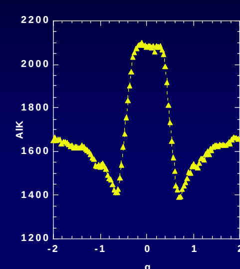
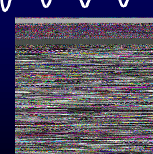
High Angular Resolution
Electron Channeling X-ray Spectroscopy
(HARECXS)
Measurements of Electron Channeling Effects
on Quantitative AEM/XEDS in Ordered Systems

Electron Channeling Induced X-ray Emission

- Original Observations of Effect
  - Duncumb ‘62, Hall ‘66, Cherns etal ‘73
- Predicted Applications
  - Cowley ‘64, ‘70
- ACHEMI Technique -
  - Tafto ‘79, Spence & Tafto ‘83
- Multi-Variate Statistical Analysis -
  - Rossouw etal , Anderson and others
    late80’-90’s





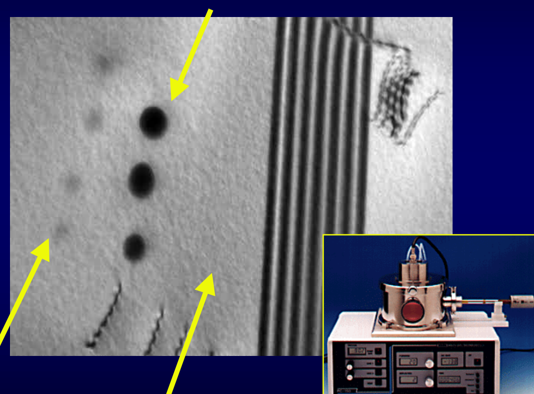
Experimental Equipment

Philips EM 420T

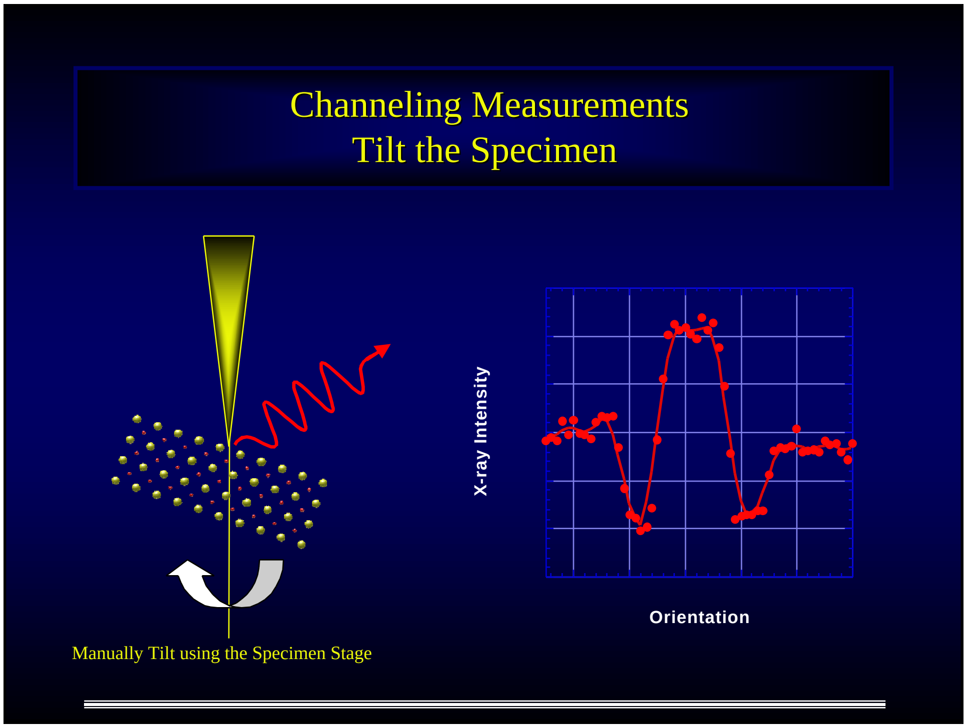
- 120kV, LaB6
- EDAX 9900
  + HomeBrew Software
- Super UTW Detector

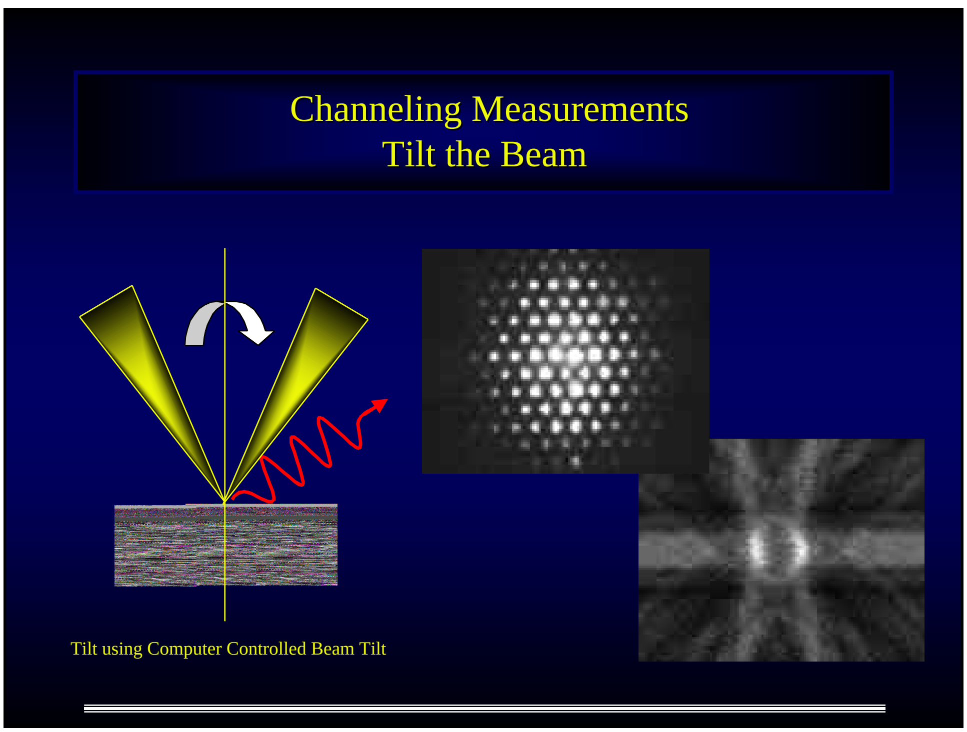
Specimen Preparation

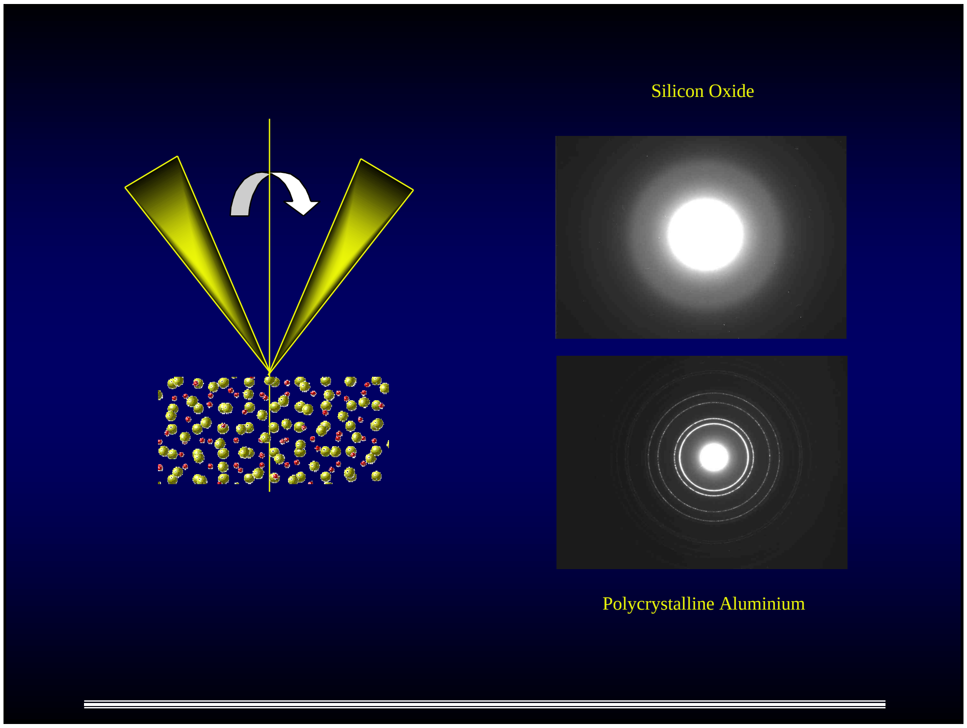
- Untreated Specimen

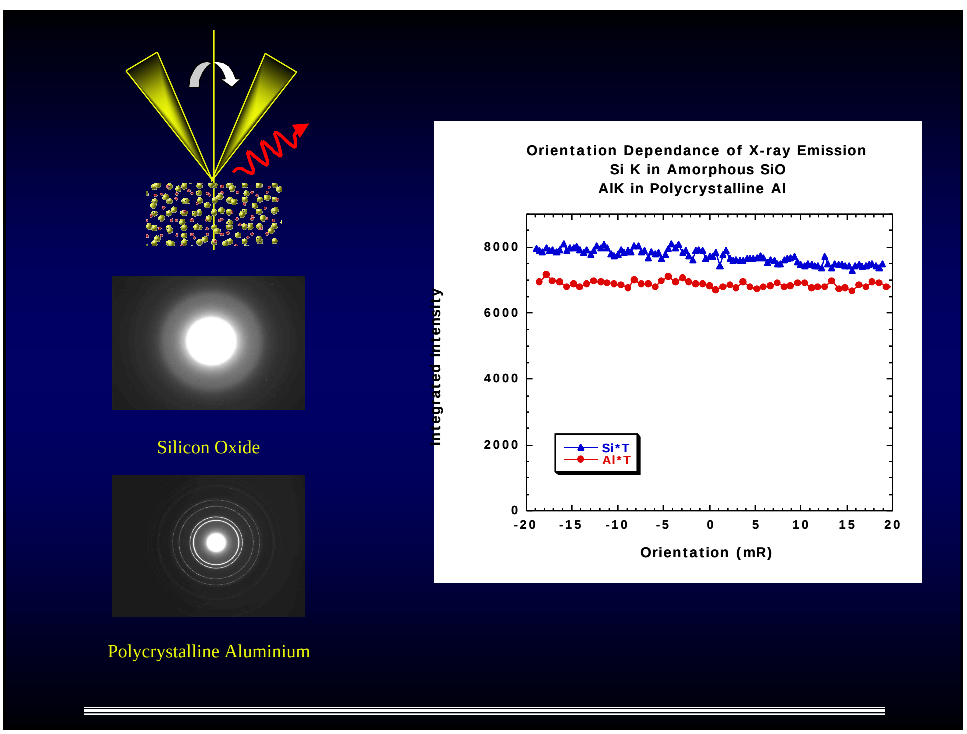


- After 5 minutes Argon Processing
- After 5 minutes of additional Oxygen Processing

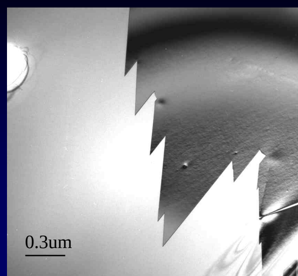

Channeling Measurements
Tilt the Specimen
X-ray Intensity
Orientation
Manually Tilt using the Specimen Stage

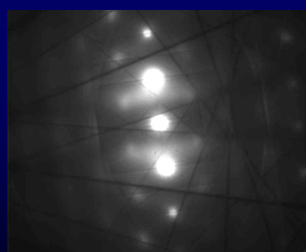

Channeling Measurements
Tilt the Beam
Tilt using Computer Controlled Beam Tilt


Silicon Oxide
Polycrystalline Aluminium

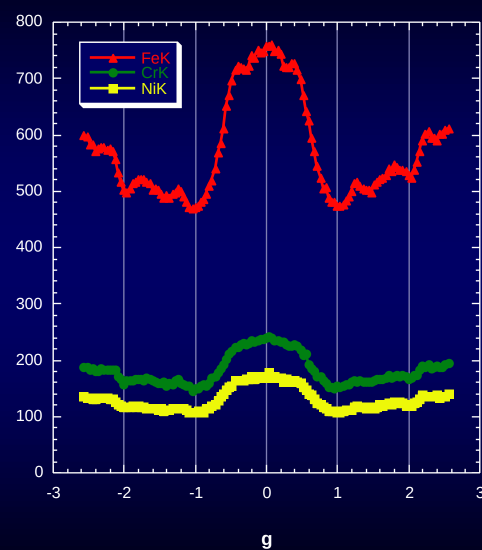

Silicon Oxide
Polycrystalline Aluminium
Orientation Dependance of X-ray Emission
Si K in Amorphous SiO
AlK in Polycrystalline Al
8000
6000
4000
2000
0
-20
-15
-10
-5
0
5
10
15
20
Orientation (mR)
Si*T
Al*T

Orientation Dependance in Homogeneous Alloys

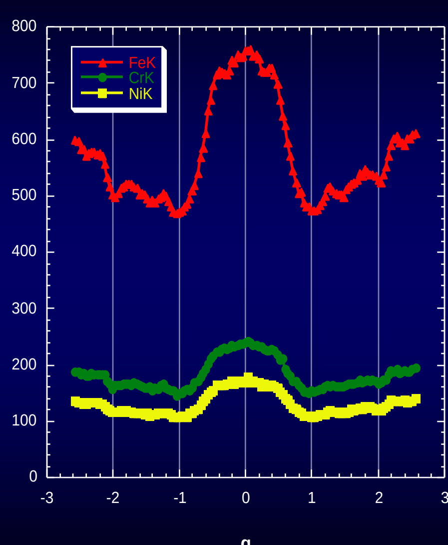


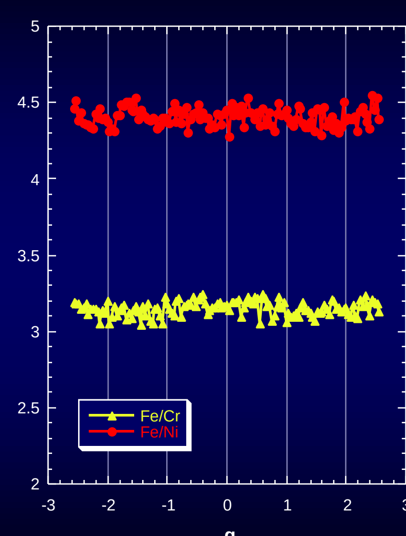


316 SS

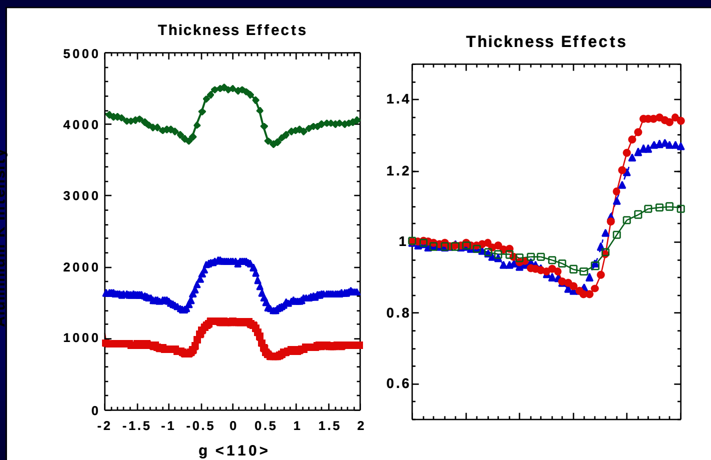


Orientation Dependance in Homogeneous Alloys



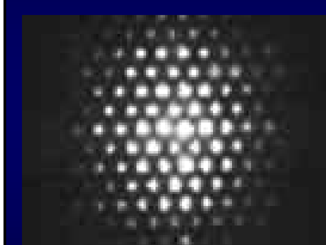


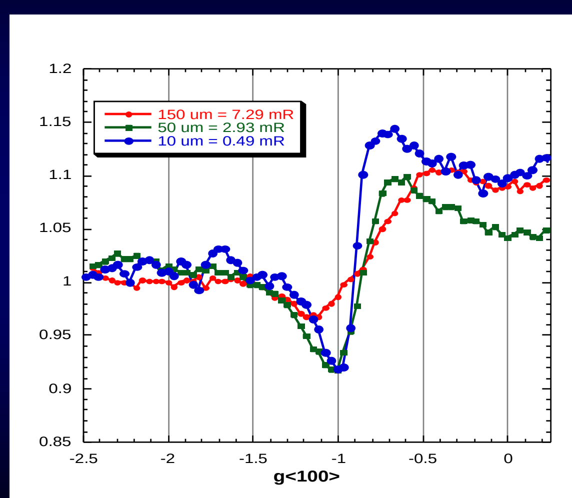
Thickness Dependance in Homogeneous Specimen



Aluminium Single Crystal along <110>

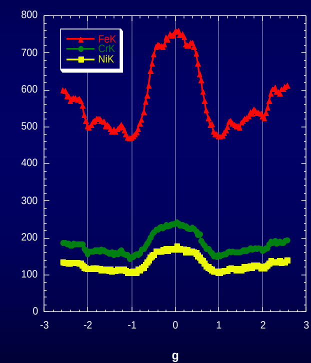
Angular Resolution Effects

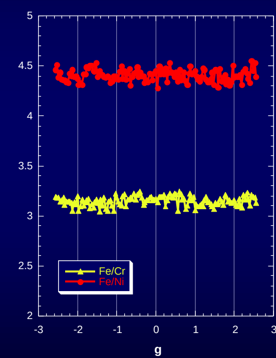


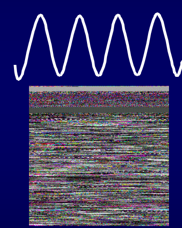


Aluminium <100>

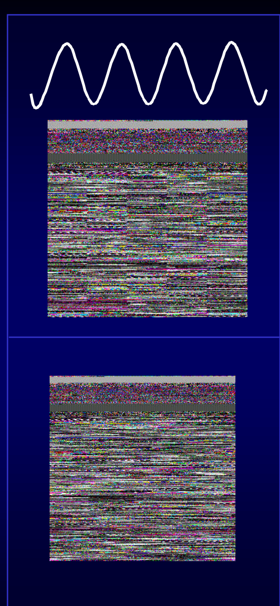
Orientation Dependance in Homogeneous Alloys

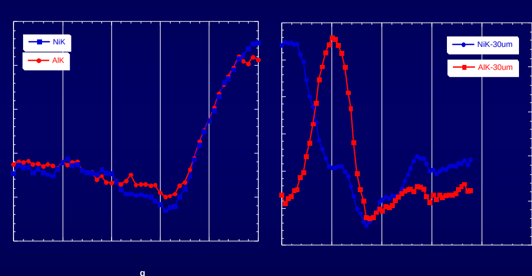




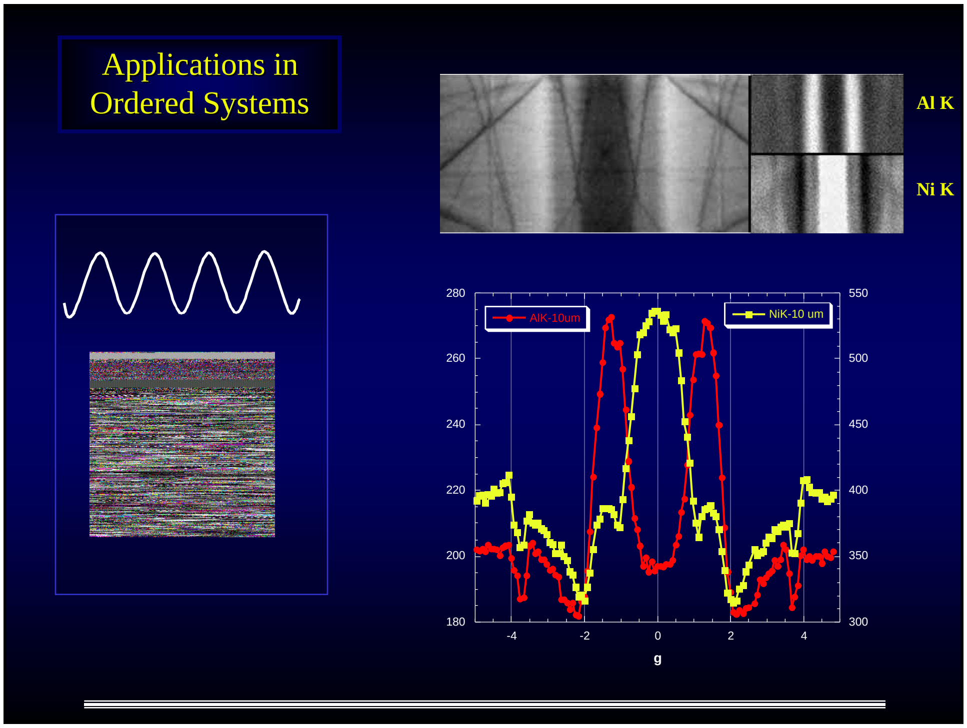


Applications in Ordered Systems

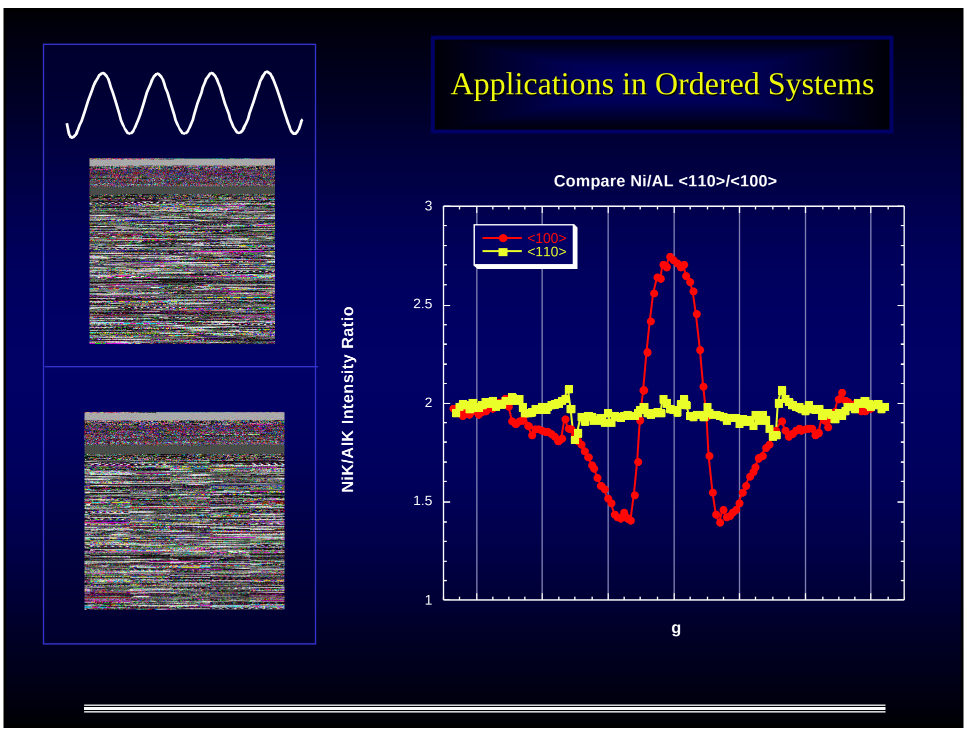




g = <110>   g = <100>

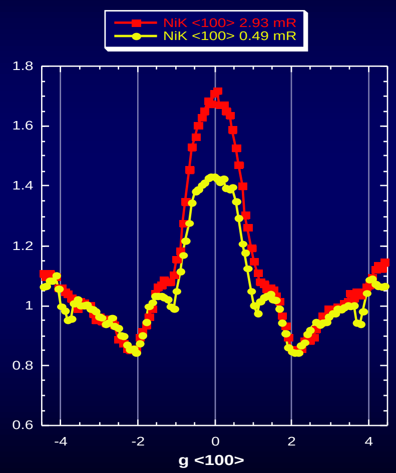


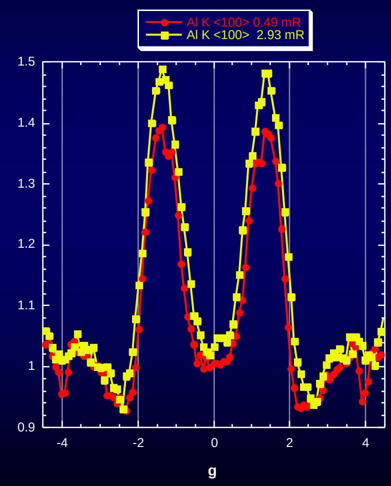
Applications in Ordered Systems
Al K
Ni K
AlK-10um
NiK-10 um
280
260
240
220
200
180
550
500
450
400
350
300
-4
-2
0
2
4
g



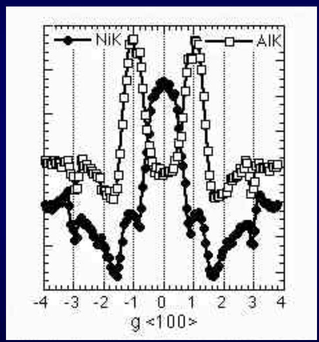
Applications in Ordered Systems
Compare Ni/AL <110>/<100>
<100>
<110>
NiK/AlK Intensity Ratio
3
2.5
2
1.5
1
g

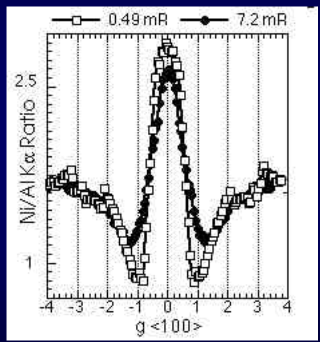
Convergence Angle Effects on the Profiles





Convergence Angle Effects on the Ratio's





Applications

Takeshi Soeda
Kyushu University, Fukuoka Japan

Al_2O_3 and $MgAl_2O_4$ are candidates for rf Windows on ITER (International Thermonuclear Exp. Reactor)

Expected to maintain structural and electrical stability from 20 -900 K to ~ 1 dpa. Required swelling must be < 5%.

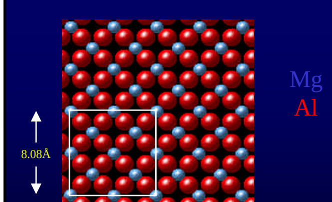
$MgAl_2O_4$ exhibit very low swelling 0.07% for ~ 200 dpa under neutron irradiation. Possibly due to the large number of structural vacancies.

Question: What is the mechanism which causes this? How can it be quantitatively measured?

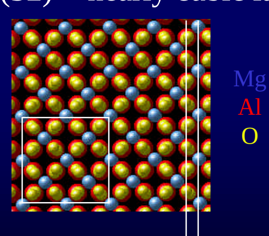
$MgAl_2O_4$ - Spinel

Space Group O_h^7 (Fd3m)

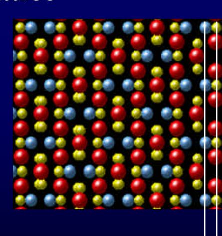
Mg (8) - 64 tetrahedral sites
Al (16) - 32 octahedral sites
O (32) - nearly cubic lattice



{100}
not showing Oxygen

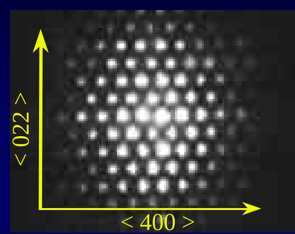


{100}
showing Oxygen

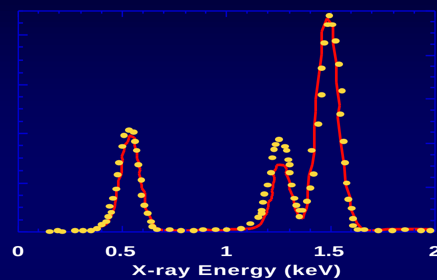


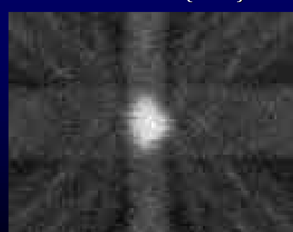
{110}

Zone Axis Orientations

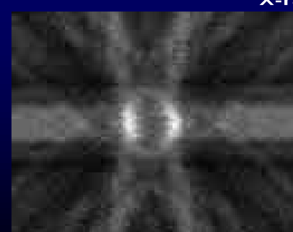


{011}





O K



Mg K

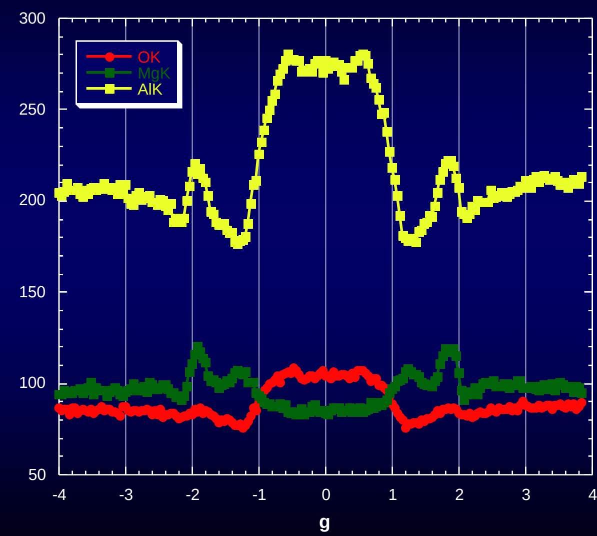
Al K

Two dimensional data acquistion allows one to identify dynamical regions immediately

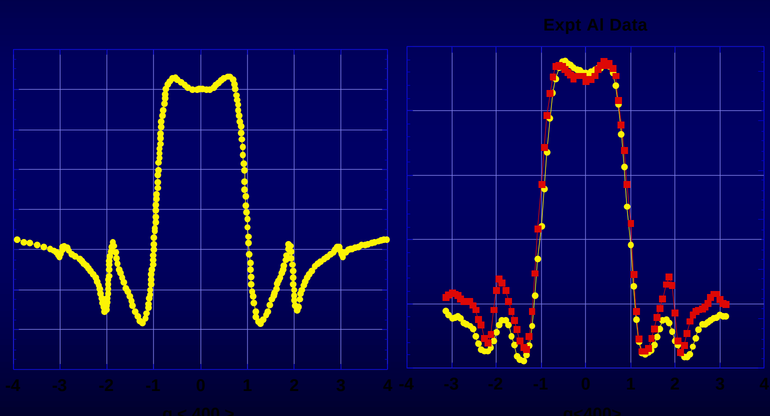
SAD { 013} - {012}

XEDS 2D Channeling Map { 013} - {012}

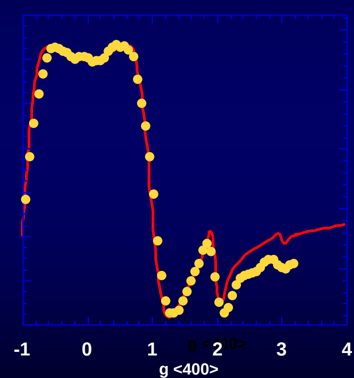
High Angular Resolution Measurements



Perfect Crystal Results

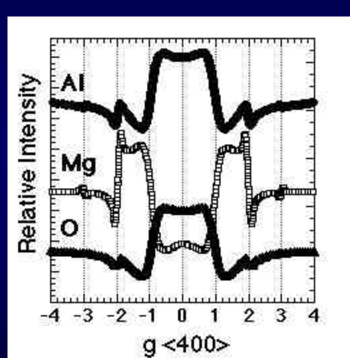


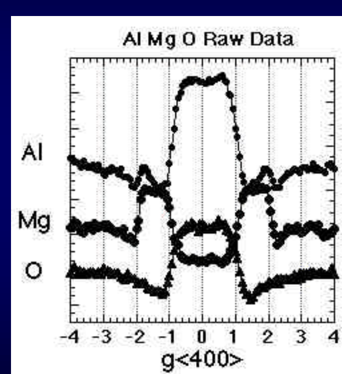
Comparison Expt. - Theory

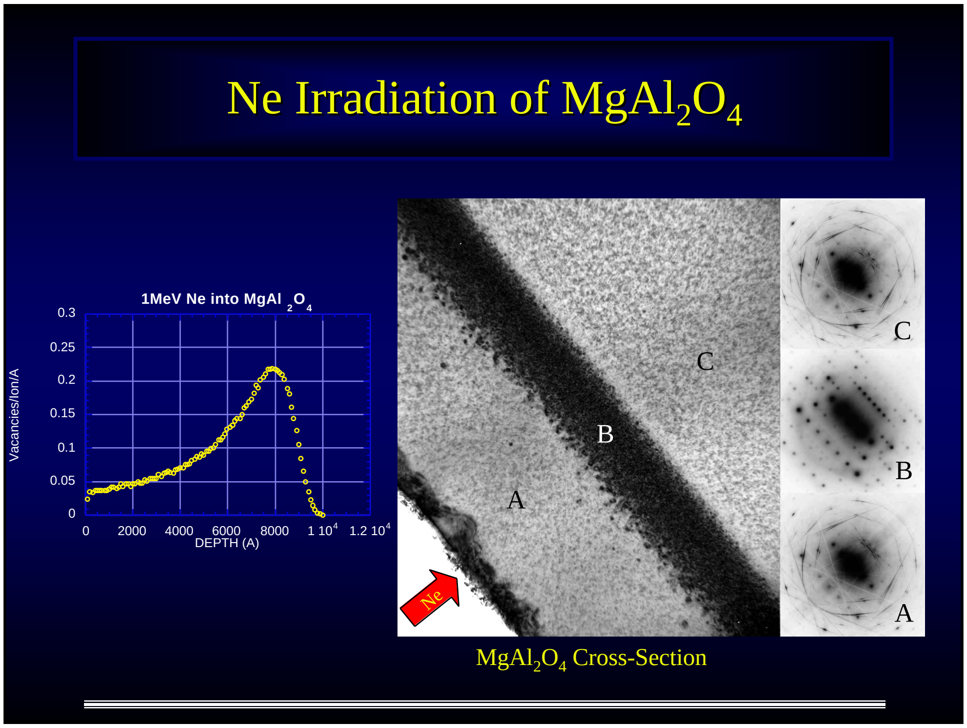


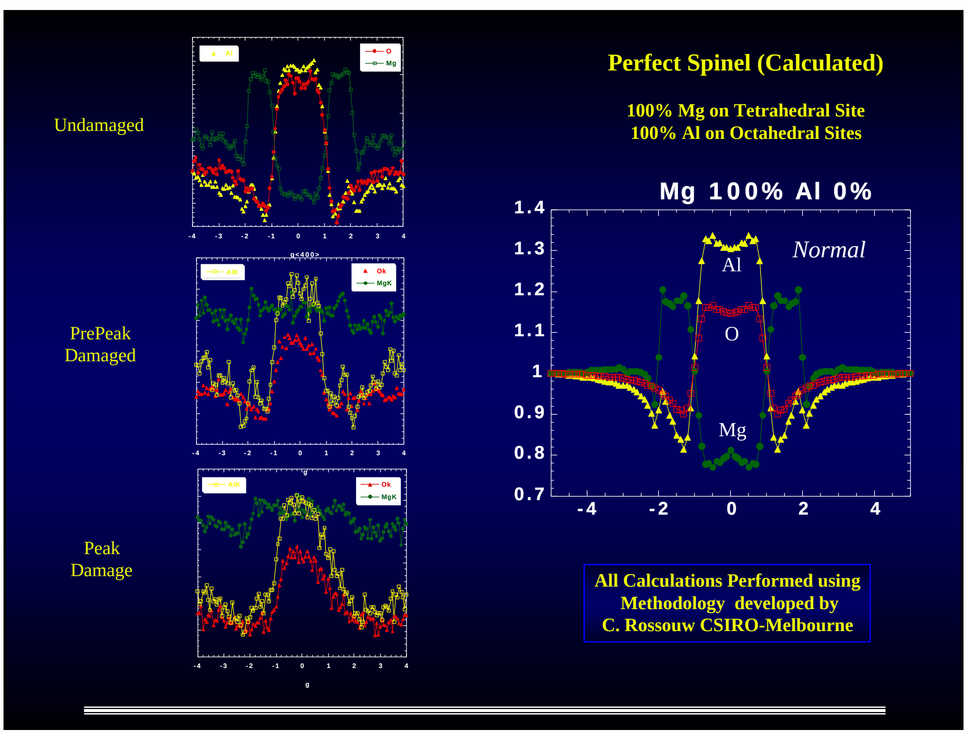
Experimental
Theoretical

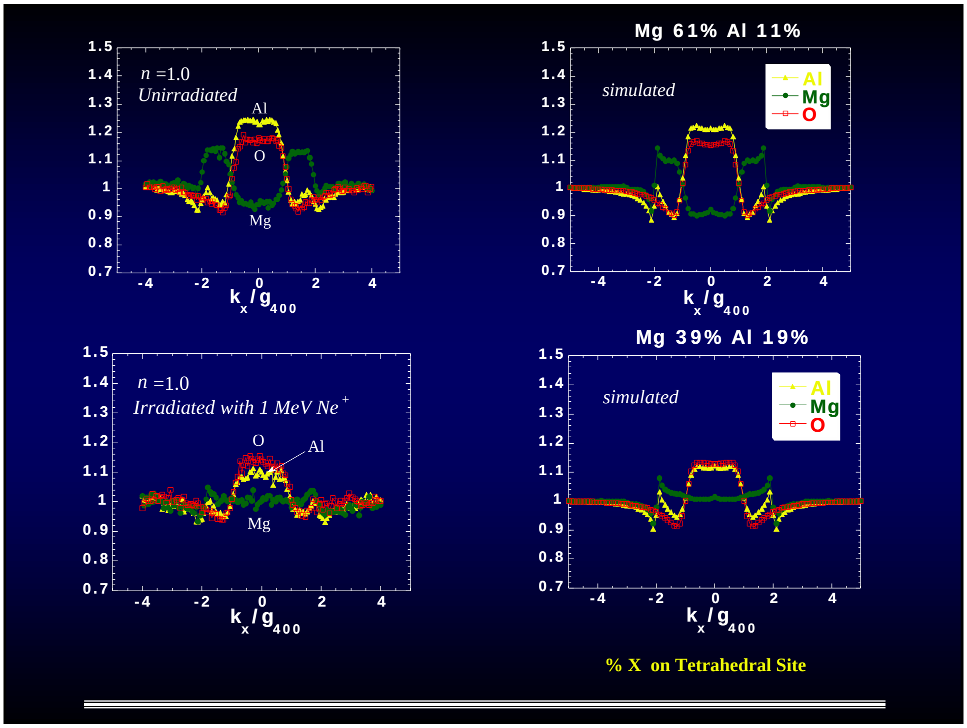
Comparison under Typical ACHEMI Conditions






Ne Irradiation of MgAl$_2$O$_4$
1MeV Ne into MgAl$_2$O$_4$
Vacancies/Ion/A
DEPTH (A)
0.3
0.25
0.2
0.15
0.1
0.05
0
0 2000 4000 6000 8000 1 10^4 1.2 10^4
Ne
A
B
C
MgAl$_2$O$_4$ Cross-Section


Undamaged
PrePeak
Damaged
Peak
Damage
Perfect Spinel (Calculated)
100% Mg on Tetrahedral Site
100% Al on Octahedral Sites
Mg 100% Al 0%
Normal
Al
O
Mg
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
-4 -2 0 2 4
All Calculations Performed using
Methodology developed by
C. Rossouw CSIRO-Melbourne


n =1.0
Unirradiated
Al
O
Mg
k x / g 400
Mg 61% Al 11%
simulated
Al
Mg
O
k x / g 400
n =1.0
Irradiated with 1 MeV Ne +
O
Al
Mg
k x / g 400
Mg 39% Al 19%
simulated
Al
Mg
O
k x / g 400
% X on Tetrahedral Site

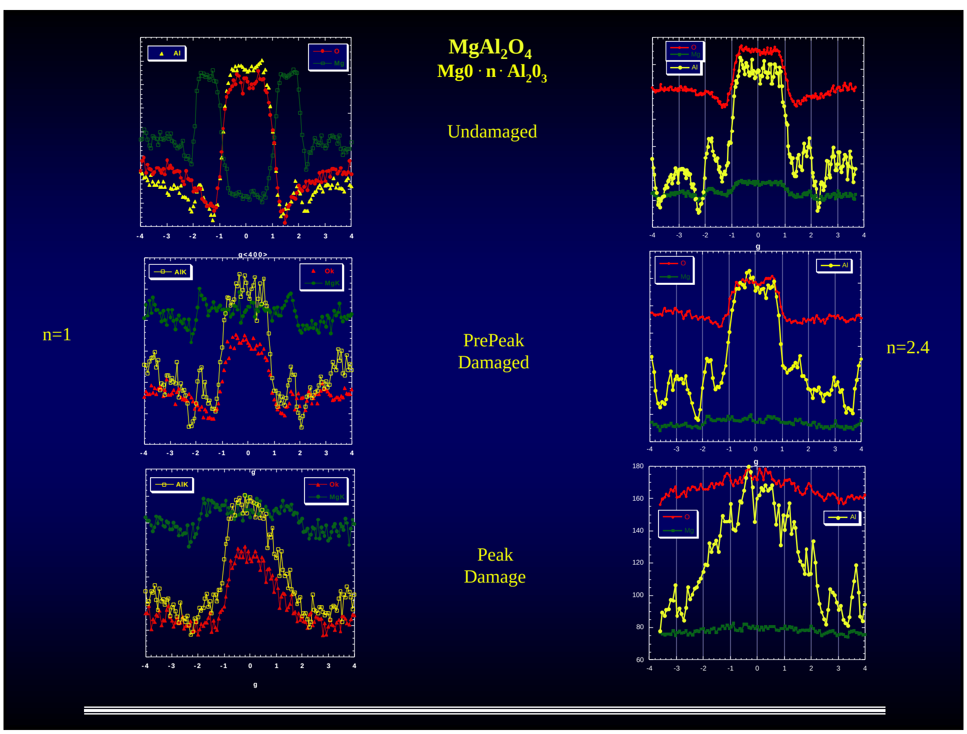

MgAl2O4
Mg0 · n · Al203
Undamaged
n=1
PrePeak
Damaged
n=2.4
Peak
Damage

Table 1: Occupation numbers of Mg^{2+}, Al^{3+} and vacancies on the IV-sites and the VIII-sites in a unit cell in annealed MgO·*n*Al_2O_3. The occupation probabilities on the IV-sites (P_i) are also tabulated.

		Number of ions			P_i
		total	IV-sites	VIII-sites	
n=1.0	Mg^{2+}	8	4.9 ± 0.1	3.1 ± 0.1	0.61 ± 0.01
	Al^{3+}	16	1.6 ± 0.3	14.4 ± 0.3	0.10 ± 0.02
	Vacancy	-	1.5 ± 0.3	0	-
n=2.4	Mg^{2+}	3.9	1.1 ± 0.1	2.8 ± 0.1	0.27 ± 0.01
	Al^{3+}	18.7	3.8 ± 0.3	14.9 ±0.3	0.20 ± 0.02
	Vacancy	1.3	3.2 ± 0.4	0	-

Table 2: Cation and vacancy configuration on the IV-sites and the VIII-sites for *n*=1 after irradiation with 1 MeV Ne^+ ions at 870 K up to 2 dpa in the pre-peak damaged area. The probabilities are given in terms of the number of ions per unit cell.

		Number of ions		
		total	IV-sites	VIII-sites
Pre-peak damaged area	Mg^{2+}	8	3.0 ± 0.1	5.0 ± 0.1
	Al^{3+}	16	3.5 ± 0.3	12.5 ± 0.3
	Vacancy	-	1.5 ± 0.3	0
Peak damaged area	Mg^{2+}	8	3.0 ± 0.1	5.0 ± 0.1
	Al^{3+}	16	2.9 ± 0.3	13.1 ± 0.3
	Vacancy	-	2.2 ± 0.3	0

Summary

•HARECXS (HR-ALCHEMI?) yields a wealth of information about ordering…

•WRT Quantitative XEDS in the AEM analysis this effect is much stronger than we have long claimed/thought it was, and can cause a lot of problems if you are not very careful

•HARECXS looks to become a good technique for characterization of structural recombination due to irradiation effects in ordered systems

•Irradiation causes dramtic changes in the HARECXS Data Sets

•Elemental redistribution is easy to see

•Quantification of what is happening is complicated and work is still in progress