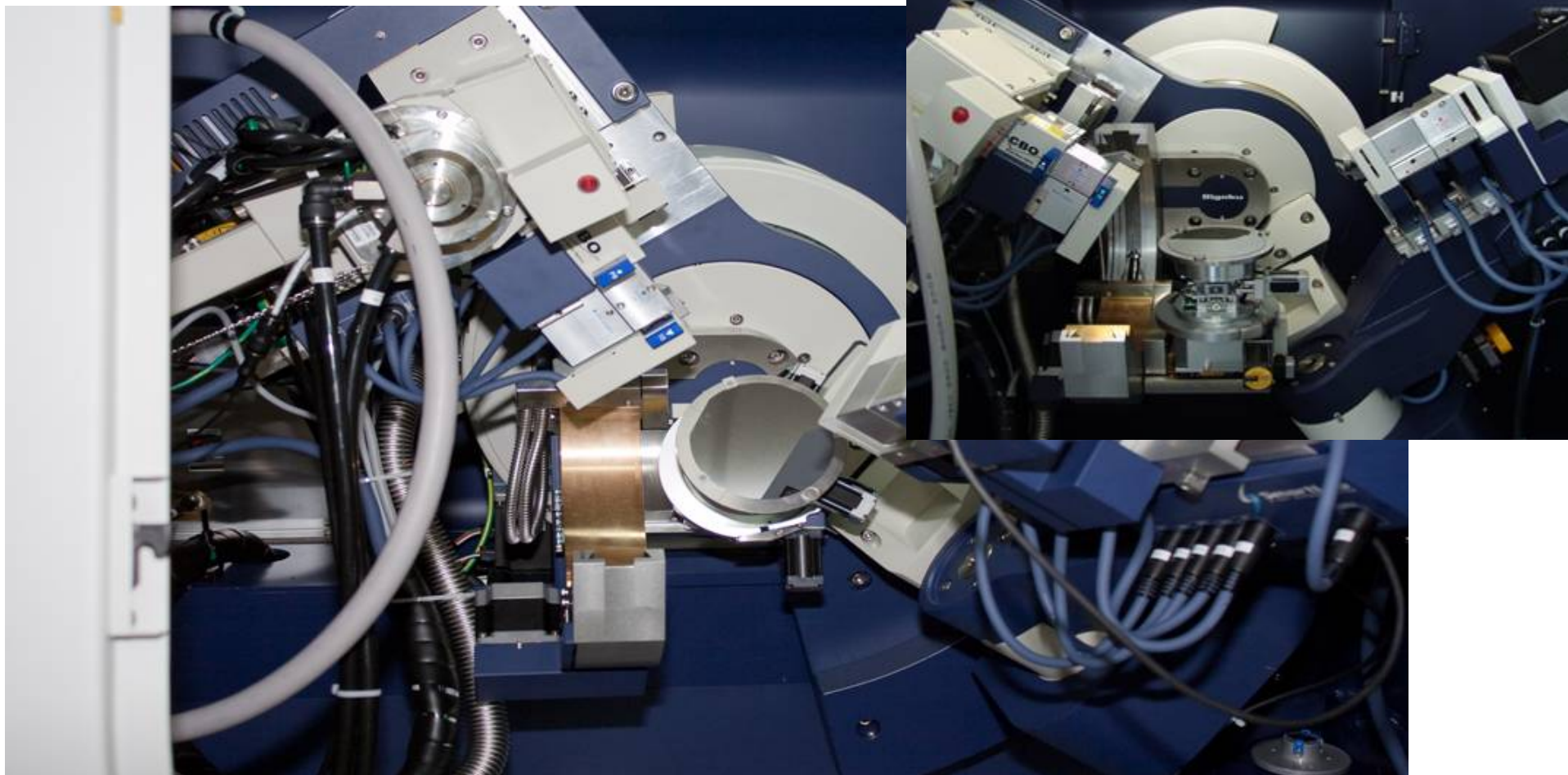
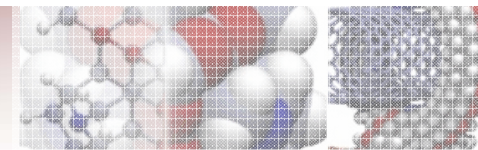
2. Analiza filmelor subțiri

- GIXRD glancing incident angle diffraction
- IPXRD in-plane XRD
- Măsurători de reflectivitate de raze X – X - ray reflectivity XRR
- Filme epitaxiale (grosimi, tensiuni, compoziție, deformari)
- Rocking curves – analiza perfecțiunii de monocristal

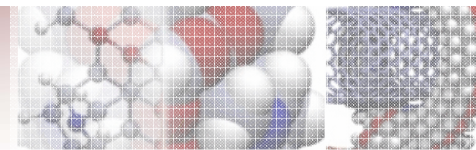
3. Analiza semiconductorilor de volum (monocristal > placheta)

- UHRXRD Difractie de Ultra Inalta rezolutie- determinarea absoluta a parametrului de retea
- RSM Reciprocal Space Map
- Determinarea dopajului
- Rocking curves – analiza perfecțiunii de monocristal

Difractometrul de inalta rezolutie cu anod rotitor 9 kW cu axa tripla Rigaku SmartLab instalat in IMT



Conditii



Metodele moderne de metrologie si control nedistructiv cu radiatii X presupun:

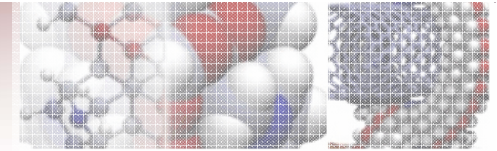
1. Proceduri automate de aliniere (modulelor optice, fascicol, proba) si achizitie date
2. Viteza mare de achizitie a datelor
3. Intensitate maxima a fascicolului pe proba
4. Repetabilitatea si fiabilitatea mare a datelor experimentale (fara artefacte)
5. Proceduri software adecvate de Analiza si Prelucrare a Datelor Experimentale (automate sau nu)
6. Stabilirea adecvata a metodei si configuratiei de analiza experimentală
7. Utilizarea uzuala de standarde, referinte si (re)calibrari uzuale

- Limite:**
1. Probe slab difractante, predominant amorfe, volum mic de faza cristalina (cazul nanomaterialelor)
 2. Metode statistice – valorile masurate sunt mediate pe multe celule elementare/cristalite/arie mare
 3. Dimensiuni/ modificari/ influente la nivel de celula elementara $Dd/d = 10^{-3} - 10^{-7}$

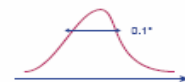
Rugozitati maxime de 4 -5 nm (XRR), grosimi maxime de 1000-2000 nm

Perspectivile sunt determinate de limitele actuale

1. Durata pulsului RX -> micșorare
2. Intensitatea/Briliana fascicolului (pe proba) -> crescuta cu ordine de marime



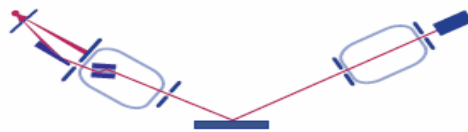
Optics



Bragg-Brentano focusing
Phase id, crystal structure, etc.
for powders



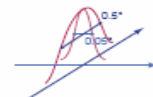
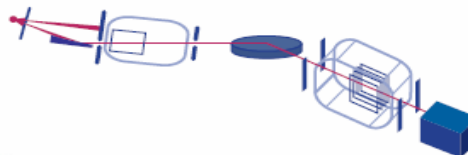
Parallel beam
Phase id, crystal structure, etc.
for thin films



Ge 2-bounce
Crystal structure, film thickness, etc.
for imperfect crystals

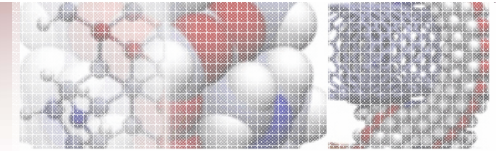


High resolution / triple-axis
Crystal structure, film thickness, etc.
for highly perfect crystals

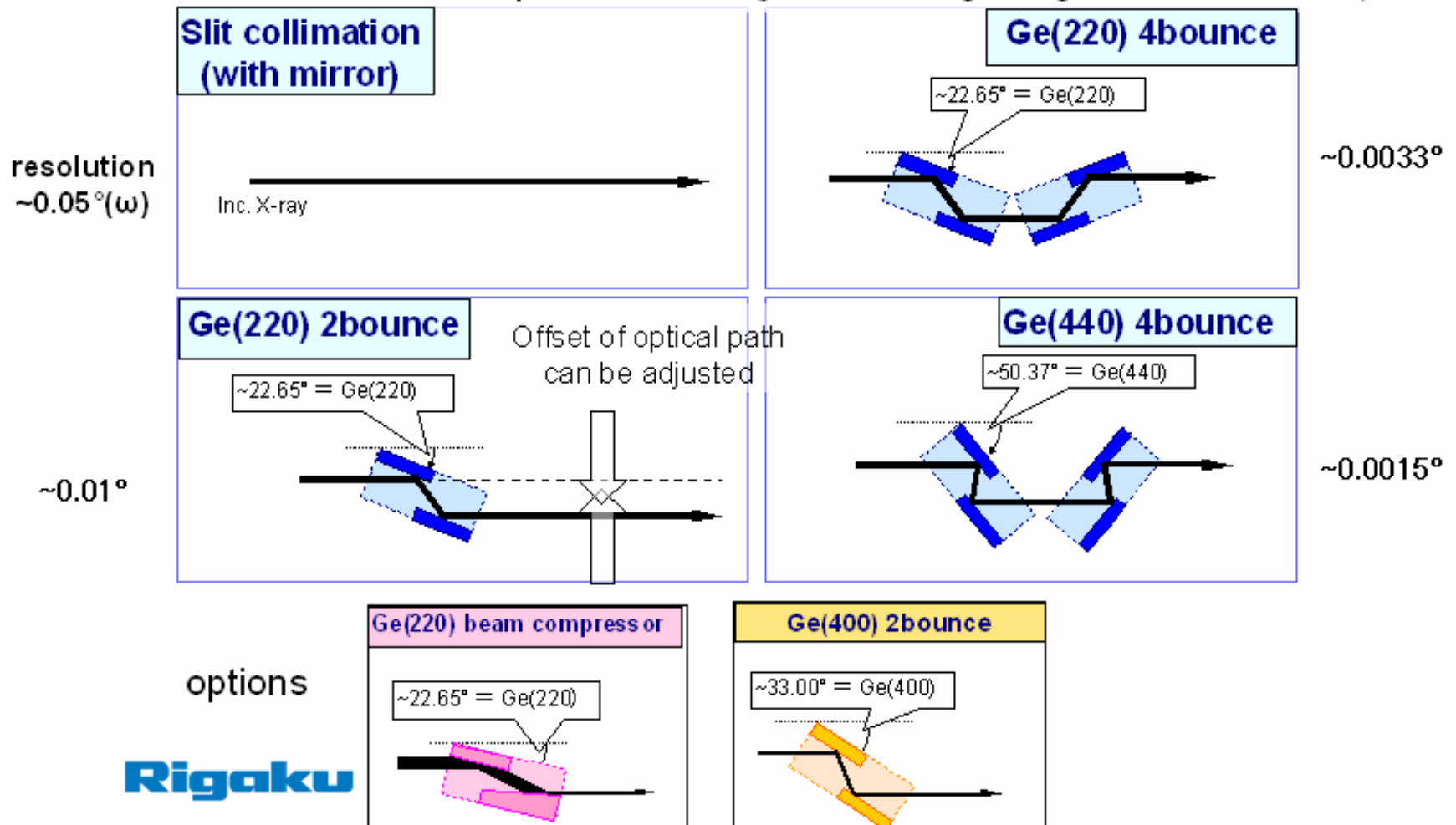


In-plane geometry
Depth controlled phase id, etc.
for ultra thin films

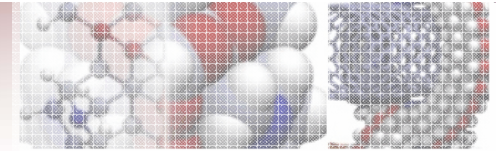




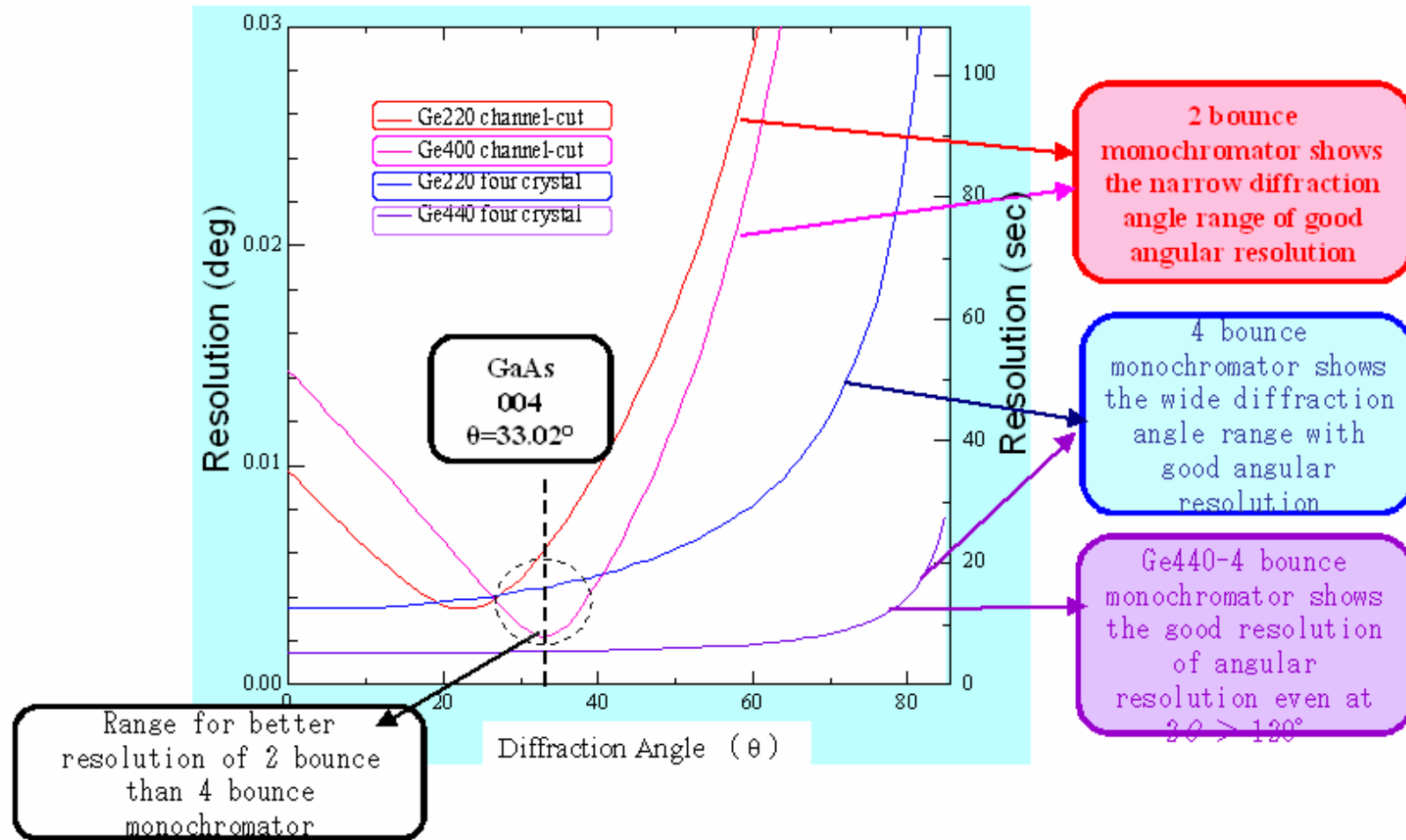
- Tunable for the best performance (just following the guidance software)



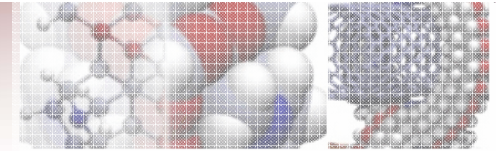
Rezolutia unghiulara tipica oferita de monocromatoarele de Ge cu reflexii multiple



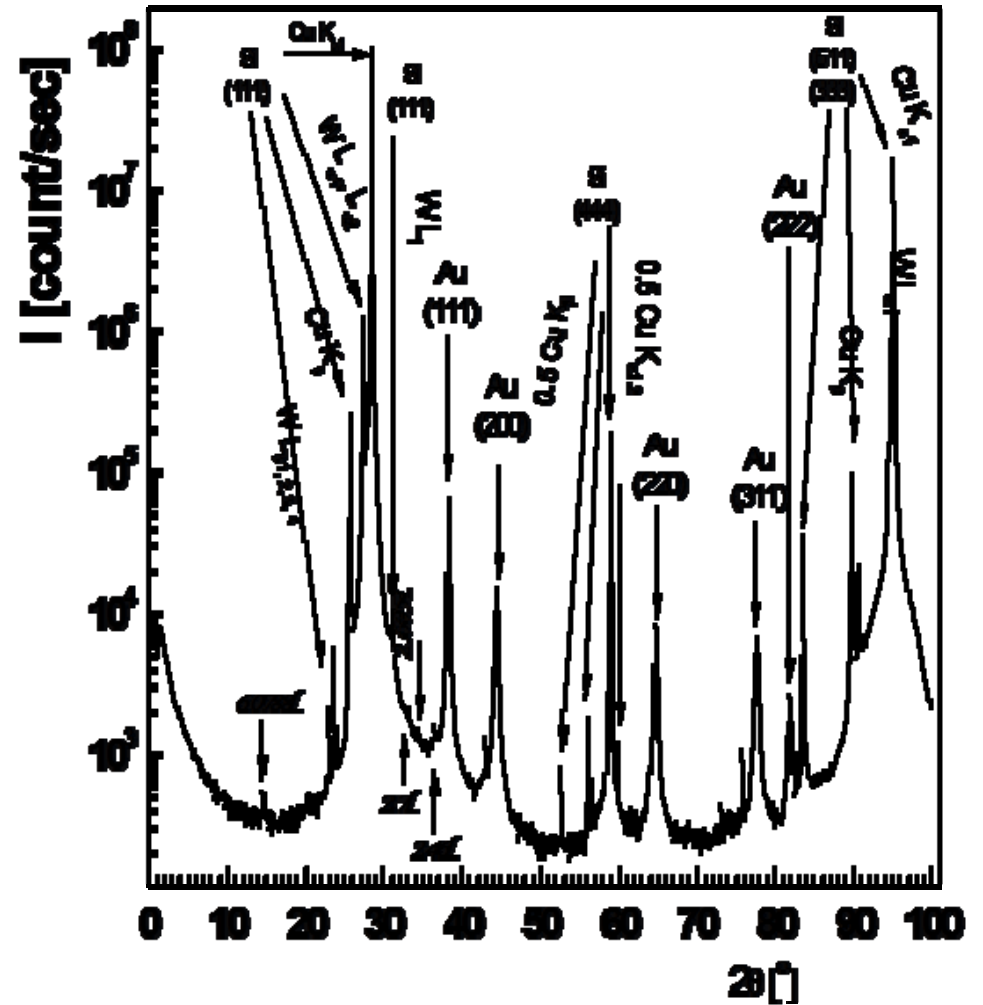
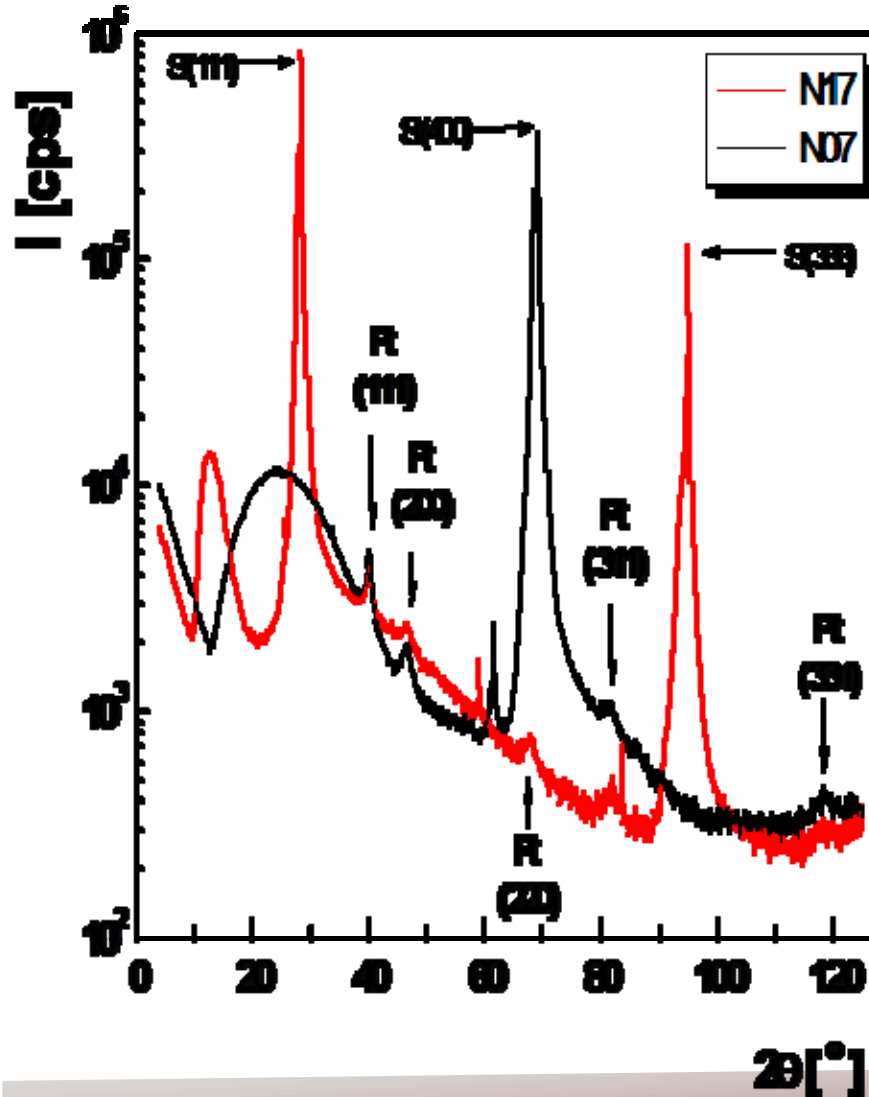
Appendix : Angular Resolution of Optical System

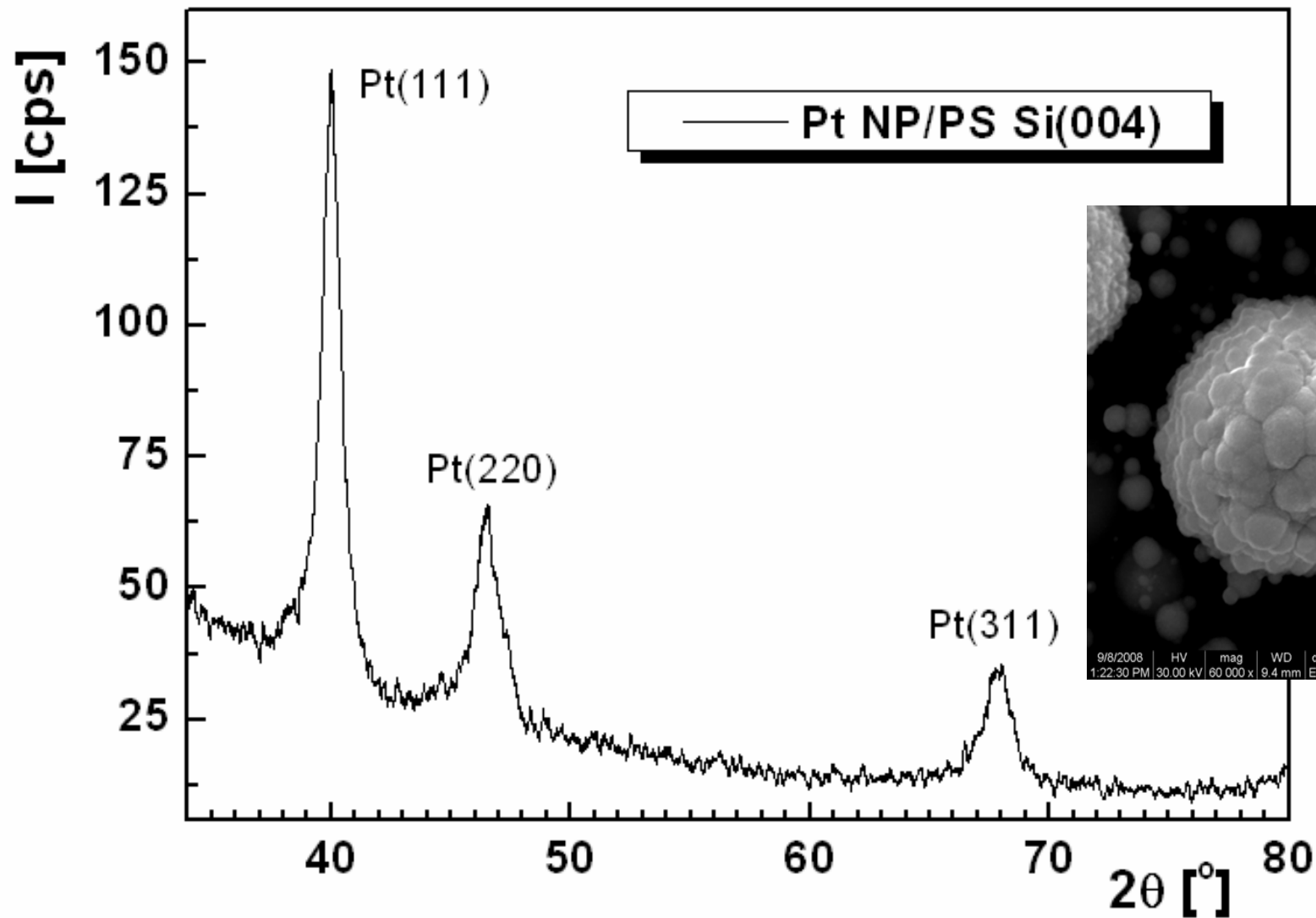
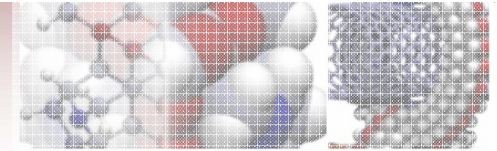


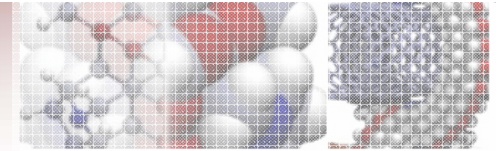
Exemple



1. Metoda A1, A2 > Microstructura filme Pt (Au) in PS - Si (400)







2. Metoda A1 > Microstructura PVA cu factor de biodegradare marit

Transmisie, Fascicol paralel nemonocromatizat

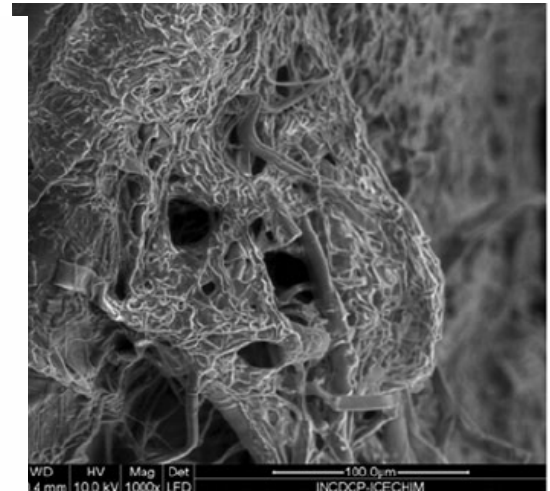
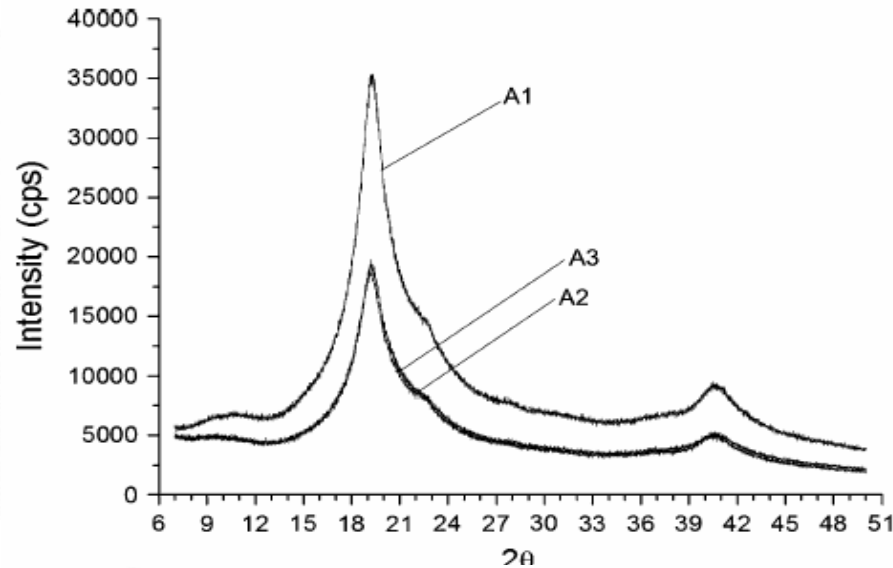
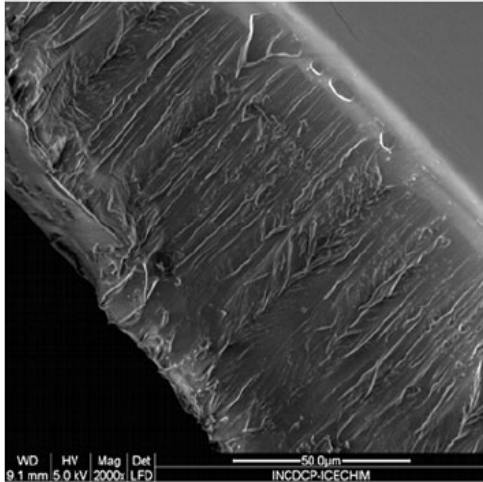


Fig 1 a, b. SEM, PVA Inainte

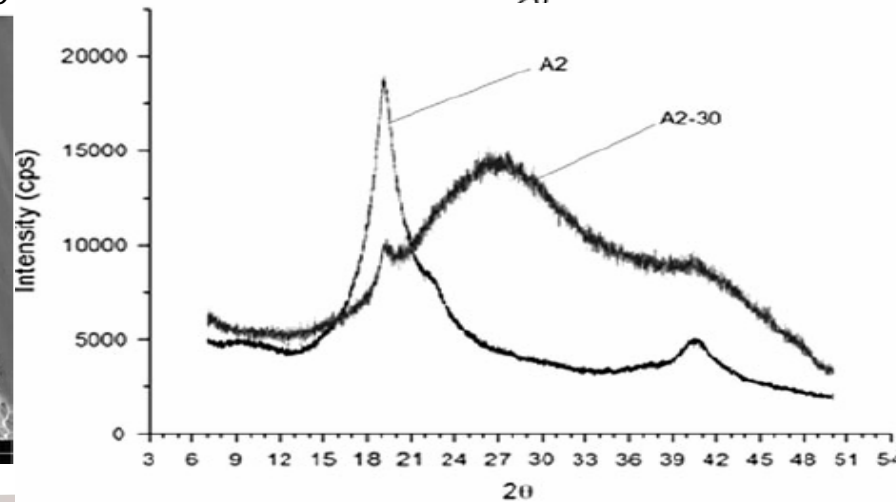
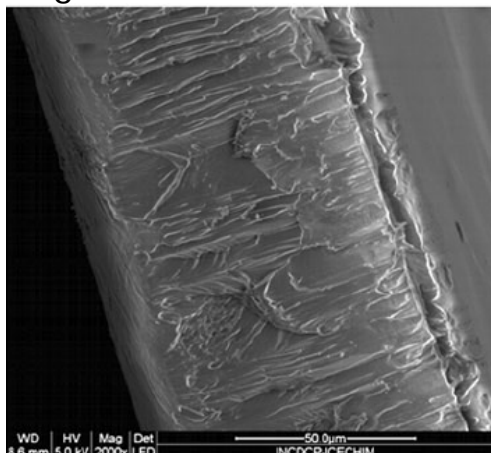
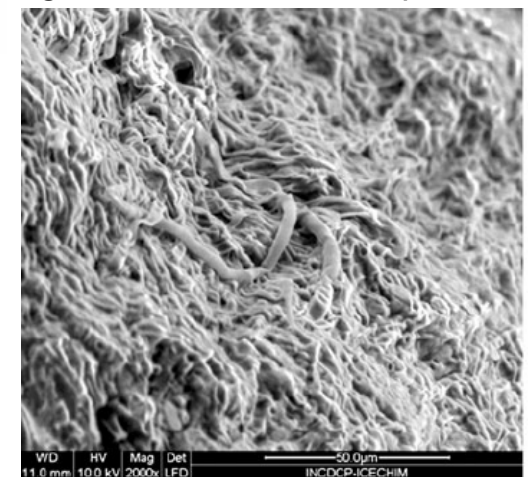
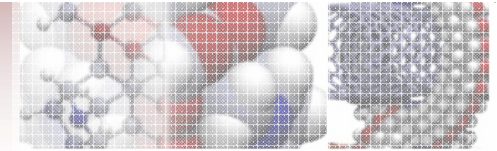


Fig 2 a, b. SEM, PVA dupa atac





3. Metoda A1,2 > Microstructura Monocristal / Filme Policristaline texturate

Reflexie, Fascicol paralel ne/monocromatizat

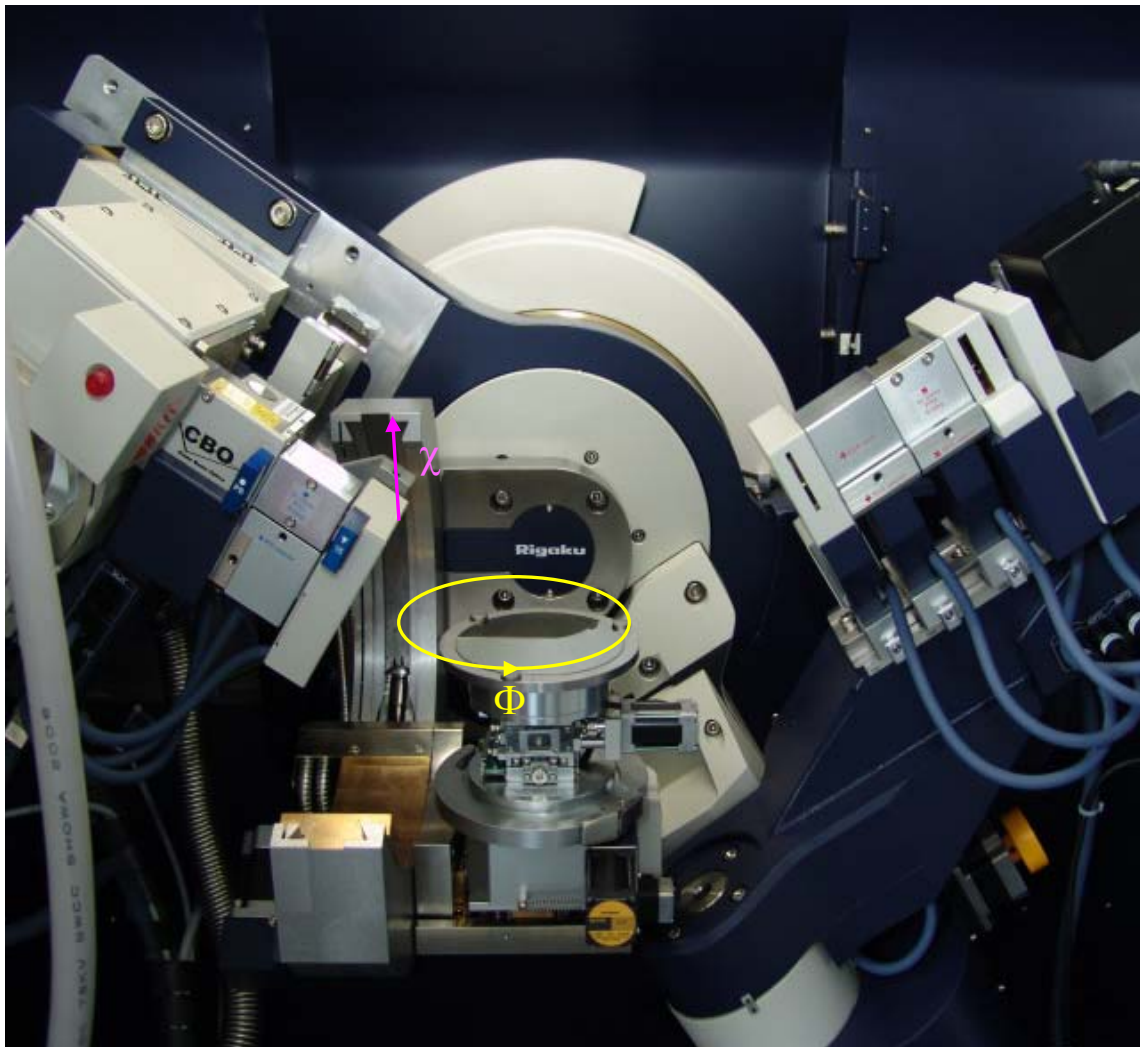
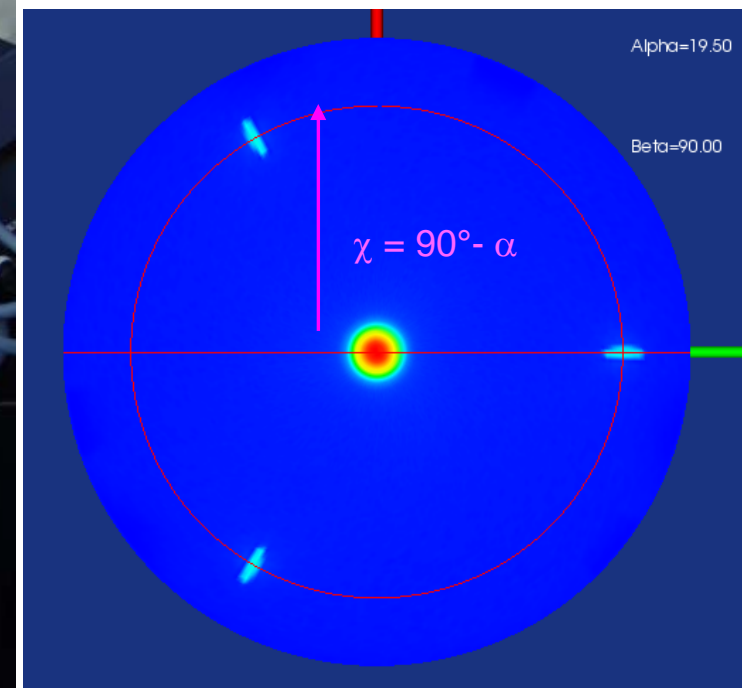


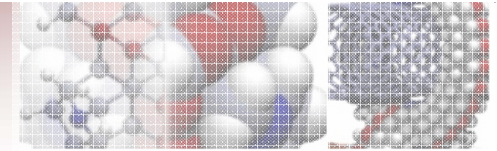
Figura de poli Si (111)

Φ – sample rotation

χ – sample inclination

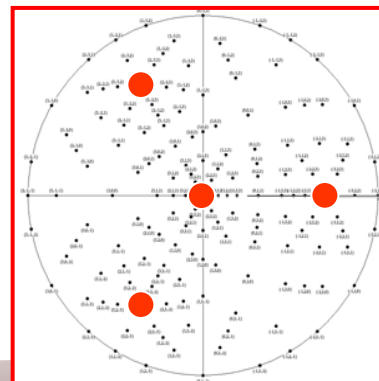
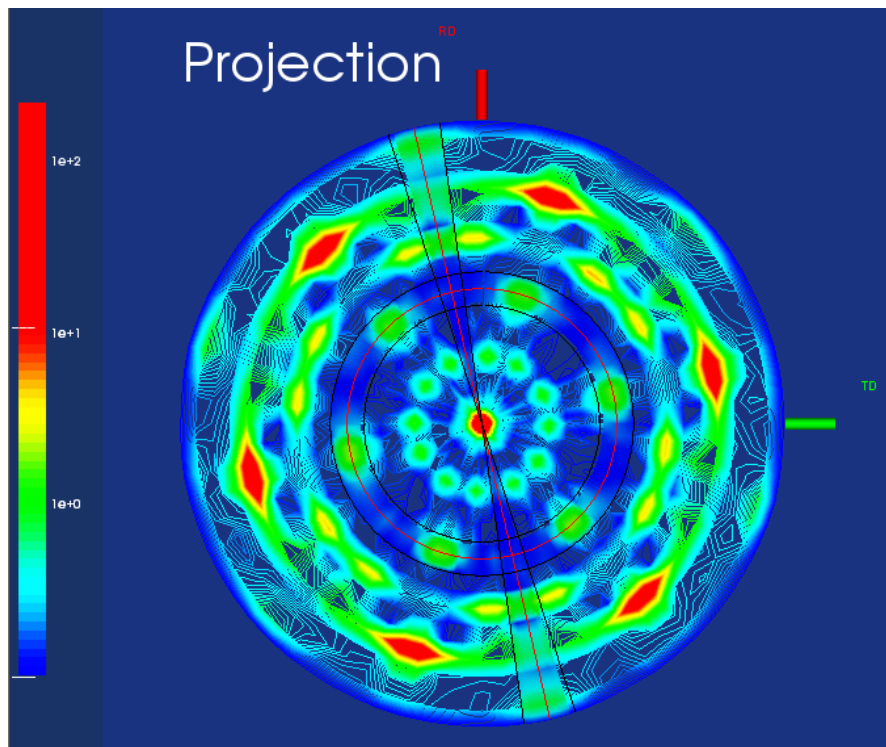
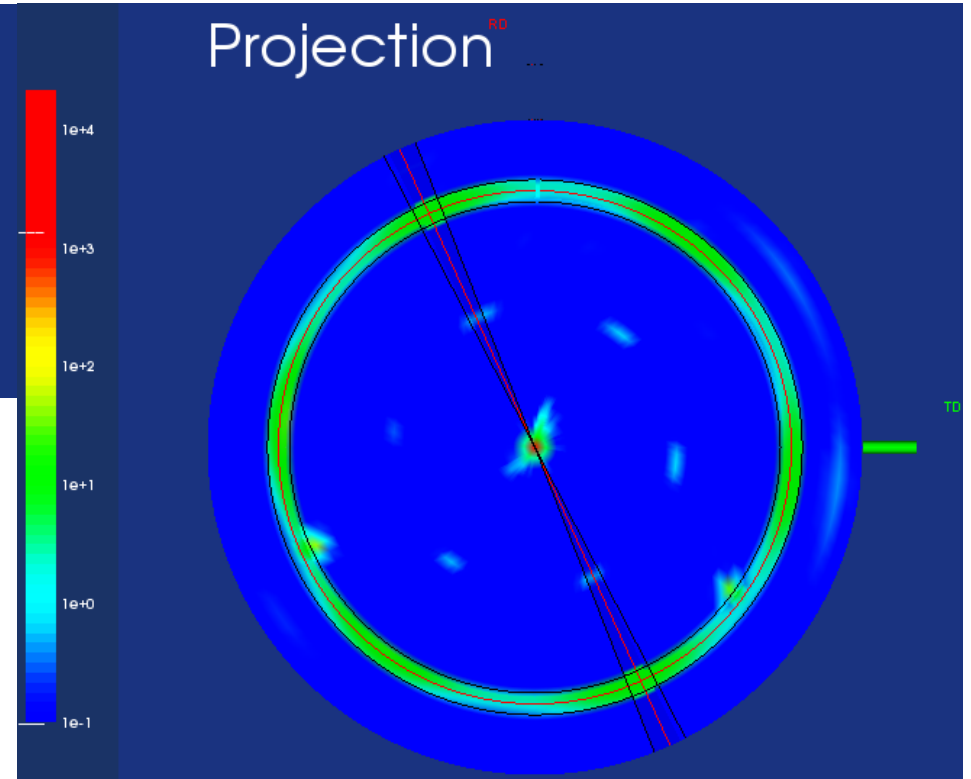
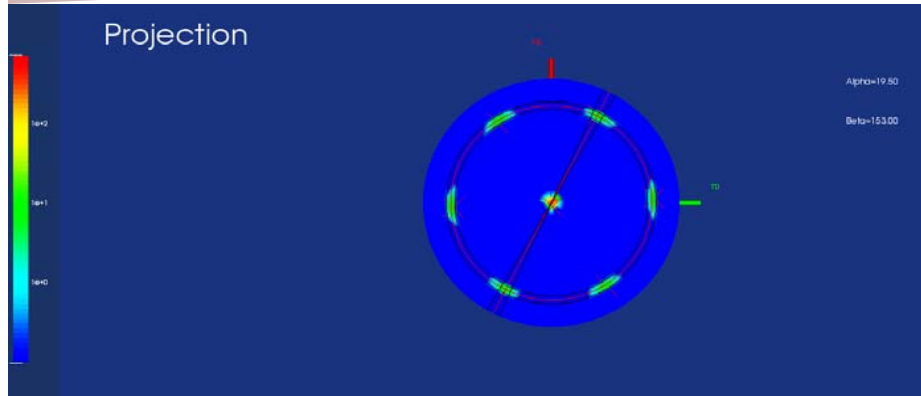


3.a. Metoda A1,2 >Textura, figuri de poli

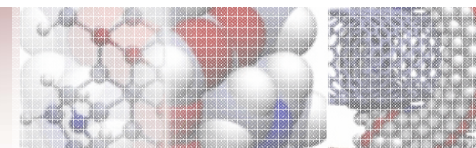


a. Monocristal Au(111) cu defecte (twinning)

b. Monocristal Au(111) cu defecte (twinning+dislocatii la 60°) si texturarea suprafetei



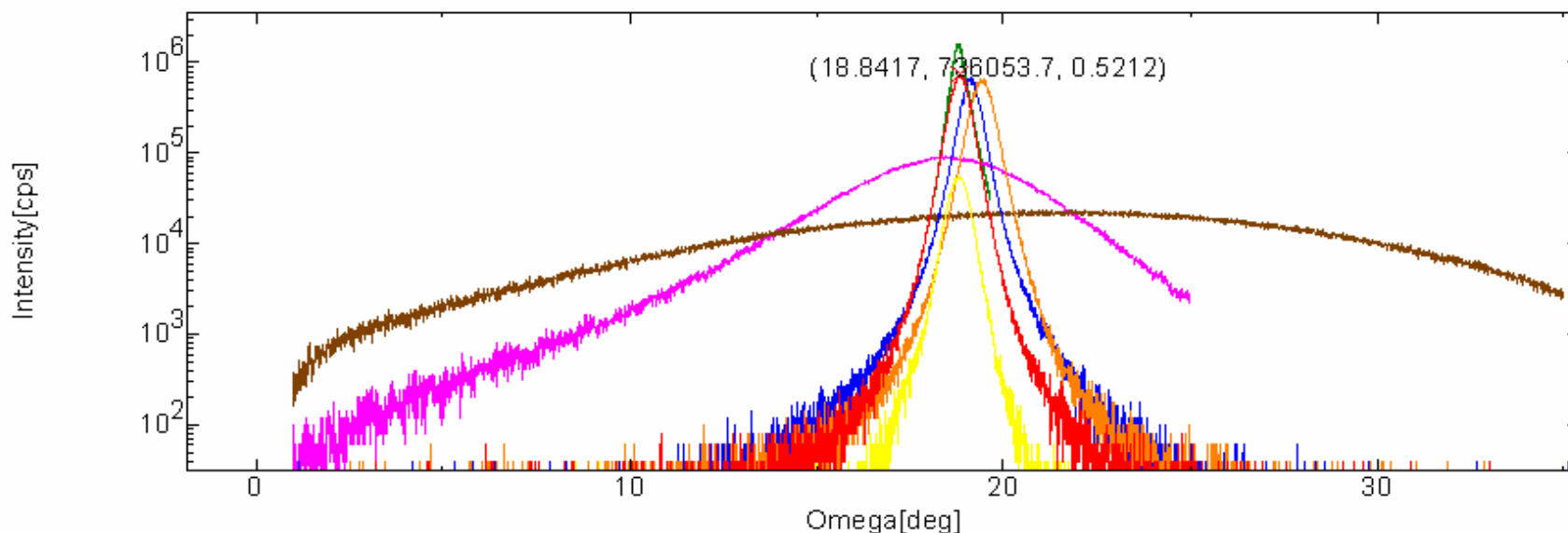
c. Textura Au(111) + blocuri monocristaline



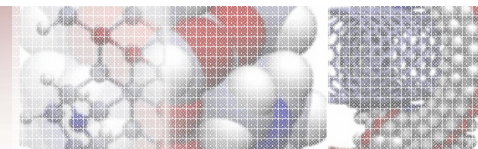
3.d. Metoda A1,2 > Textura, rocking curve

Comparatie Au11_cu IMT_2x mica.ras / Collected Data-4

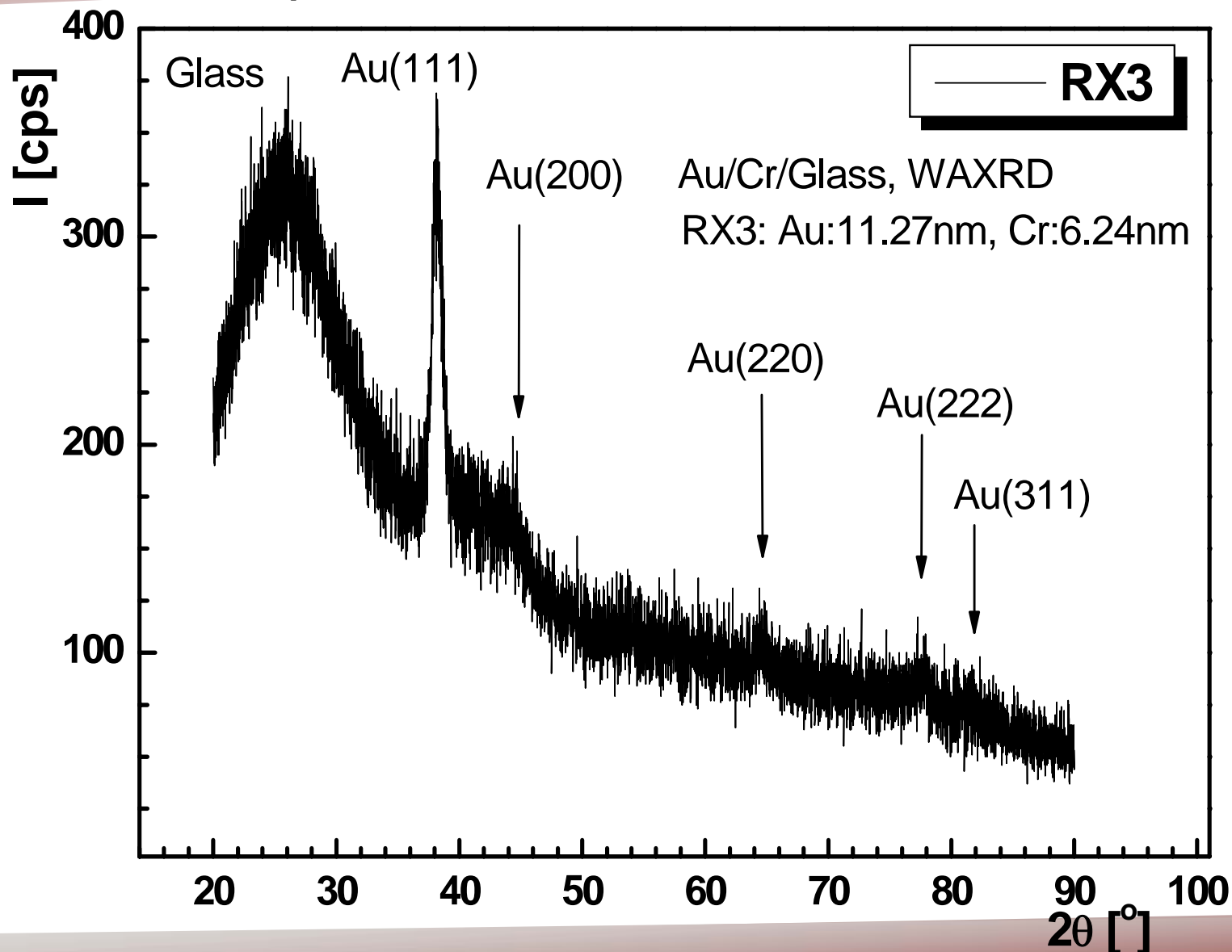
Sample Name	: None	Scan Mode	: CONTINUOUS	CBO selection slit	: PB
Comment	: Height alignment	Scan Speed	: 12.0000 deg/min	Incident parallel slit	: Soller_slit_5.0deg
Date	: 11/19/09 12:19:06	Scan Step	: 0.0100 deg	Incident slit	: 1.000mm
Operator	: Admin	Scan Axis	: Omega	Length limiting slit	: 0.5mm
X-Ray	: 40 kV , 80 mA	Scan Range	: 1.0001 - 35.0001 deg	Receiving slit # 1	: 1.000mm
Wavelength	: CuK α / 1.541867 Å	2-Theta	: 38.2276 deg	Filter	: None
Goniometer	: SmartLab(In-plane)	Omega	: 0.9951 deg	Receiving optical device	: PSA_open
Base Att. Config	: Std. chi cradle	Chi	: 0.000 deg	Receiving parallel slit	: Soller_slit_5.0deg
Attachment	: RxRy	Phi	: 0.000 deg	Receiving slit # 2	: 1.000mm
Detector	: SC-70	2-ThetaChi	: 0.0000 deg	Attenuator	: Open
Memo	: None			Diffracted beam mono.	: Flat
Optics Attribute	: Medium resolution parallel beam/RS			Monochromator slit	: None

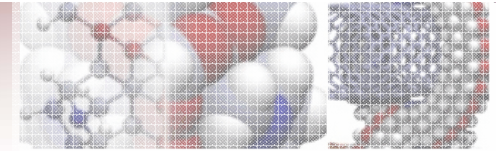


4. Metoda A1 > Microstructura filme policristaline subtiri

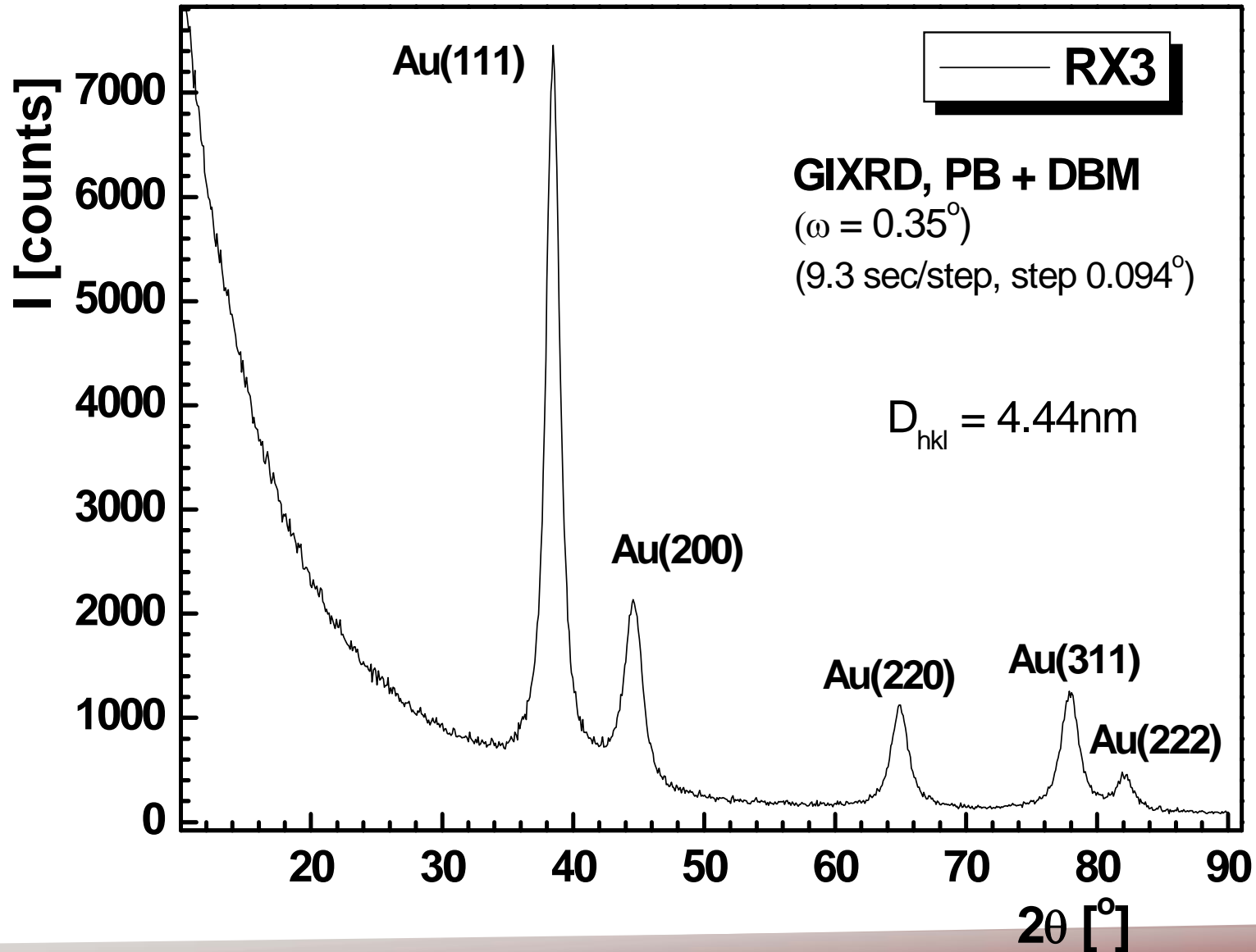


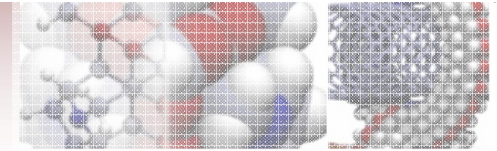
A. Reflexie, Fascicul paralel ne/monocromatizat, WAXRD





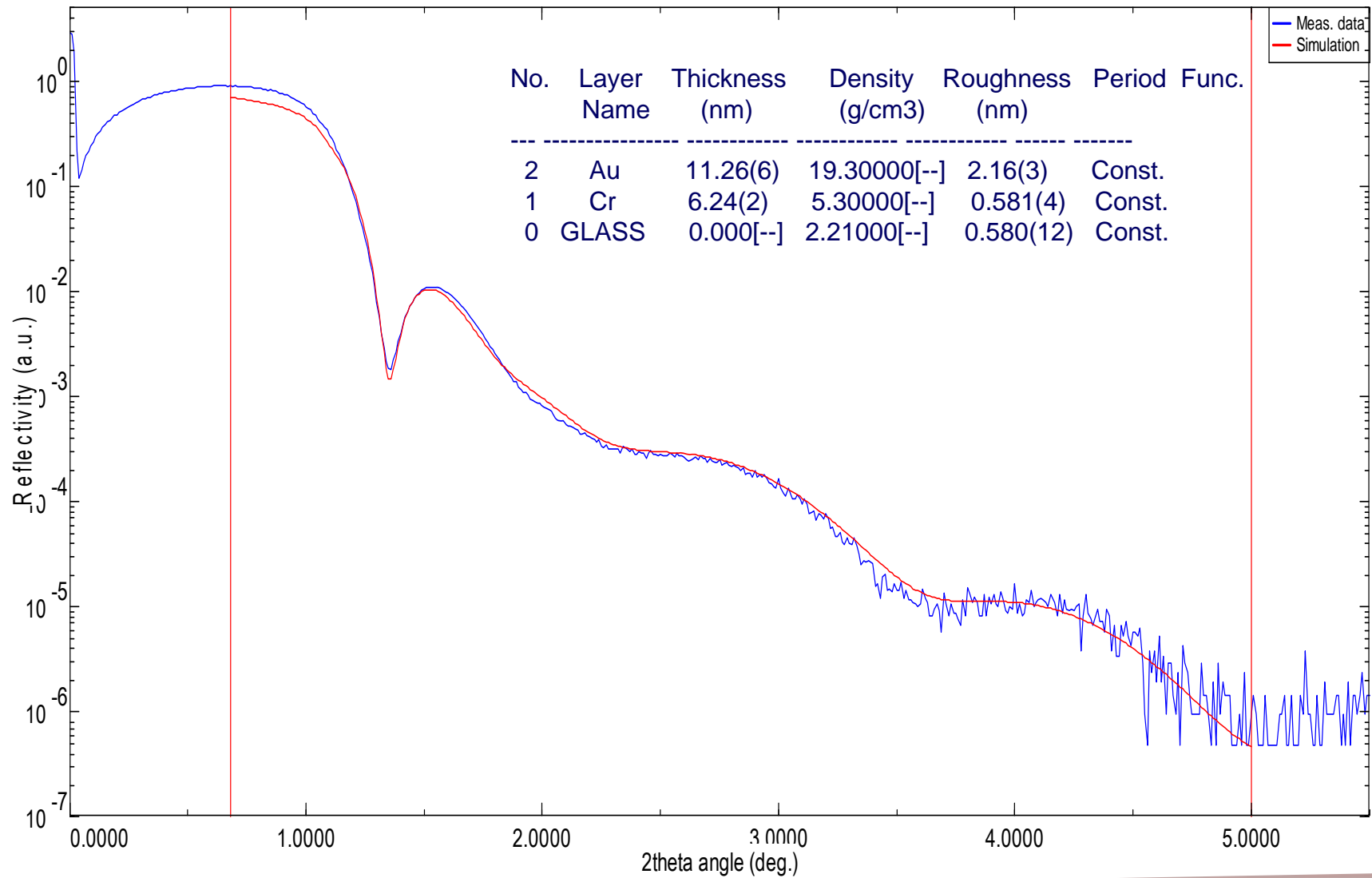
B. Metoda A1, GIXRD



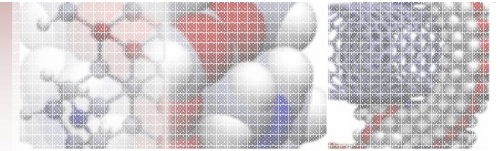


Metoda B. XRR Determinarea grosimii filmelor pe substrat ⁽³⁾

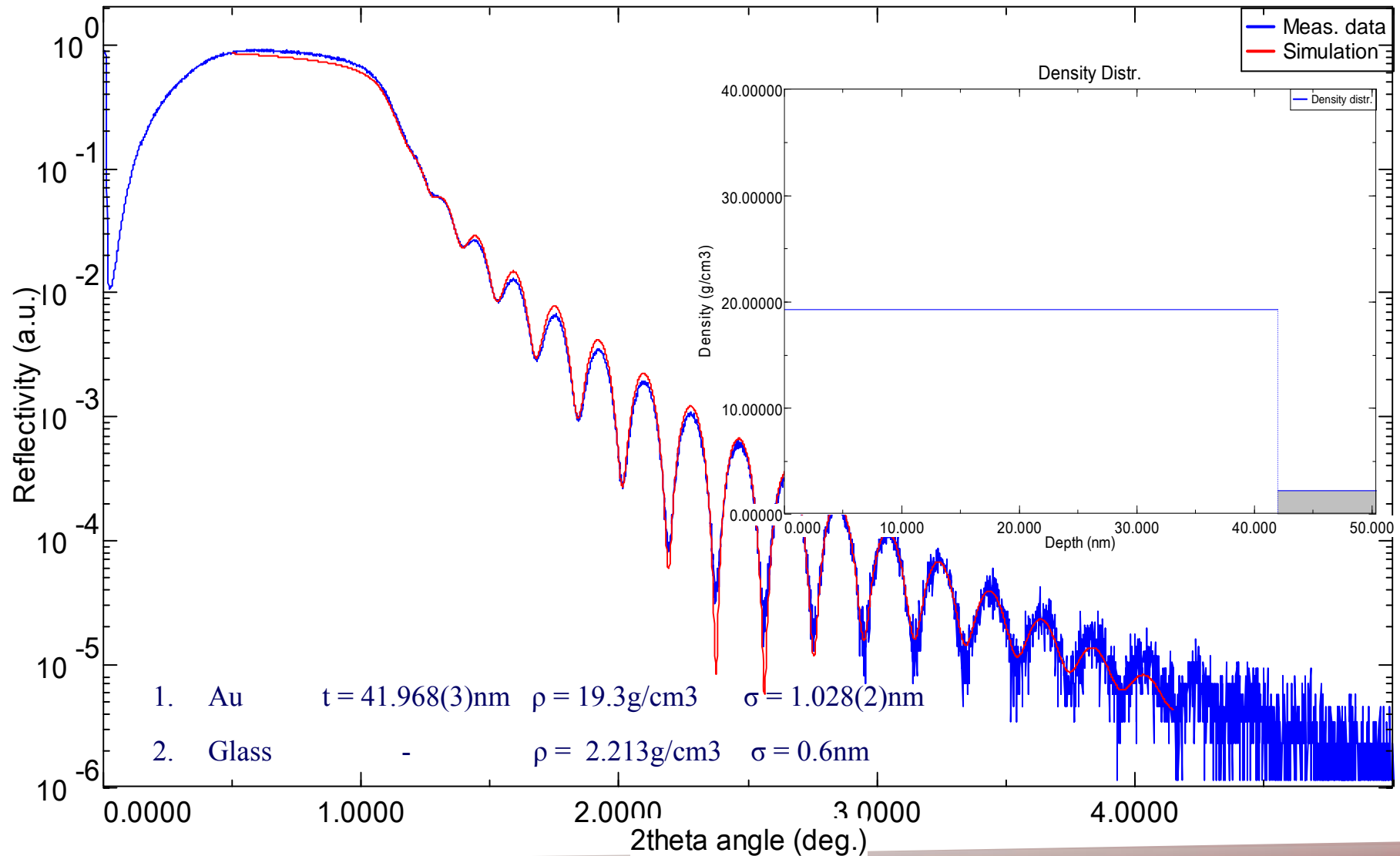
REFLECTIVITY PROFILE



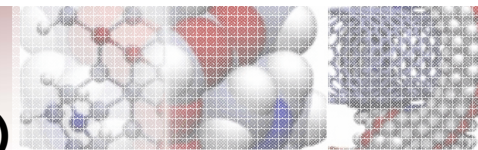
5. Metoda B, XRR, film Au fara strat de aderenta de Cr



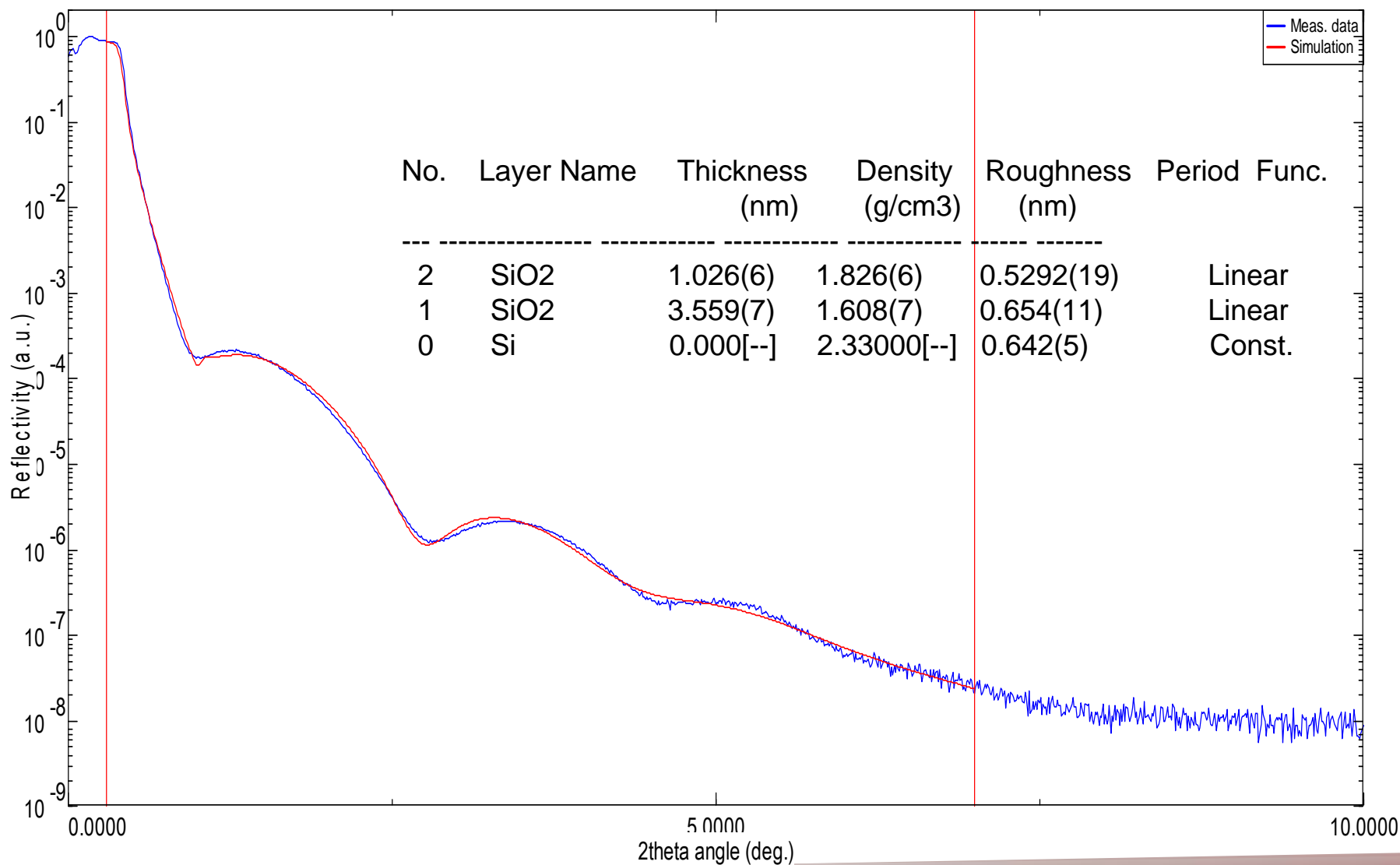
REFLECTIVITY PROFILE



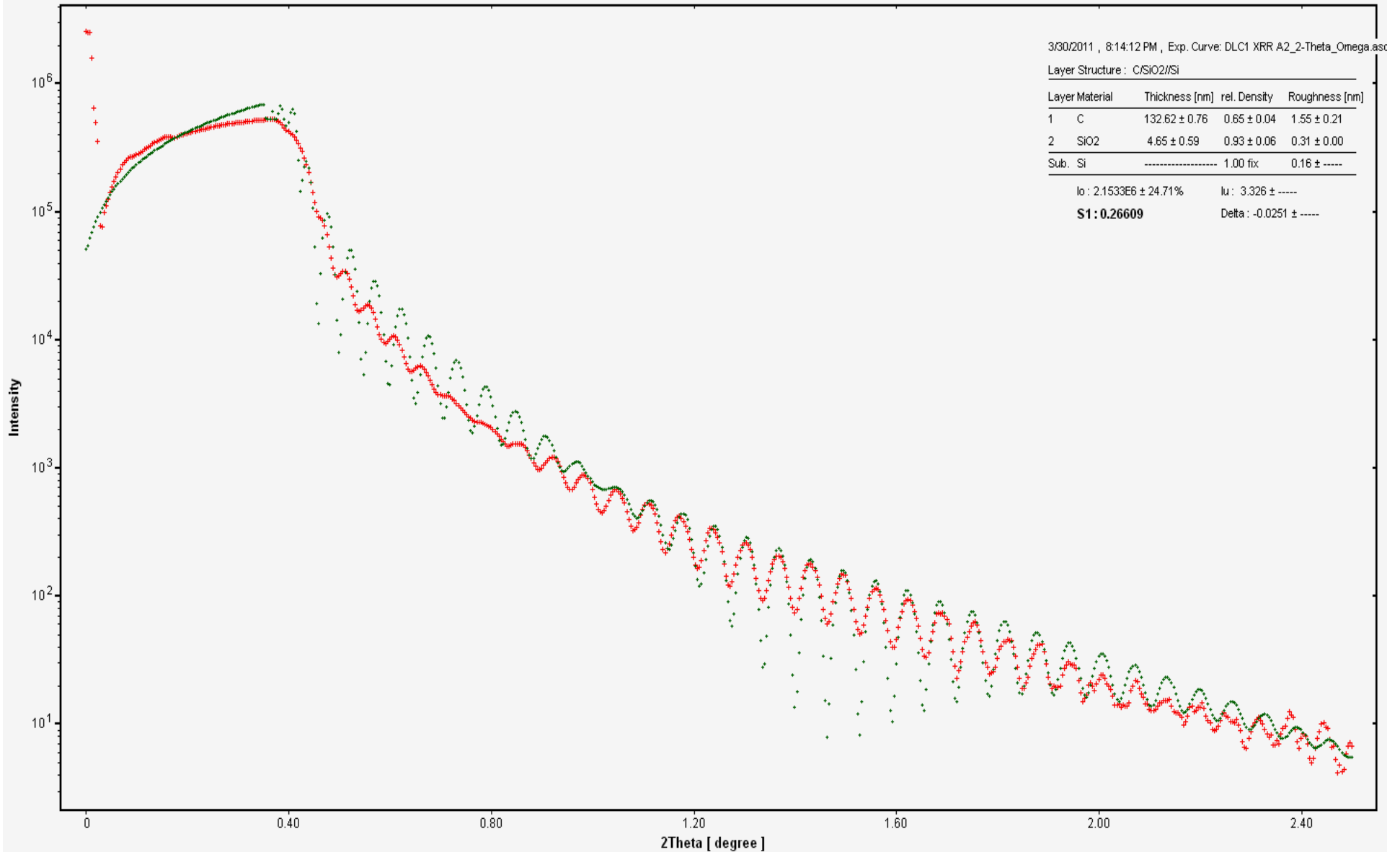
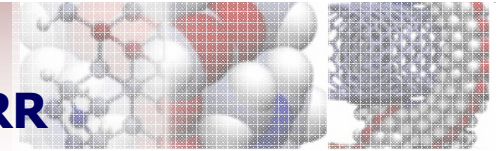
6. XRR, Oxid SiO₂ nativ amorf (poros, densitate < val. Teoretica)



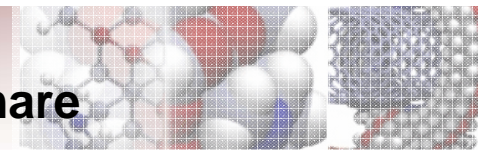
REFLECTIVITY PROFILE



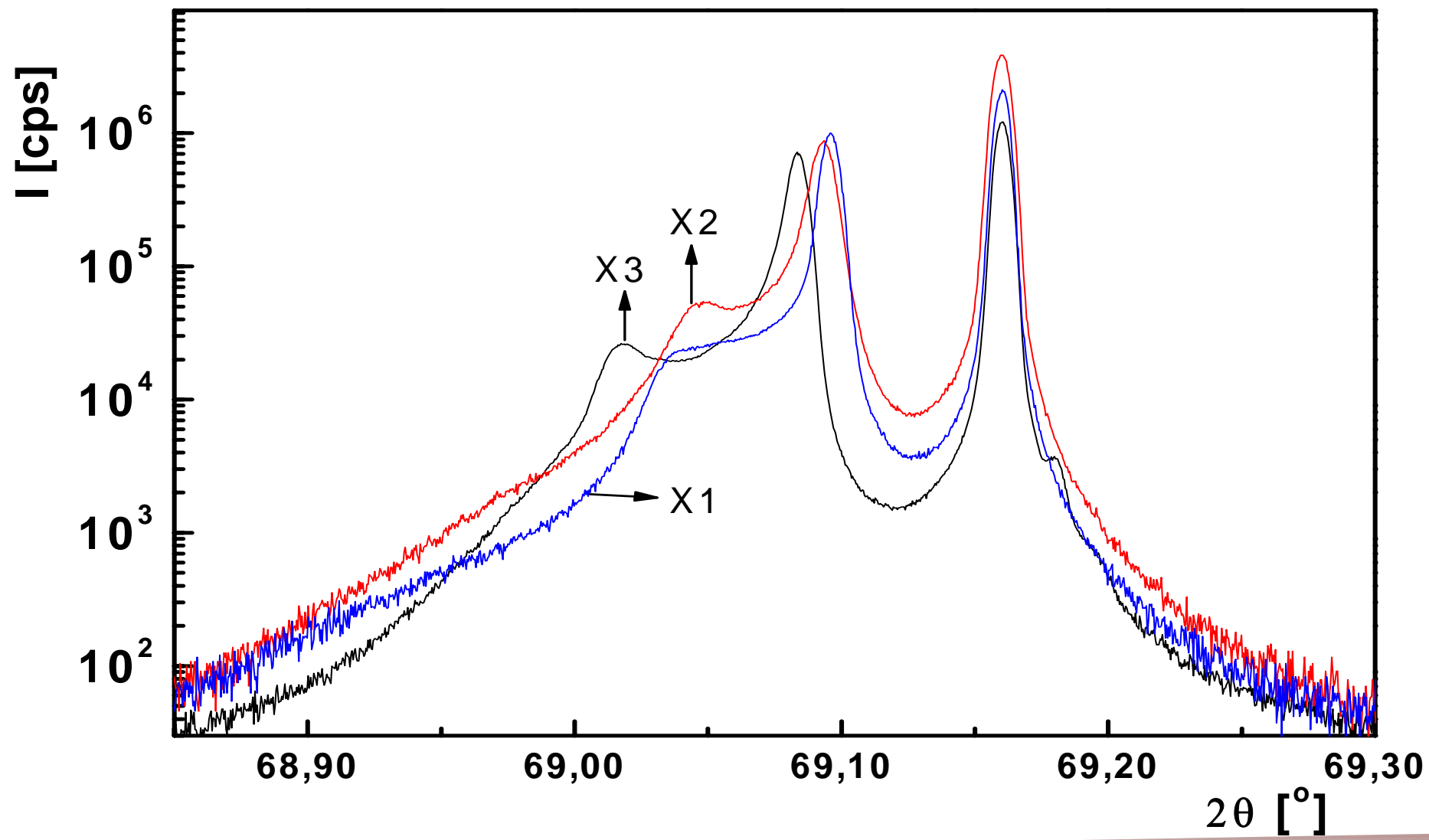
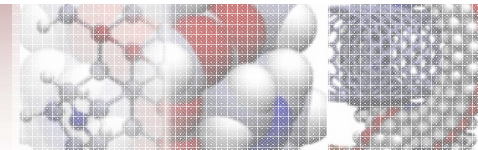
7. Metoda B, DLC (diamond like Carbon)/SiO₂ nativ/Si, XRR

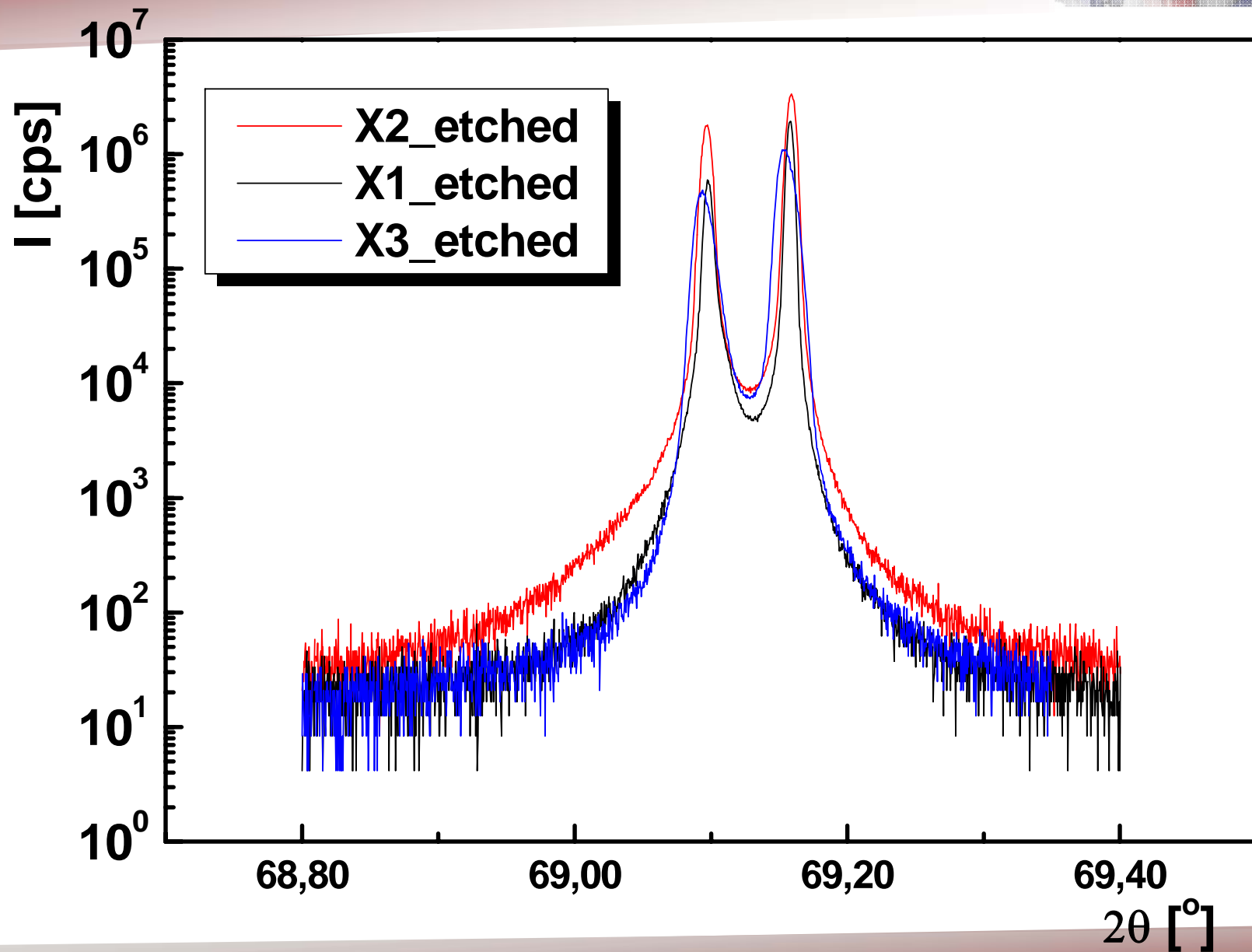
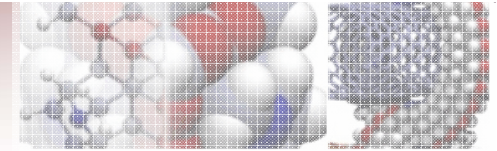


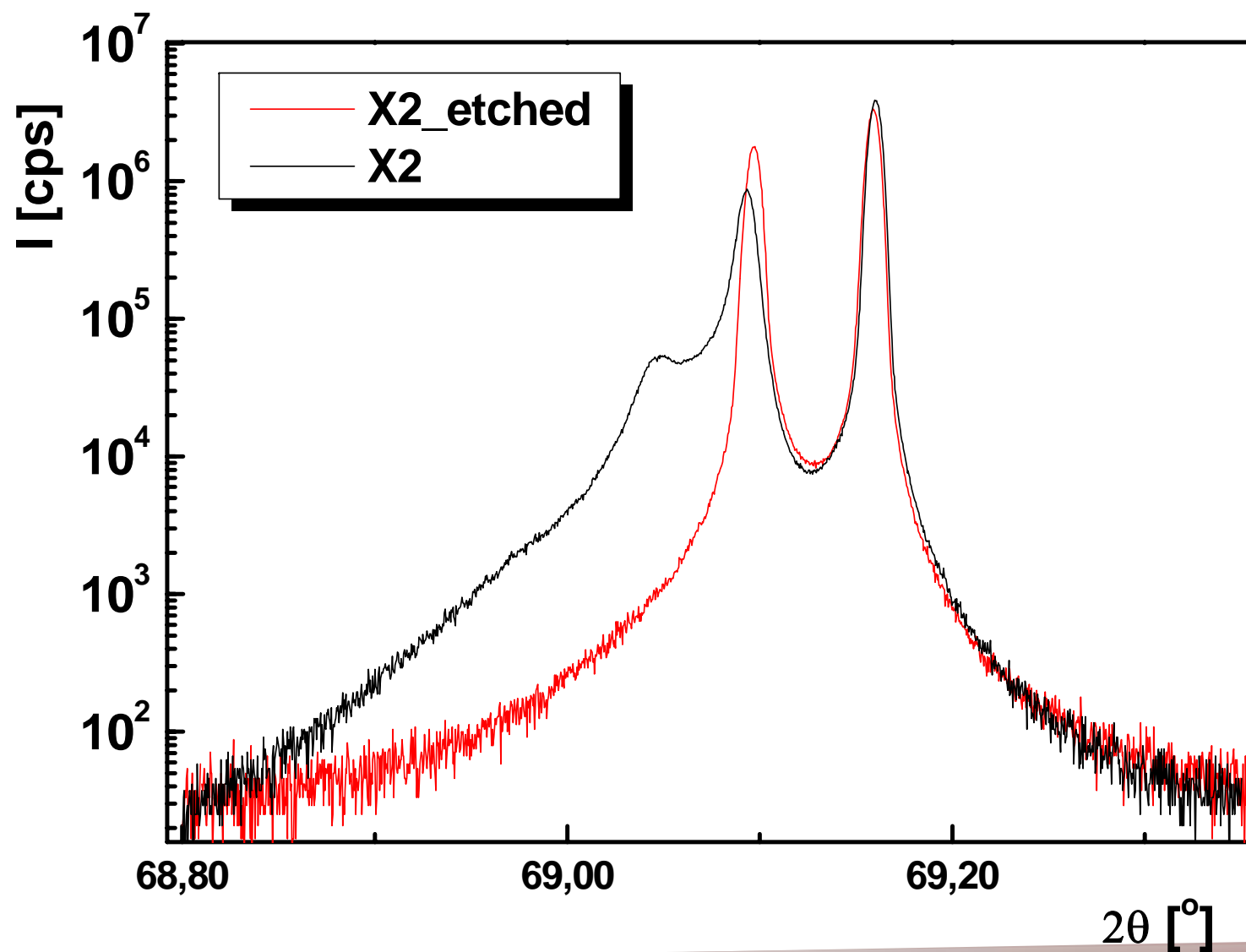
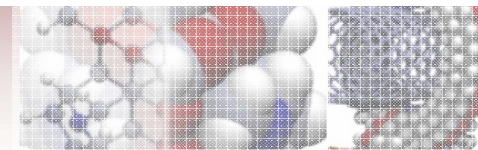
8. HRMRXR D Straturi PS-Si (Si poros) cu gradient de deformare

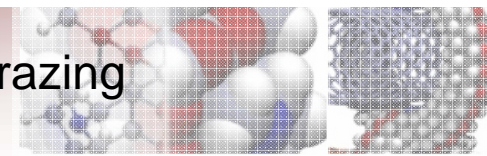


	SAXS	SEM	SEM	
	Particle Size [nm]	Pore Size [nm]	Grosime PS [μm]	Densitate pori [%]
X1 10mA/cm²	4,20	4,7	5,83	52,81
X2 25mA/cm²	3,70	6,6	10,82	64,08
X3 40mA/cm²	3,32	8,5-10,0	15,70	75,08



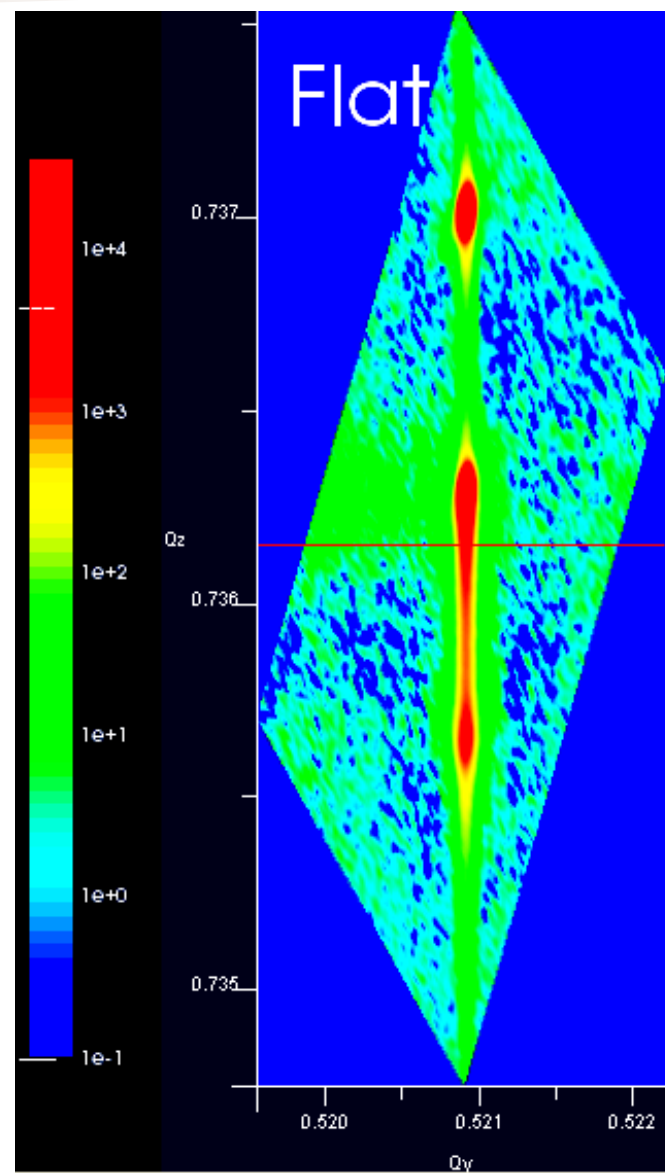
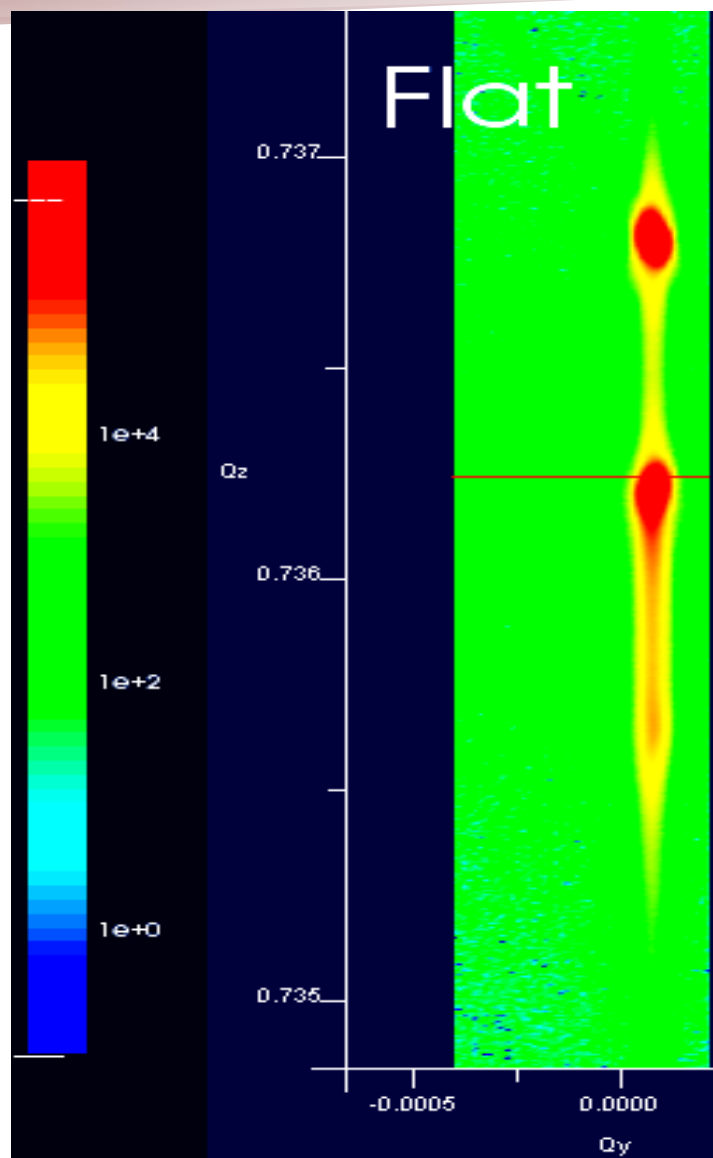




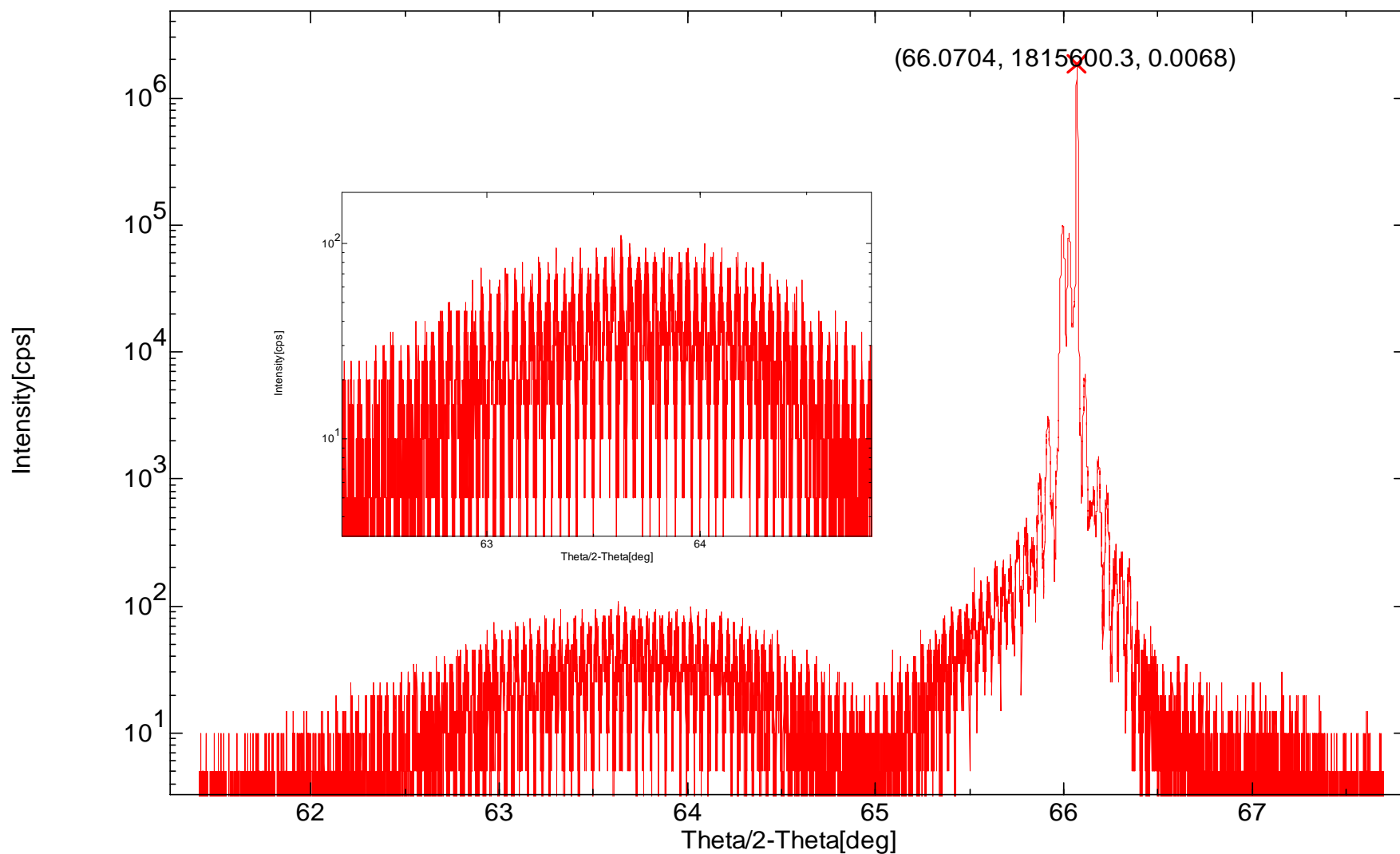
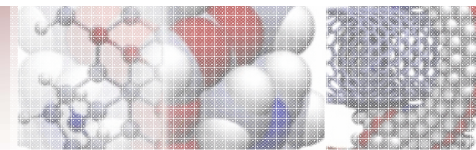


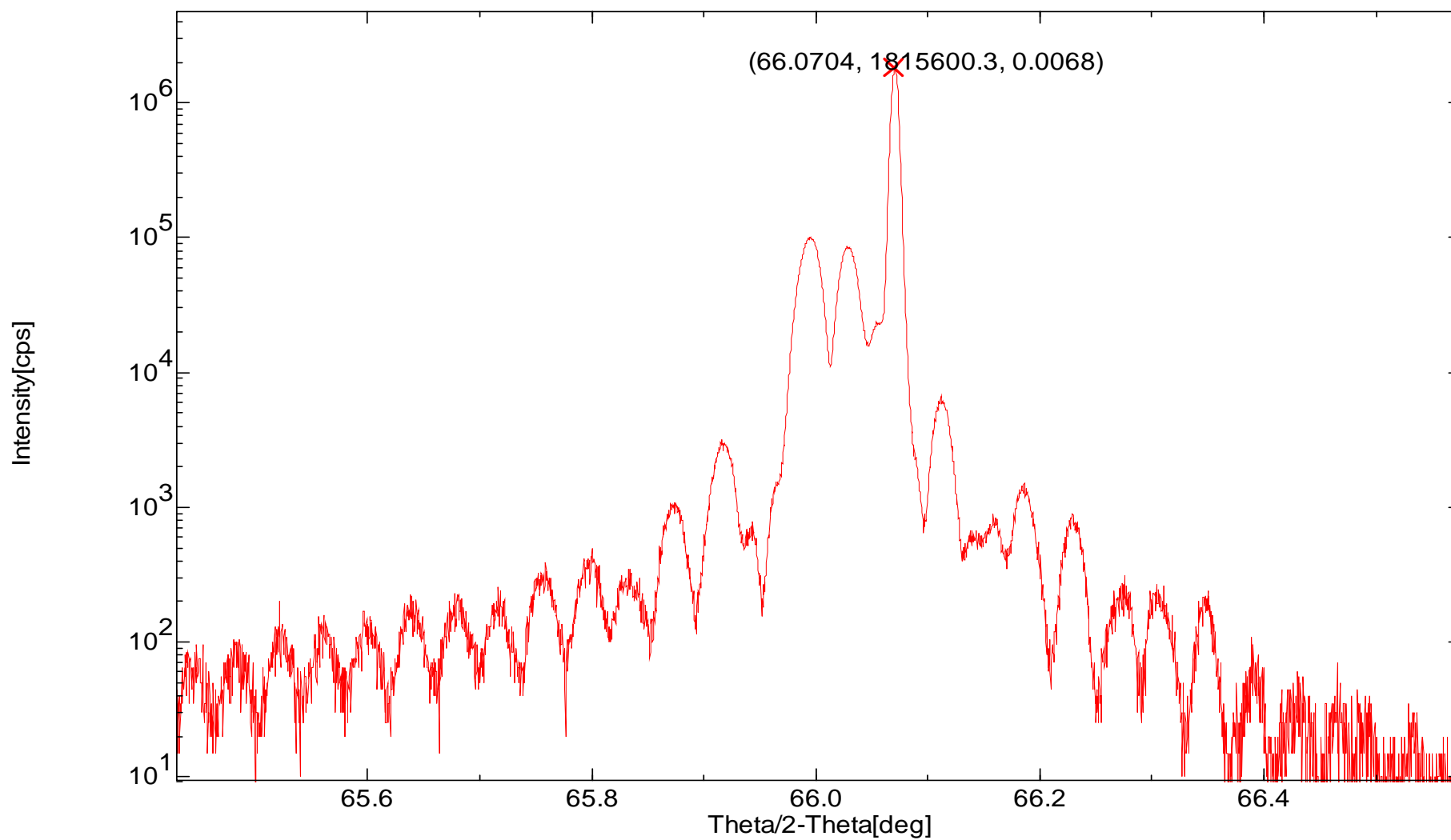
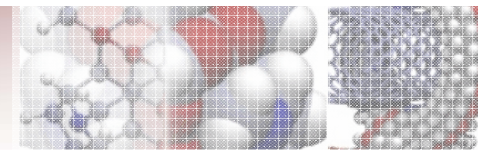
RSM symmetric space map
Si(004), X2 before etch

RSM asymmetric space map, grazing
exit, **Si(224)**, X3 before etch

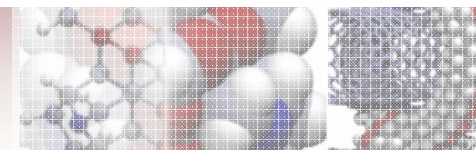


9. HRMXRD Laser structures, Superlattices + QD + MQW, InGaAs/GaAs AlGaAs Buffer on GaAs (004)



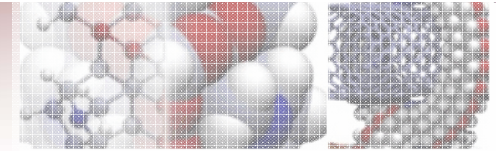


Structura fitata

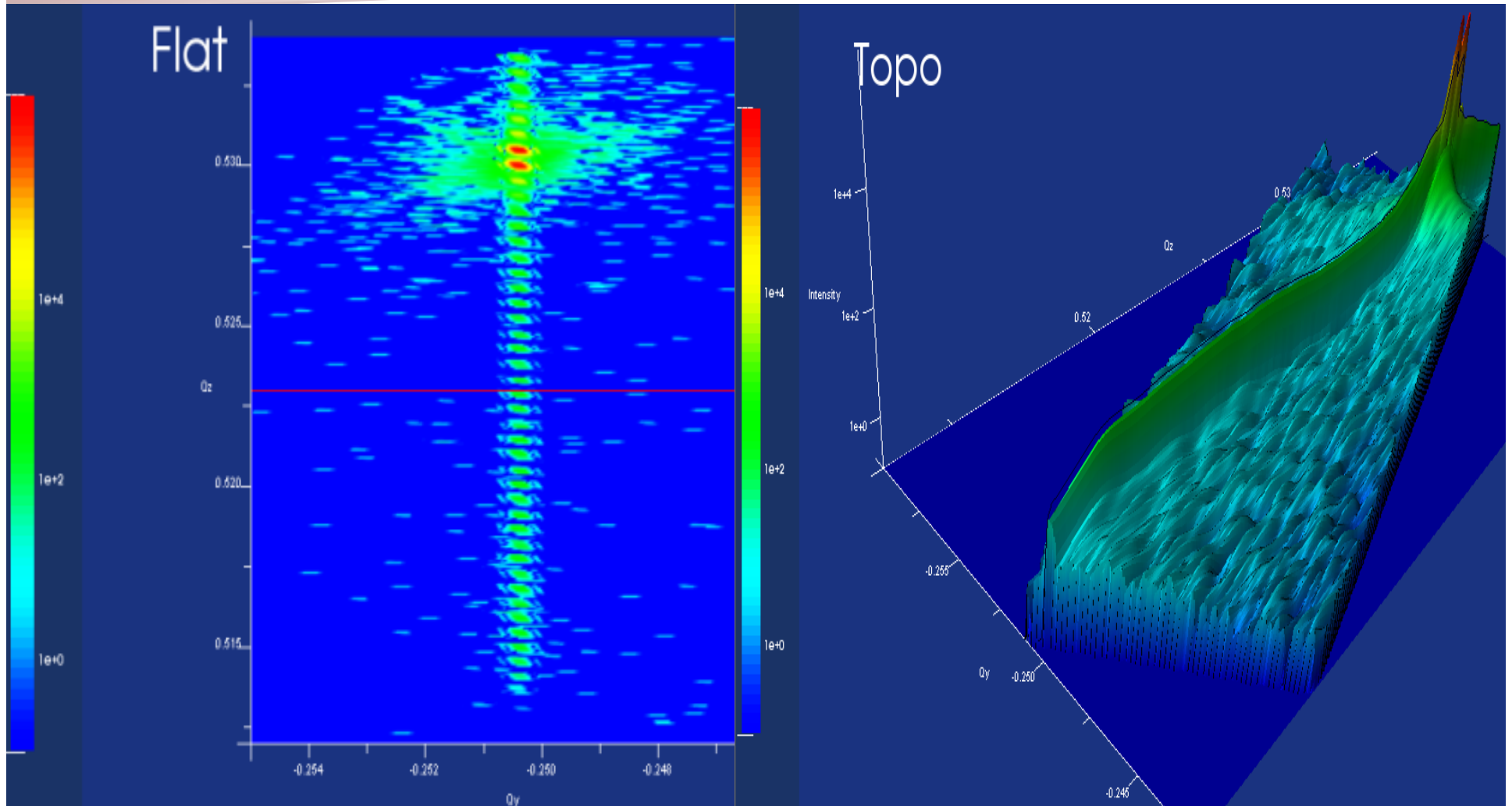


InGaAs	1.2nm	
GaAs	15.0nm	
:		} 20 Period
AlGaAs	2.0nm	
GaAs	1.3nm	
AlGaAs	100.0nm	
:		} 20 Period
GaAs	1.3nm	
AlGaAs	2.0nm	
GaAs	15.0nm	
InGaAs	1.2nm	
GaAs	15.0nm	
:		} 20 Period
AlGaAs	2.0nm	
GaAs	1.3nm	
AlGaAs	100.0nm	
SUBSTRATE : GaAs		
(001,004)		

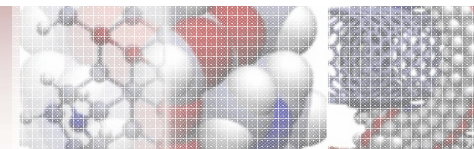
InGaAs	8.0nm	
GaAs	17.0nm	
:		} 20 Period
AlGaAs	2.6nm	
GaAs	1.5nm	
AlGaAs	106.8nm	
:		} 20 Period
GaAs	1.4nm	
AlGaAs	1.9nm	
GaAs	15.1nm	
InGaAs	6.9nm	
GaAs	14.3nm	
:		} 20 Period
AlGaAs	2.5nm	
GaAs	1.1nm	
GaAs	1.3nm	
AlGaAs	101.6nm	
SUBSTRATE : GaAs		
(001,004)		



RSM InGaAs/GaAs

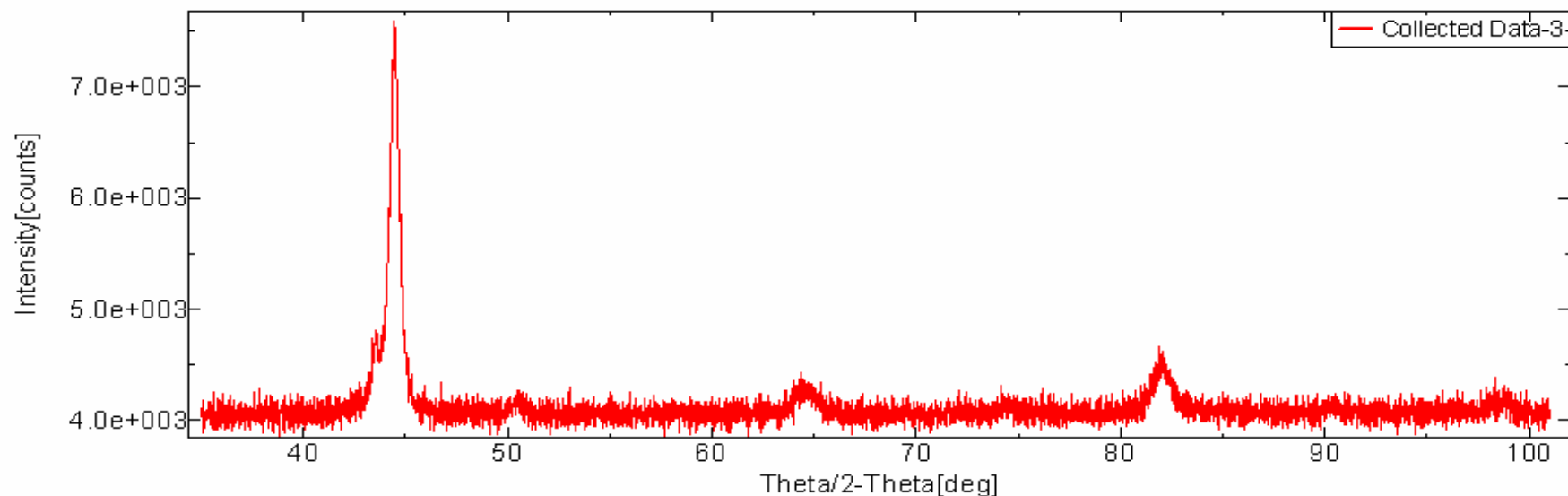


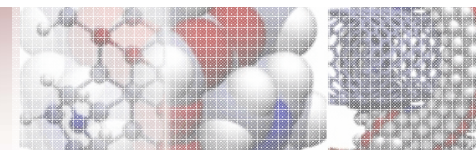
10. Determinarea continutului de Austenita reziduala (oteluri)



V1517_PB.ras / Collected Data-3

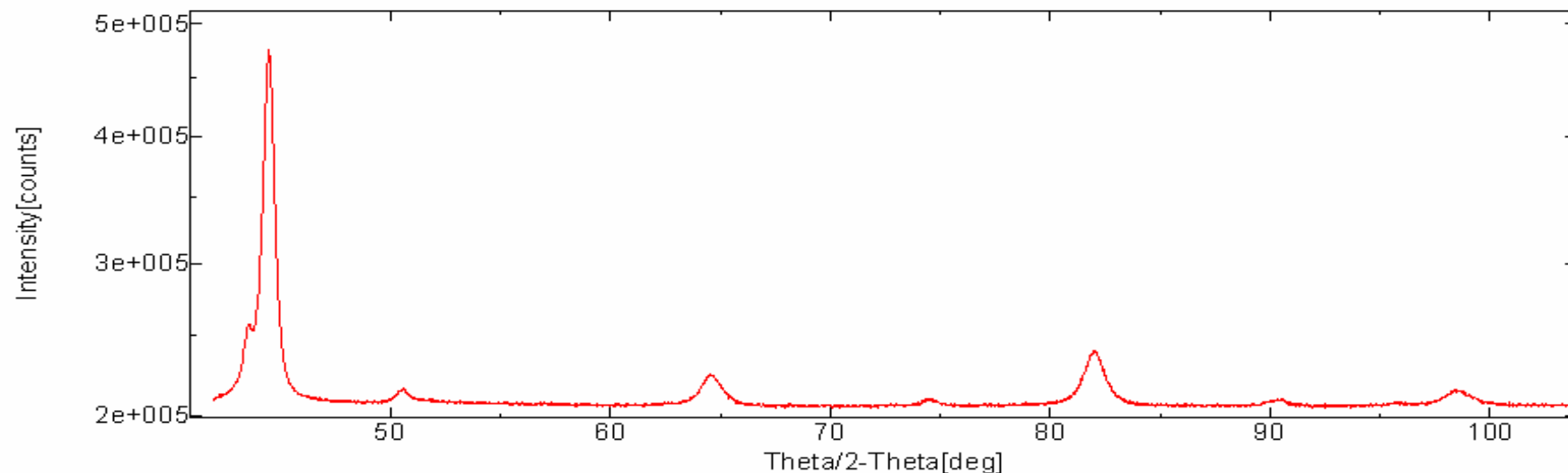
Sample Name	: V1517	Scan Mode	: CONTINUOUS	CBO selection slit	: PB
Comment	: None	Scan Speed	: 6.0000 deg/min	Incident parallel slit	: Soller_slit_5.0deg
Date	: 05/03/11 14:29:14	Scan Step	: 0.0100 deg	Incident slit	: 1.000mm
Operator	: Admin	Scan Axis	: Theta/2-Theta	Length limiting slit	: 15.0mm
X-Ray	: 45 kV , 200 mA	Scan Range	: 35.0000 - 101.0000 deg	Receiving slit # 1	: 1.000mm
Wavelength	: CuKa / 1.541867 A	2-Theta	: 34.9950 deg	Filter	: None
Goniometer	: SmartLab(In-plane)	Omega	: 17.4975 deg	Receiving optical device	: PSA_open
Base Att. Config.	: Std. chi cradle	Chi	: 0.000 deg	Receiving parallel slit	: Soller_slit_5.0deg
Attachment	: RxRy	Phi	: 0.000 deg	Receiving slit # 2	: 1.000mm
Detector	: SC-70	2-ThetaChi	: 0.0000 deg	Attenuator	: 1/10000
Memo	: Austenita reziduala			Diffracted beam mono.	: None
Optics Attribute	: Medium resolution parallel beam/RS			Monochromator slit	: None

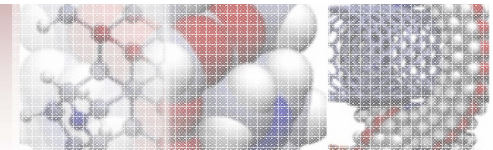




V1517_BB_L.ras / Collected Data-4

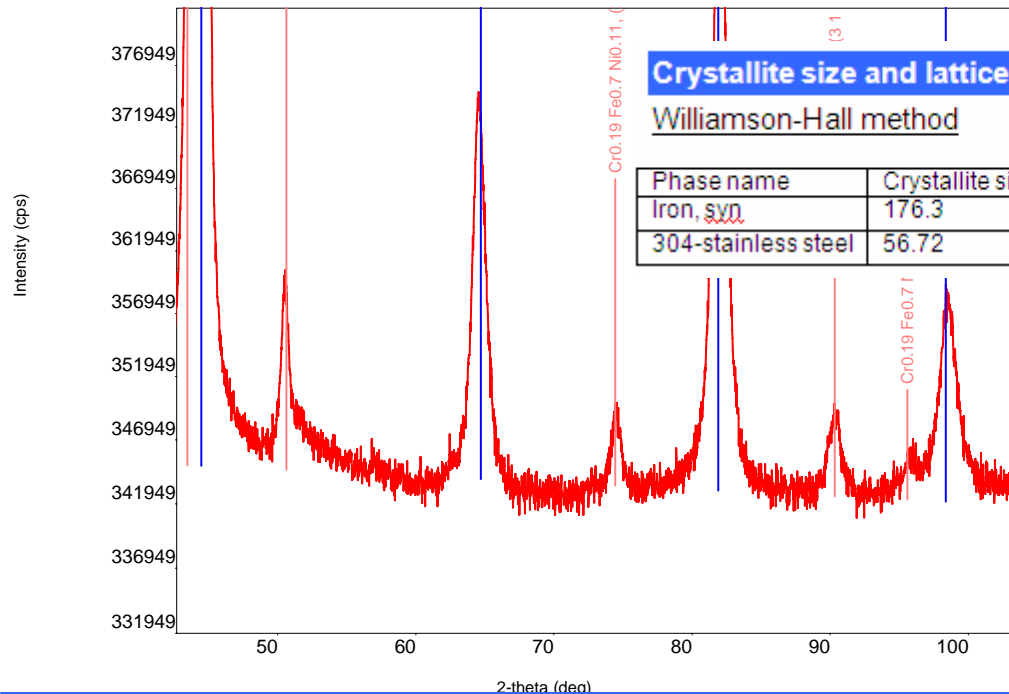
Sample Name	: V1517_BB	Scan Mode	: CONTINUOUS	CBO selection slit	: BB
Comment	: austenita rez	Scan Speed	: 2.0000 deg/min	Incident parallel slit	: Soller_slit_5.0deg
Date	: 05/03/11 16:15:38	Scan Step	: 0.0200 deg	Incident slit	: 2/3deg
Operator	: Admin	Scan Axis	: Theta/2-Theta	Length limiting slit	: 15.0mm
X-Ray	: 45 kV , 200 mA	Scan Range	: 42.0000 - 104.0000 deg	Receiving slit # 1	: 4.000deg
Wavelength	: CuK α / 1.541867 Å	2-Theta	: 0.0000 deg	Filter	: None
Goniometer	: SmartLab(In-plane)	Omega	: 0.0000 deg	Receiving parallel slit	: Soller_slit_5.0deg
Base Att. Config.	: Std. chi cradle	Chi	: 0.000 deg	Receiving slit # 2	: 13.000mm
Attachment	: RxRy	Phi	: 0.000 deg	Attenuator	: Open
Detector	: D/teX Ultra	2-ThetaChi	: 0.0000 deg	Diffracted beam mono.	: No_unit
Memo	: AUSTENITA, BB			Monochromator slit	: None
Optics Attribute	: Bragg-Brentano focusing				





Qualitative analysis results

Phase name	Formula	Figure of merit	Phase reg. detail	DB card number
Iron, syn	Fe	0.283	ICDD (PDF2.DAT)	00-006-0696
304-stainless steel	Cr0.19 Fe0.7 Ni0.11	0.492	ICDD (PDF2.DAT)	00-033-0397



Measurement conditions

X-Ray	45 kV , 200 mA	Scan speed / Duration time	2.0000 deg./min.
Goniometer		Step width	0.0200 deg.
Attachment	-	Scan axis	2theta/theta
Filter		Scan range	42.0000 - 104.0000 deg.
CBO selection slit	-	Incident slit	2/3deg
Diffacted beam mono.	No_unit	Length limiting slit	-
Detector	D/teX Ultra	Receiving slit #1	4.000deg
Scan mode	CONTINUOUS	Receiving slit #2	13.000mm

Target phase: 304-stainless steel

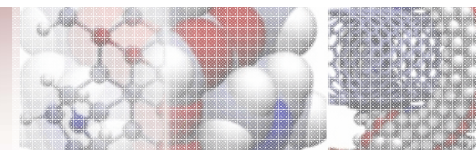
No.	2-theta (deg)	Int. W
<input checked="" type="checkbox"/> 1	43.482(4)	0.597(17)
<input checked="" type="checkbox"/> 3	50.46(3)	1.62(5)
<input checked="" type="checkbox"/> 5	74.40(5)	0.75(7)
<input checked="" type="checkbox"/> 7	90.27(5)	1.05(8)
<input checked="" type="checkbox"/> 8	95.9239	1.587582

Width correction
 No correction
 Use external standard
 Use internal standard
 Internal standard: Iron, syn

Analyze: Lattice strain and crystallite size
 Method: Halder-Wagner method

Formula: $Y = 0.028X - 0.0005$
 Crystallite size(ang.): 59(10)
 Lattice strain(%): 0.000000e+000

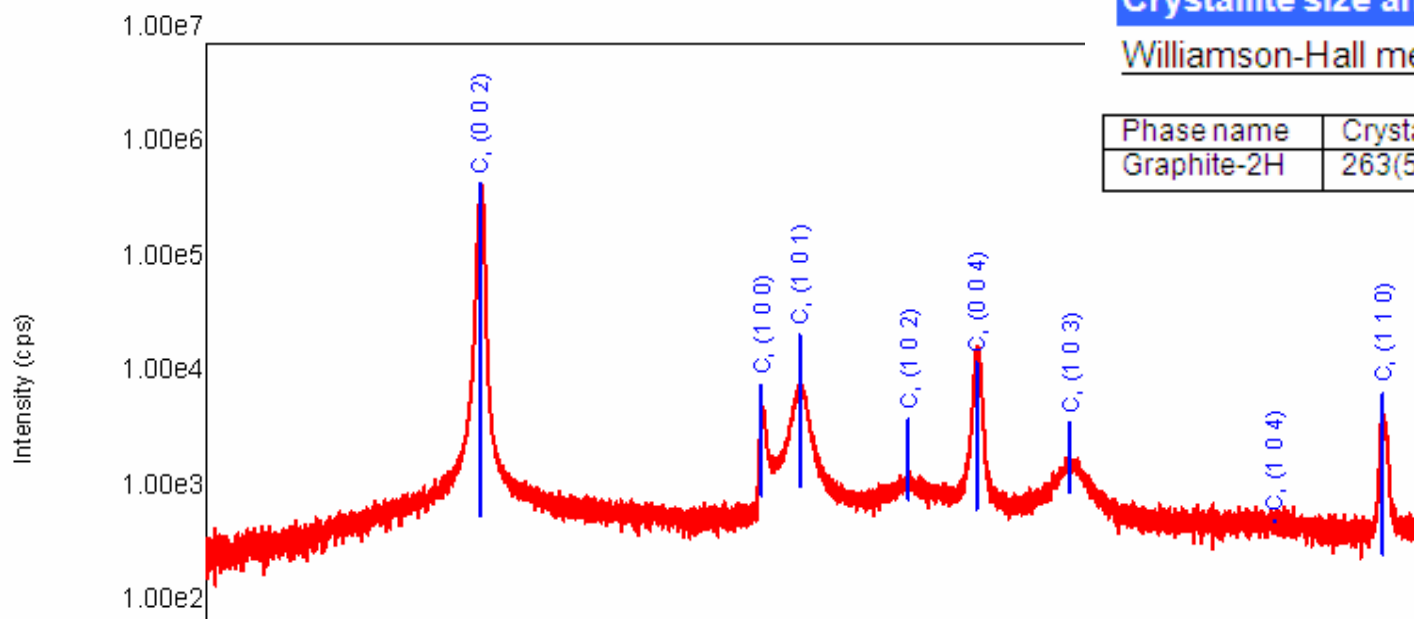
11. Pulbere Grafit de puritate spectrala (Cehia), D=26nm



Crystallite size and lattice strain

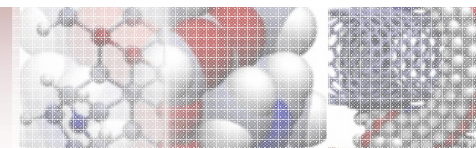
Williamson-Hall method

Phase name	Crystallite size(A)	Strain (%)
Graphite-2H	263(54)	0.14(19)

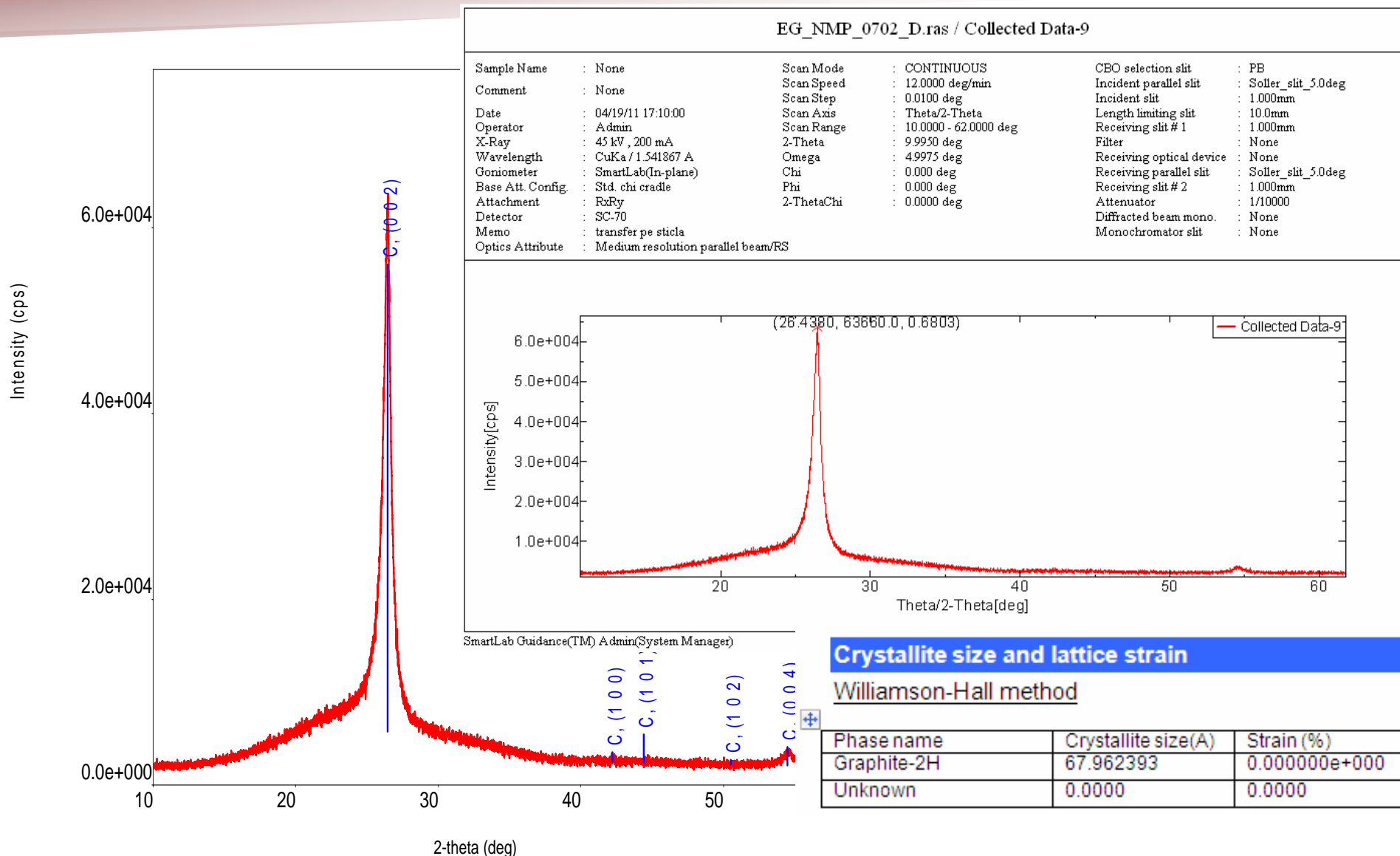


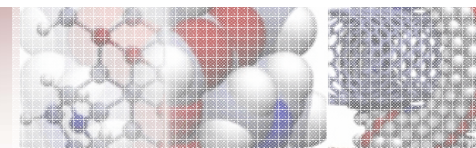
Measurement conditions

X-Ray	45 kV , 200 mA	Scan speed / Duration time	6.0000 deg./min.
Goniometer		Step width	0.0100 deg.
Attachment	-	Scan axis	2theta/theta
Filter		Scan range	11.0000 - 80.0000 deg.
CBO selection slit	-	Incident slit	1.000mm
Diffrected beam mono.	No_unit	Length limiting slit	-
Detector	SC-70	Receiving slit#1	1.000mm
Scan mode	CONTINUOUS	Receiving slit#2	1.000mm



12. Film grafene multilayer (Si, Sticla) D=6.7nm

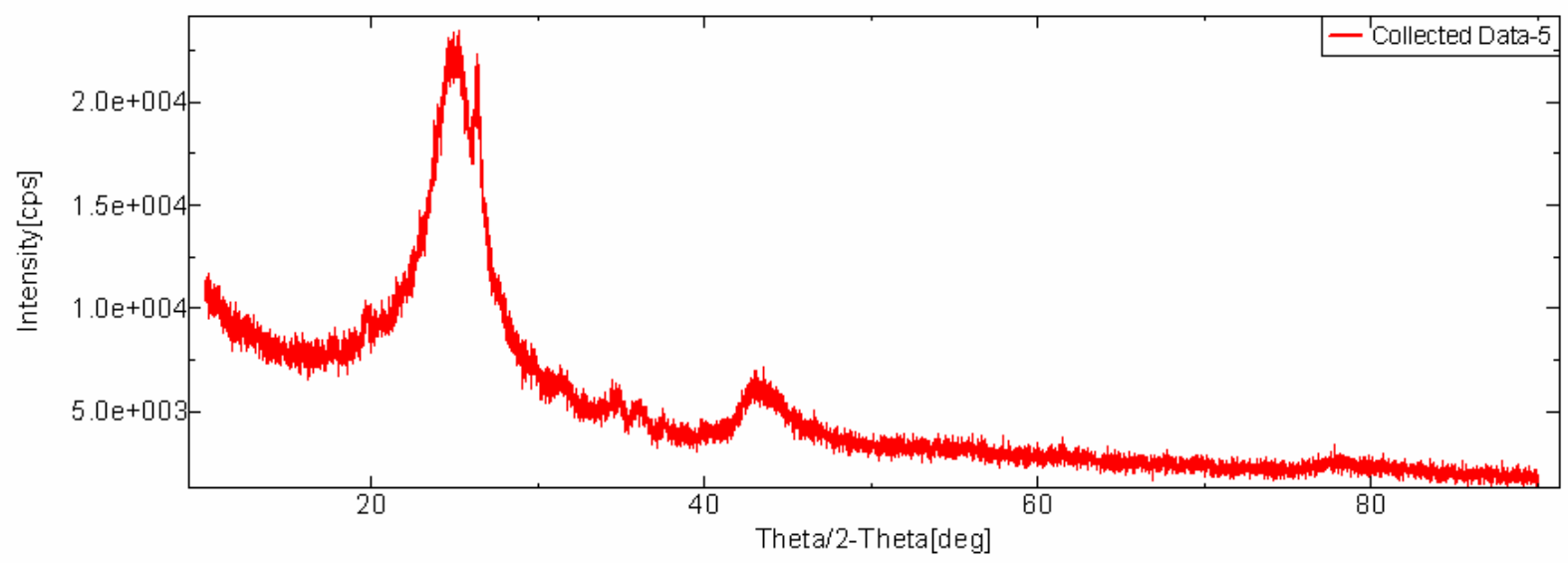


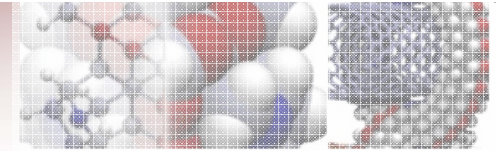


13. Film carbon (GO – oxid de grafit), strat C redus

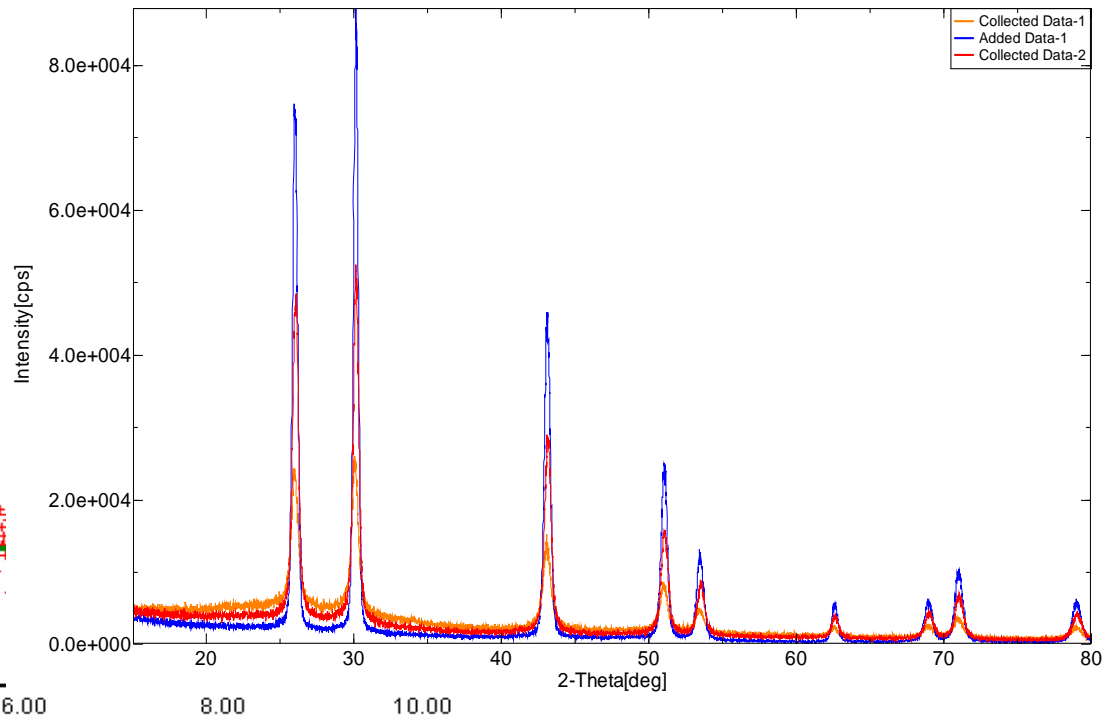
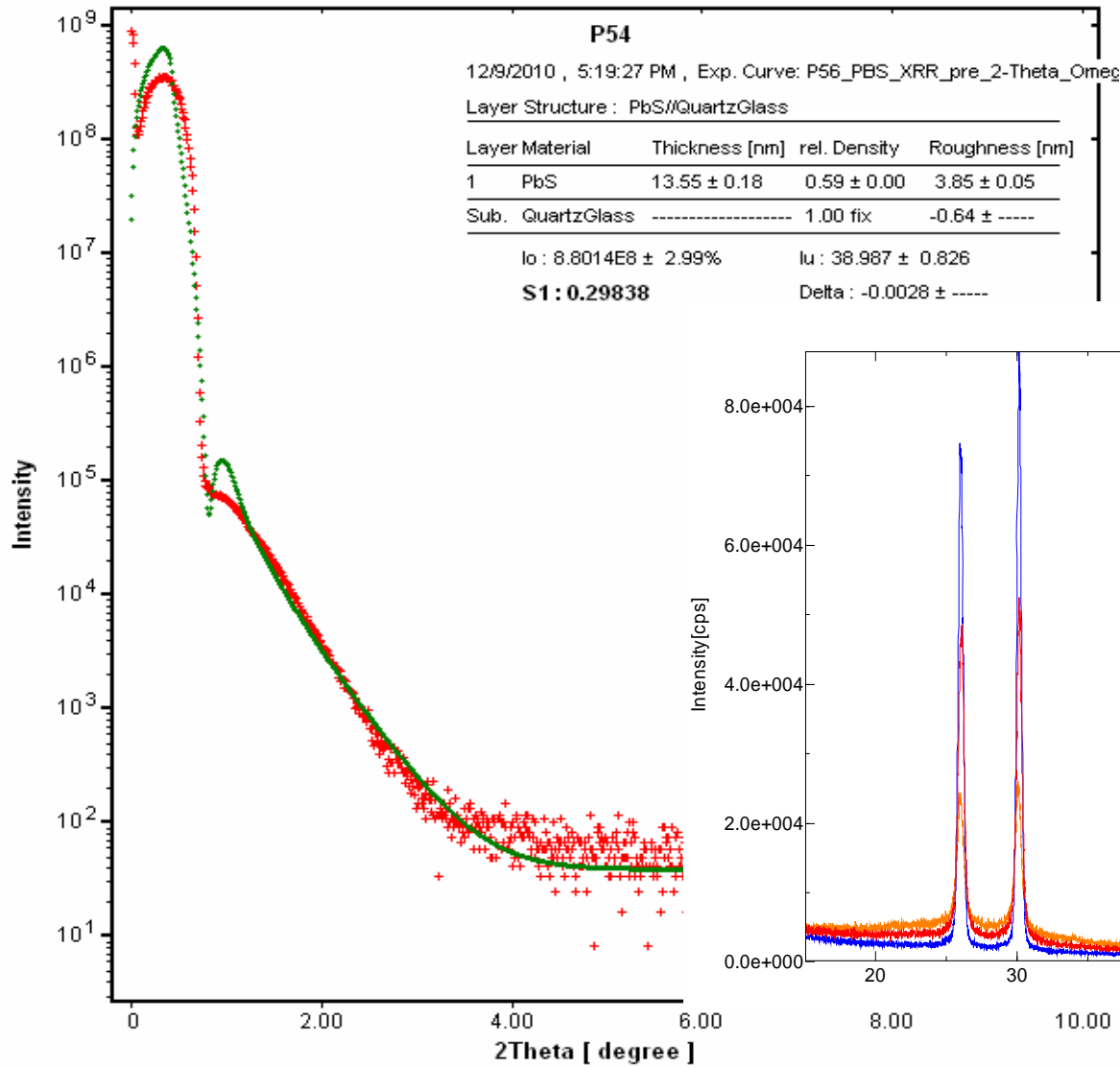
r-GOX SCW_T.ras / Collected Data-5

Sample Name	: None	Scan Mode	: CONTINUOUS	CBO selection slit	: PB
Comment	: None	Scan Speed	: 12.0000 deg/min	Incident parallel slit	: Soller_slit_5.0deg
Date	: 04/12/11 12:54:20	Scan Step	: 0.0100 deg	Incident slit	: 1.000mm
Operator	: Admin	Scan Axis	: Theta/2-Theta	Length limiting slit	: 10.0mm
X-Ray	: 45 kV , 200 mA	Scan Range	: 10.0000 - 90.0000 deg	Receiving slit # 1	: 1.000mm
Wavelength	: CuK α 1 / 1.540593 A	2-Theta	: 9.9950 deg	Filter	: None
Goniometer	: SmartLab(In-plane)	Omega	: 4.9975 deg	Receiving optical device	: PSA_open
Base Att. Config.	: Std. chi cradle	Chi	: 0.000 deg	Receiving parallel slit	: Soller_slit_5.0deg
Attachment	: RxRy	Phi	: 0.000 deg	Receiving slit # 2	: 1.000mm
Detector	: SC-70	2-ThetaChi	: 0.0000 deg	Attenuator	: 1/10000
Memo	: None			Diffracted beam mono.	: None
Optics Attribute	: Medium resolution parallel beam/RS			Monochromator slit	: None

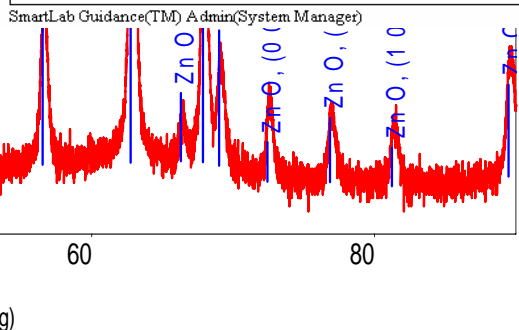
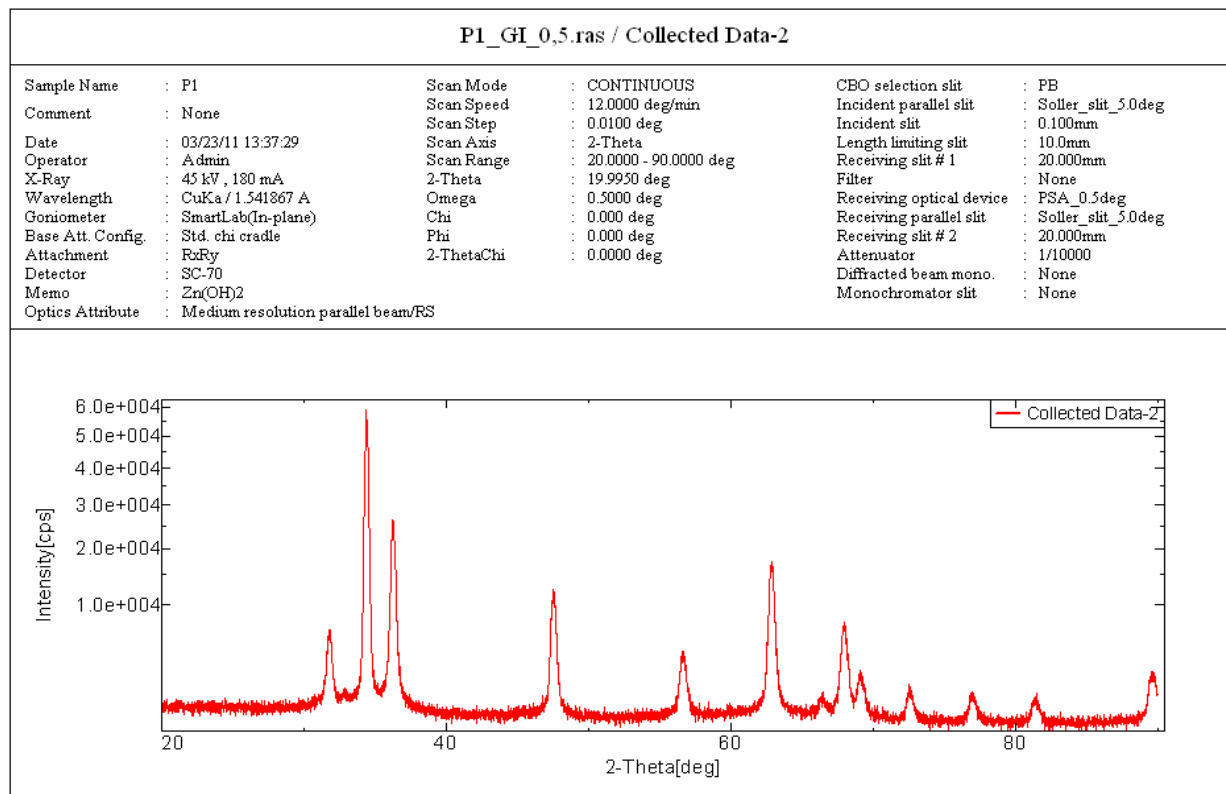
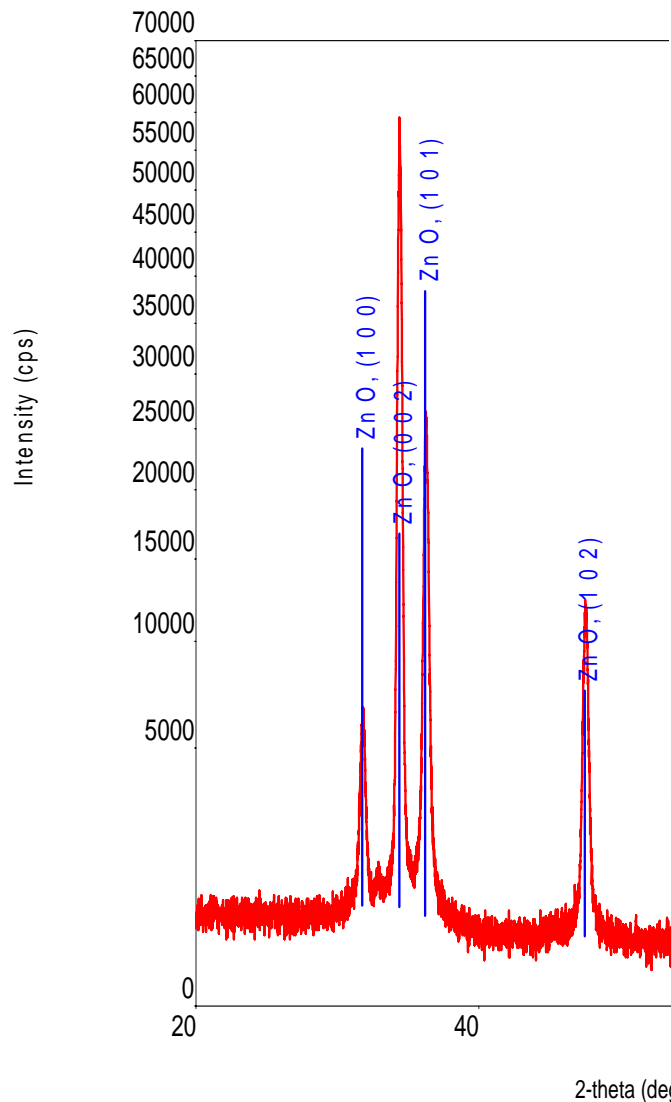
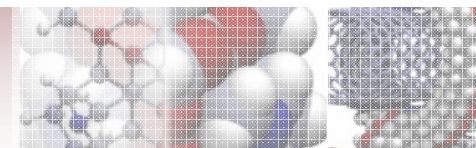




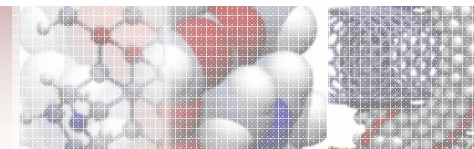
14. Filme PbS texturate, GIXRD, XRR



15. Film ZnO, GIXRD

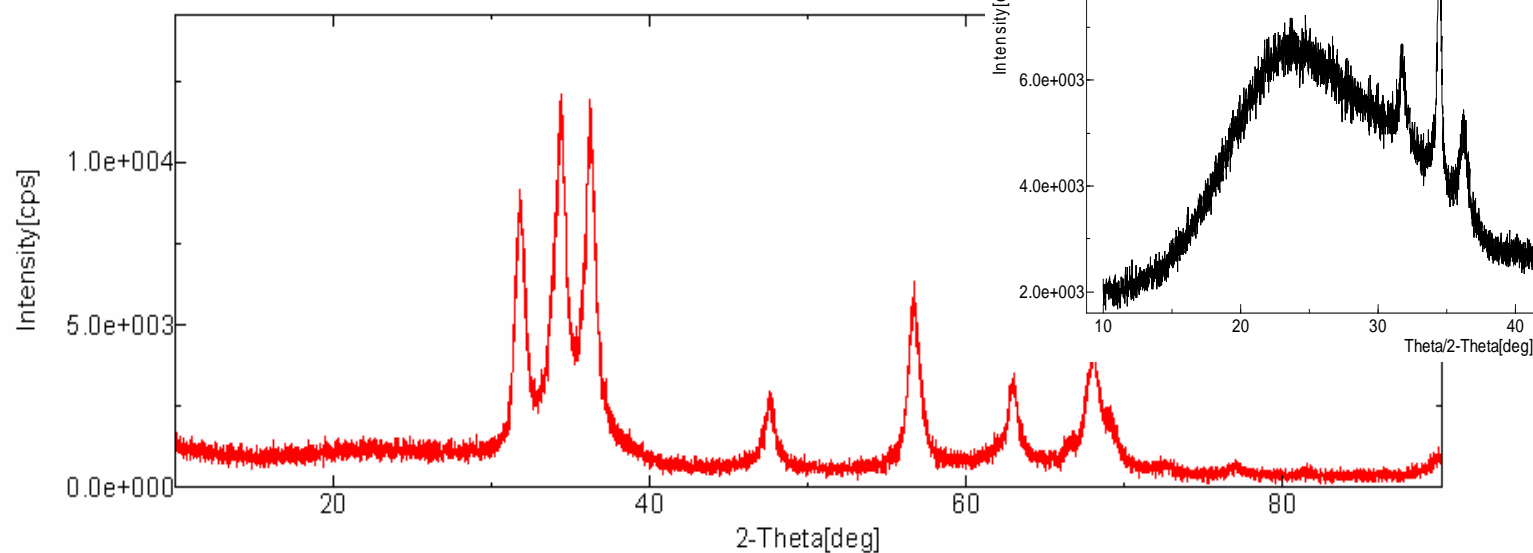


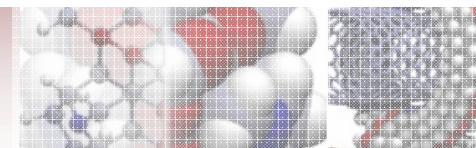
17. Nanofire de ZnO/Sticla, GIXRD+ WAXRD



P5_PB_GLras / Collected Data-1

Sample Name	: P5	Scan Mode	: CONTINUOUS	CBO selection slit	: PB
Comment	: None	Scan Speed	: 12.0000 deg/min	Incident parallel slit	: Soller_slit_5.0deg
Date	: 04/13/11 14:19:13	Scan Step	: 0.0100 deg	Incident slit	: 0.100mm
Operator	: Admin	Scan Axis	: 2-Theta	Length limiting slit	: 10.0mm
X-Ray	: 45 kV, 200 mA	Scan Range	: 10.0000 - 90.0000 deg	Receiving slit # 1	: 20.000mm
Wavelength	: CuK α 1 / 1.540593 A	2-Theta	: 9.9950 deg		
Goniometer	: SmartLab(In-plane)	Omega	: 0.5000 deg		
Base Att. Config.	: Std. chi cradle	Chi	: 0.000 deg		
Attachment	: RxRy	Phi	: 0.000 deg		
Detector	: SC-70	2-ThetaChi	: 0.0000 deg		
Memo	: None				
Optics Attribute	: Medium resolution parallel beam/RS				





18. Pulbere ZnO, Proiect FP7 Nanosustain (D= 8.7nm)

Comparatie detector 0D NaI (SC70) cu 1D (Dtex)

Analysis Results

Crystallite size and lattice strain

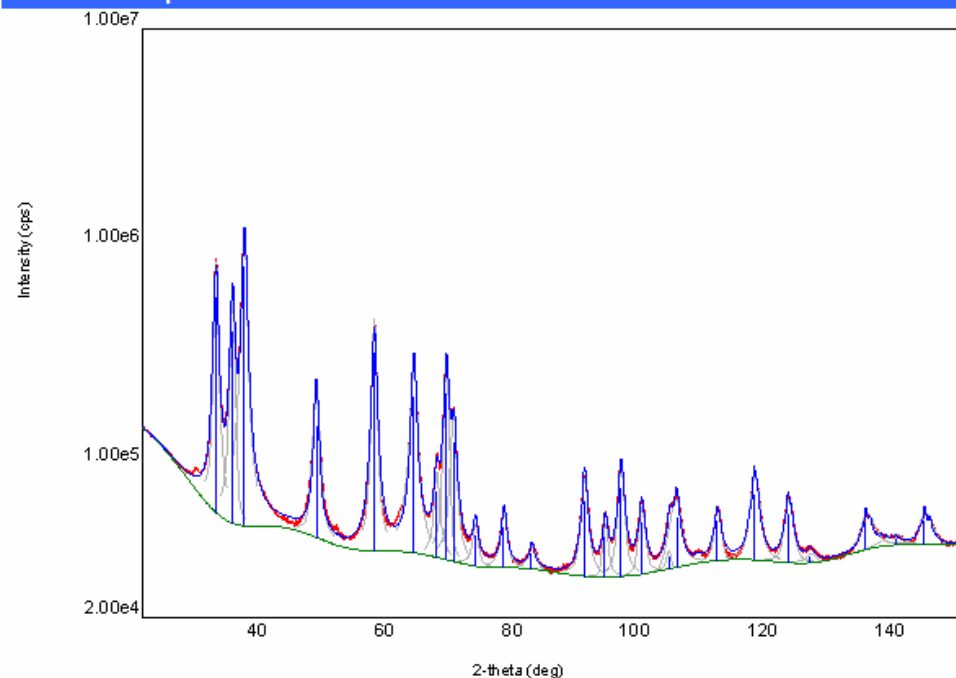
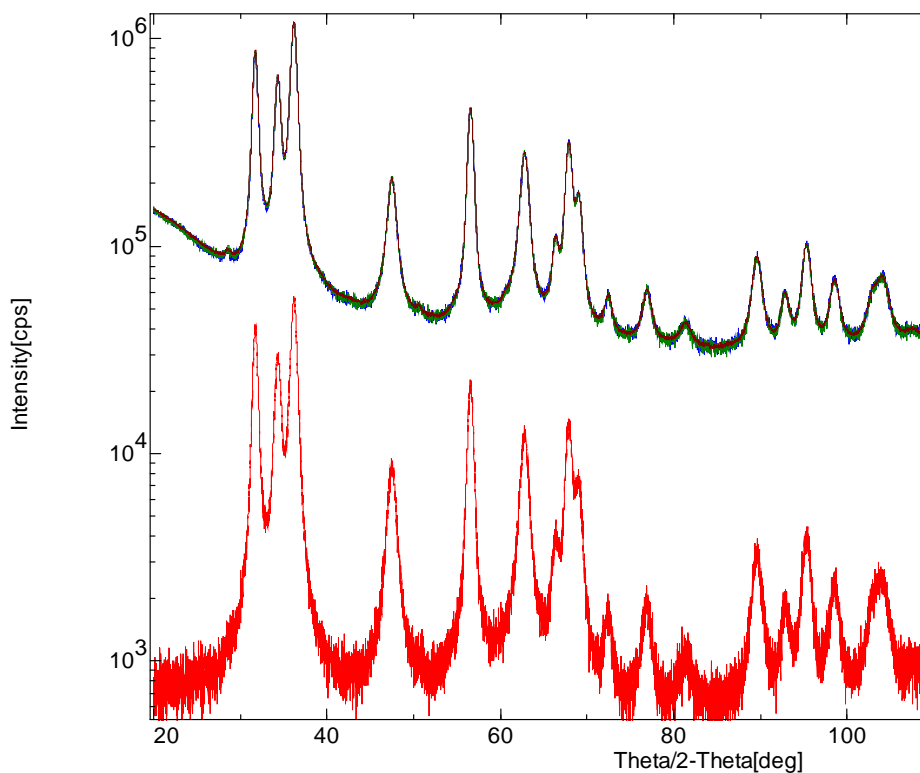
Williamson-Hall method

Phase name	Crystallite size(A)	Strain (%)
Zinc Oxide	87(6)	0.20(19)

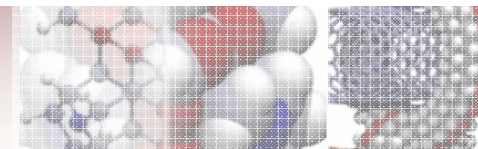
General Information

Analysis date	2/11/2011 8:23:55 PM	Measurement date
Sample name	NRCWE_031_203	Operator
File name	NRCWE_031.asc	
Comment		

Measurement profile

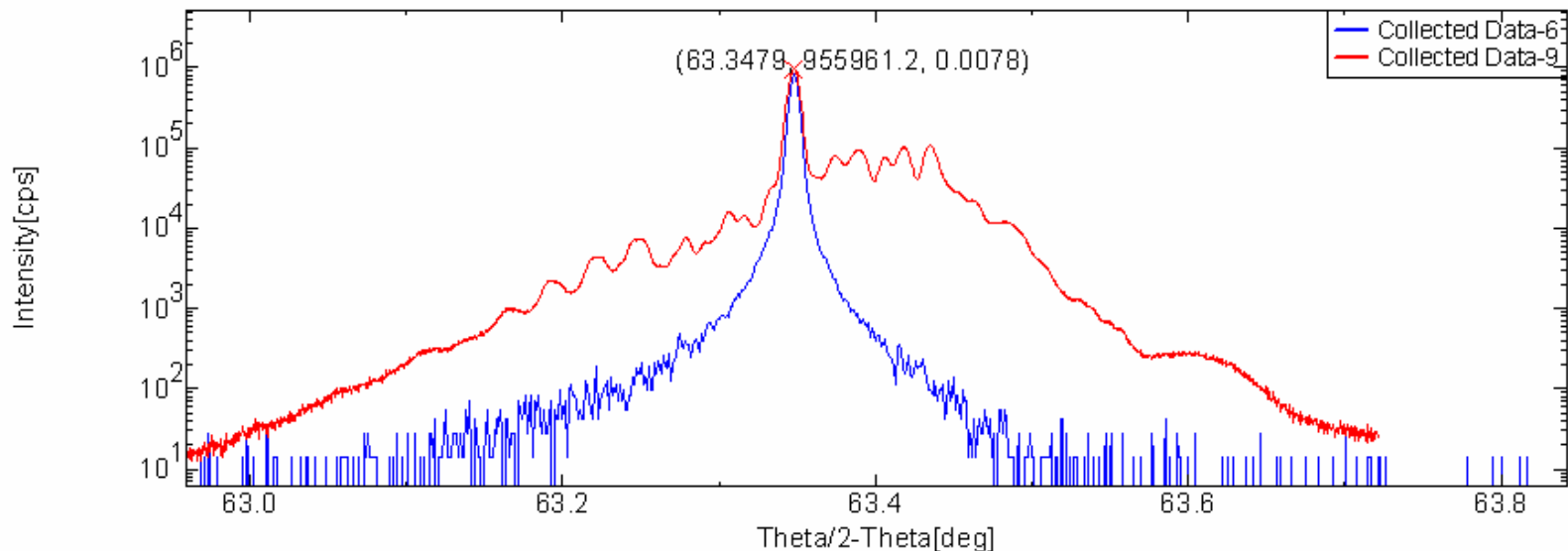


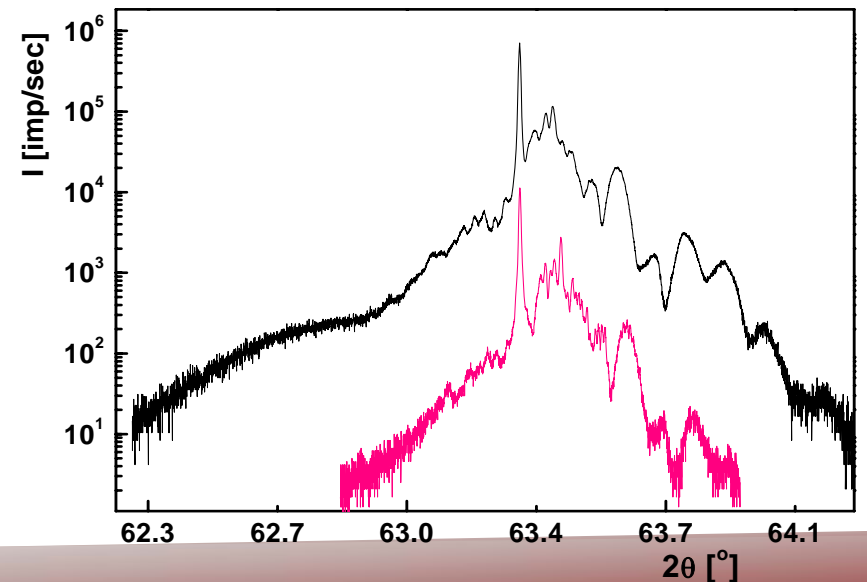
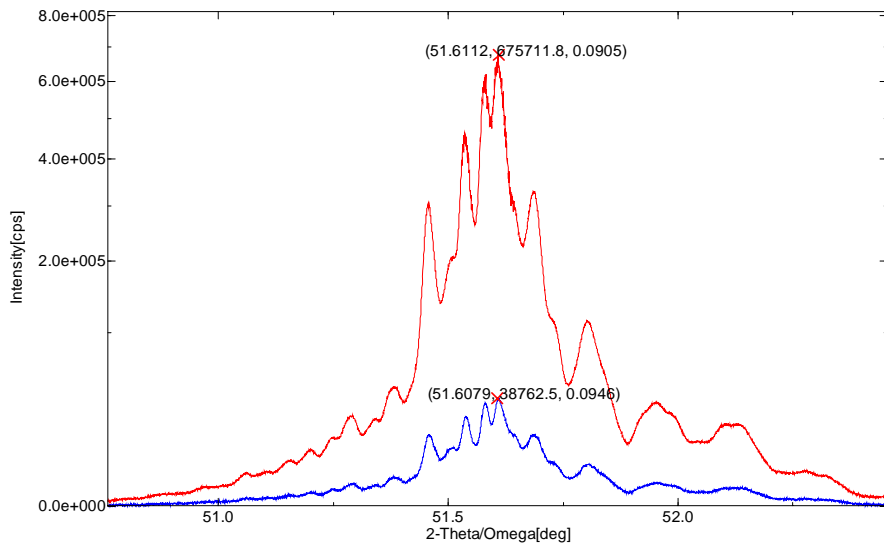
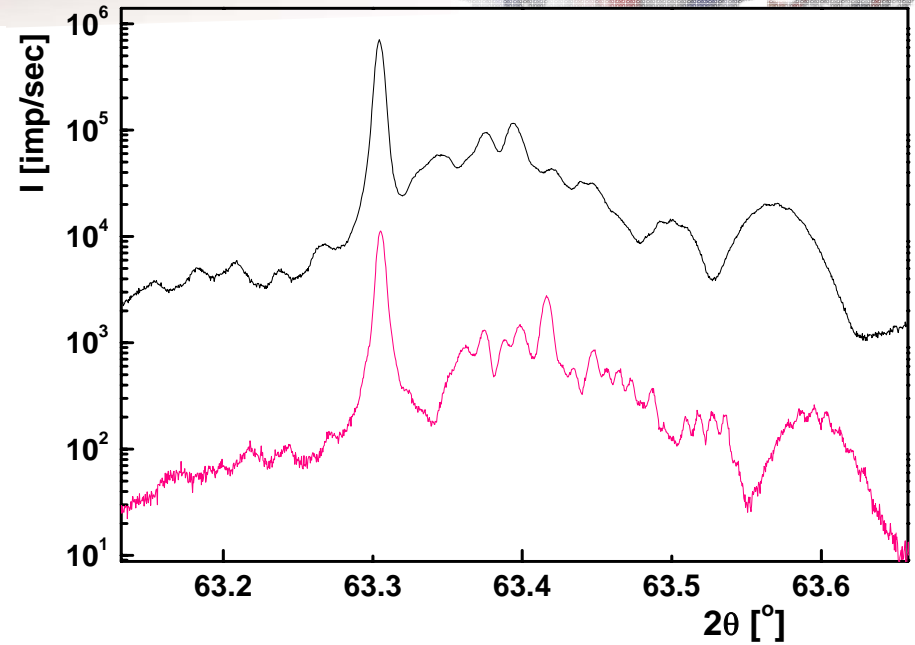
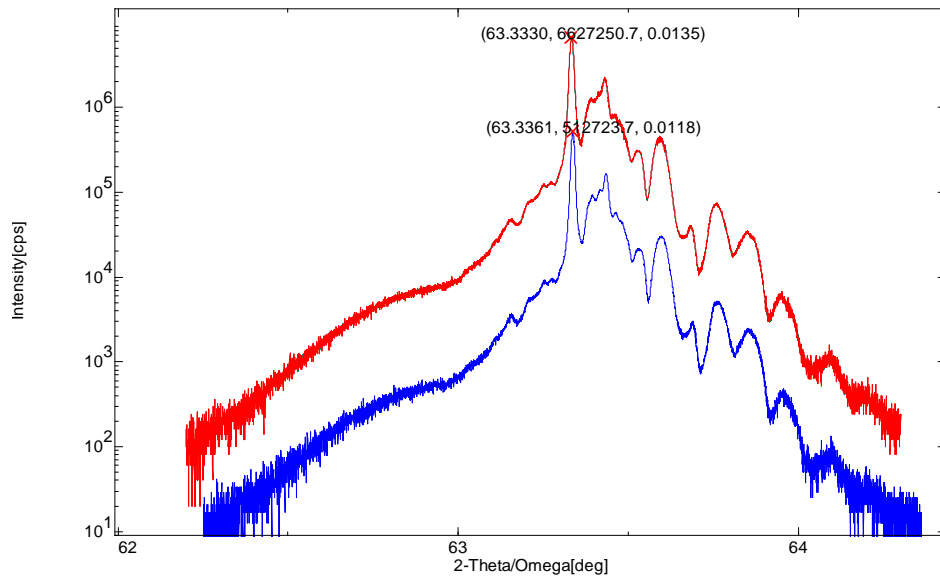
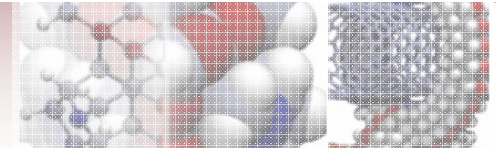
19. HBT InP/InGaAS, Comparatie rezolutie monocromatoare



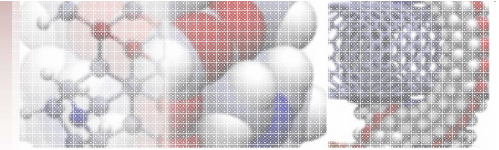
InP_npn_6l_b.ras / Collected Data-9

Sample Name	: None	Scan Mode	: CONTINUOUS	CBO selection slit	: PB
Comment	: None	Scan Speed	: 0.0100 deg/min	Incident optical device	: Ge(220)x4
Date	: 02/05/10 20:54:57	Scan Step	: 0.0004 deg	Incident parallel slit	: No_unit
Operator	: Admin	Scan Axis	: Theta/2-Theta	Incident slit	: 1.000mm
X-Ray	: 45 kV , 200 mA	Scan Range	: 62.9500 - 63.7400 deg	Length limiting slit	: 10.0mm
Wavelength	: CuK α 1 / 1.540593 Å	2-Theta	: 62.9498 deg	Receiving slit # 1	: 1.000mm
Goniometer	: SmartLab(In-plane)	Omega	: 31.4749 deg	Filter	: None
Base Att. Config	: Std. chi cradle	Chi	: 0.000 deg	Receiving optical device	: Ge(220)x2
Attachment	: RxRy	Phi	: 0.000 deg	Receiving parallel slit	: Soller_slit_5.0deg
Detector	: SC-70	2-ThetaChi	: 0.0000 deg	Receiving slit # 2	: 1.000mm
Memo	: None			Attenuator	: Open
Optics Attribute	: Ultra high res. parallel beam Ge(220)x4/RS			Diffracted beam mono.	: None
				Monochromator slit	: None





Concluzii

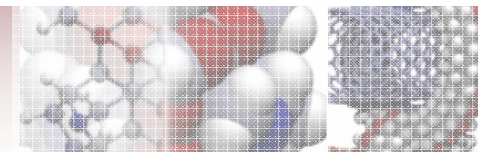


1. Metodele metrologice care folosesc difractia radiatiilor X

- sunt aplicabile unui spectru larg de materiale, procese si tehnologii,
- au un grad inalt de precizie si reproductibilitate
- se preteaza integrarii in linia de productie (fab-line, de ex. In industria semiconductorilor, farmaceutica, metalurgica,etc),
- necesita alegerea judicioasa a metodei de masura, a conditiilor experimentale si a metodelor (software) de prelucrare ulterioara si extragerea parametrilor fizici de interes.

2. Metoda metrologica de determinare a grosimii filmelor subtiri prin reflexia radiatiilor X - XRR are avantajul ca:

- Permite analiza filmelor subtiri cu grosimi de la 1nm (in unele cazuri de 0.1 nm) la cateva mii de nm
- Permite analiza rugozitatii si largimii interfetei (atribuita rugozitatii topografice si interdifuziei)
- Permite determinarea directa a densitatii filmului atunci cand compozitia e cunoscuta
- Permite controlul nedistructiv
- Este aplicabila unui domeniu larg de materiale, de la semiconductori, materiale magnetice, polimeri, indiferent de forma de cristalizare/starea de agregare.



Bibliografie

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2. **“1100nm InGaAs/(Al)GaAs quantum dot lasers for high-power applications ”**, E-M Pavelescu, C Gilfert, P Weinmann, **M Danila**, **A Dinescu**, M Jacob, M Kamp and J-P Reithmaier, **J. Phys. D: Appl. Phys. 44 (2011) 145104 (4pp)**, doi:10.1088/0022-3727/44/14/145104, **EU project WWW.BRIGHTER:EU, financial support offered by the EU project MIMOMEMS (Ref. Nr. 202897)**
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