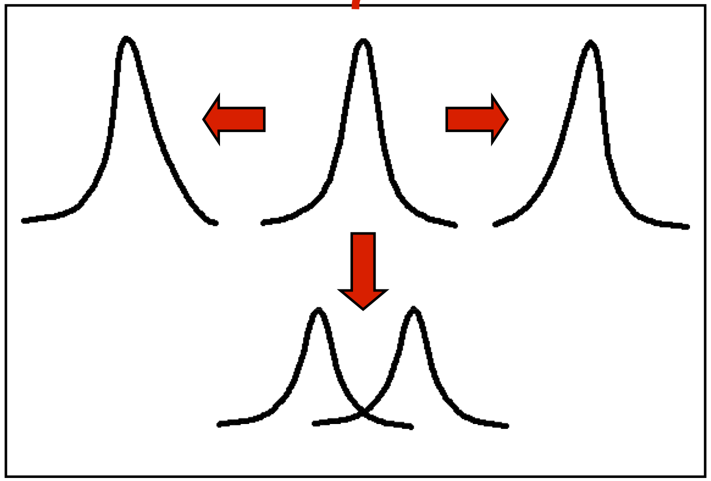
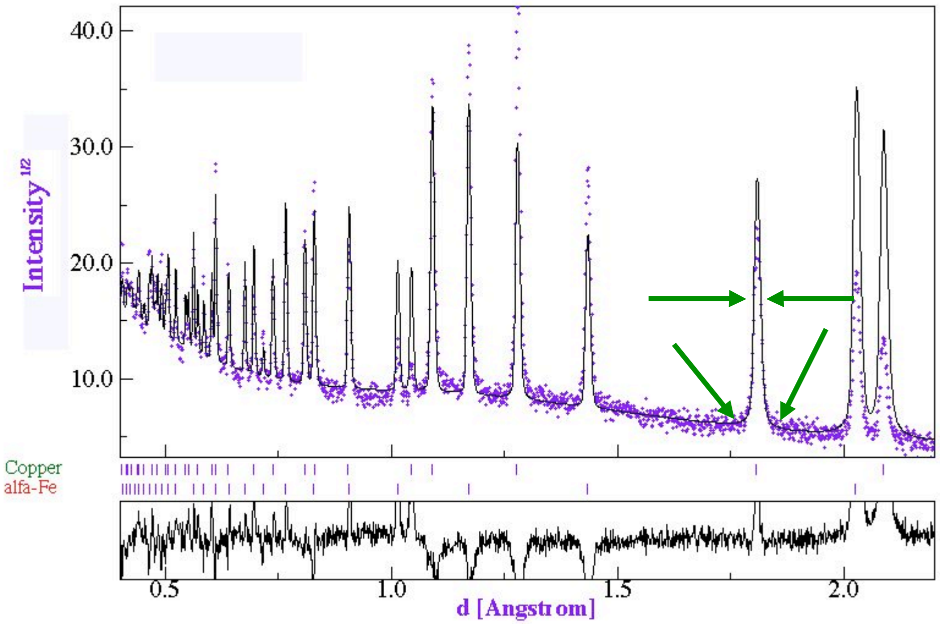
Informations: microstructure

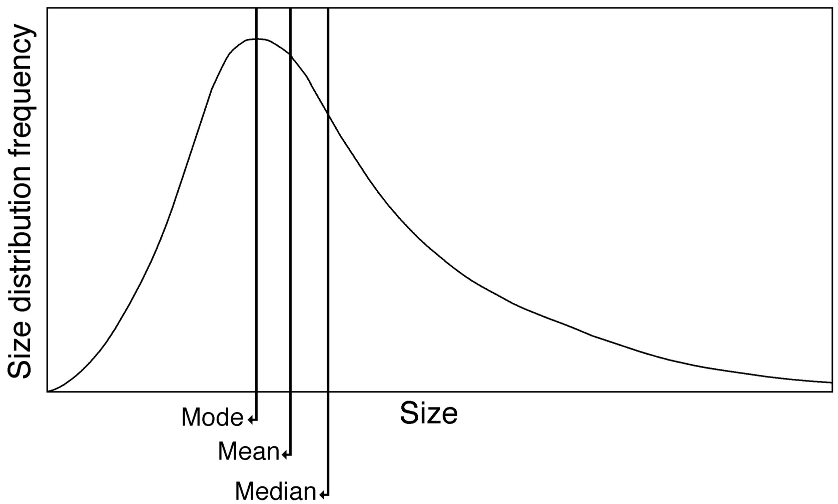
- Crystallite sizes, anisotropic, distribution
- Microstrain (III kind), distribution, dislocation or point defects density
- Antiphase domains (intermetallics…)
- Stacking and deformation faults probability, intrinsic, extrinsic





Crystallites distribution

Name	Definition
Mean length	$\langle M \rangle = \frac{\sum_{i=1}^{N} n_i d_i}{\sum_{i=1}^{N} n_i}$
Length weighted or area-length	$\langle D \rangle = \frac{\sum_{i=1}^{N} n_i d_i^2}{\sum_{i=1}^{N} n_i d_i}$
Area weighted or volume-area	$\langle D_a \rangle = \frac{\sum_{i=1}^{N} n_i d_i^3}{\sum_{i=1}^{N} n_i d_i^2}$
Volume-weighted	$\langle D_V \rangle = \frac{\sum_{i=1}^{N} n_i d_i^4}{\sum_{i=1}^{N} n_i d_i^3}$



Line broadening methods

- Are based on profile analysis:
  - Crystallites and microstrain have a different broadening behavior in 2theta
- Several methods available:
  - Scherrer equation (1918):

$$\langle D \rangle_v = \frac{K\lambda}{\beta_S(2\theta)\ \cos\theta}$$

  - Williamson-Hall plot
  - Warren-Averbach or Fourier analysis
  - ......
- Instrumental broadening must be subtracted
- Two reflections are needed to separate crystallite and microstrain broadening
- More reflections for anisotropic broadening
- Planar defects analysis was developed by Warren (see book)

The Warren-Averbach analysis

Warren treatment for a 00l peak:

$$S(2\theta) = \int S_S(2\theta - z) S_I(z)\, dz$$

(Stokes treatment: $A = A_S A_I$)

Fourier coefficients:

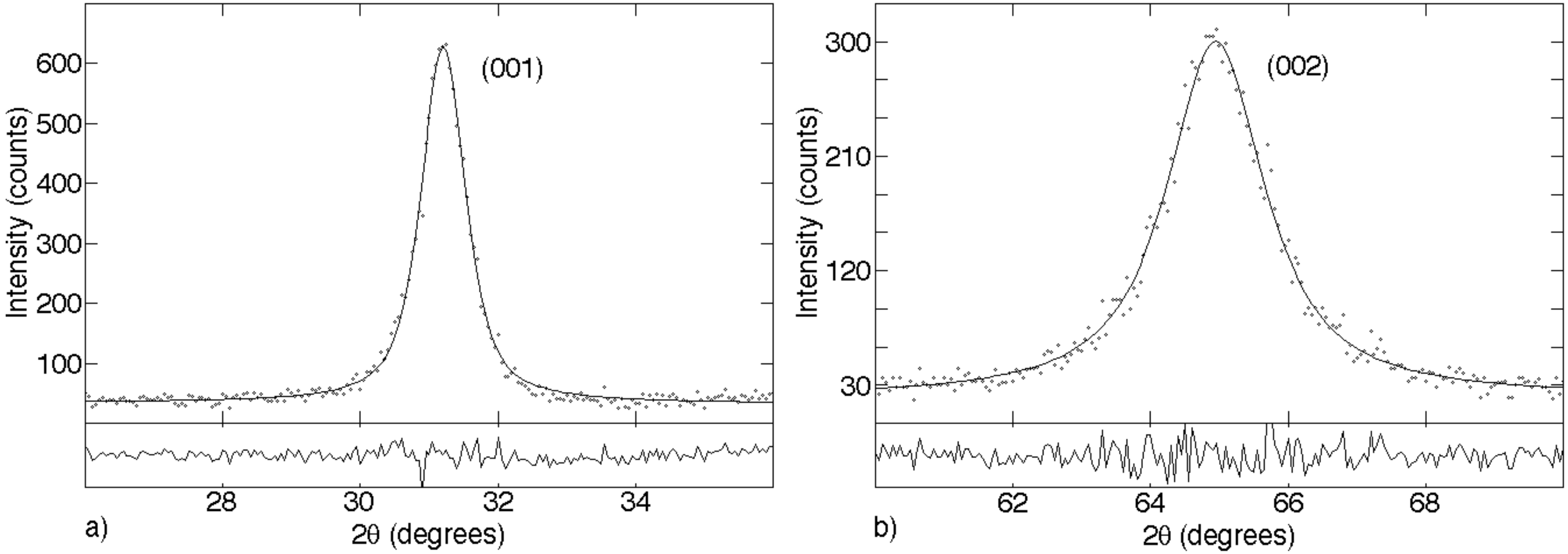
$$A_n = A_n^S A_n^D = \frac{N_n}{N_3} \langle \cos 2\pi l Z_n \rangle$$

$$A_n^S = \frac{N_n}{N_3} = \frac{1}{N_3} \sum_{i=|n|}^{\inf} (i - |n|) p(i)$$

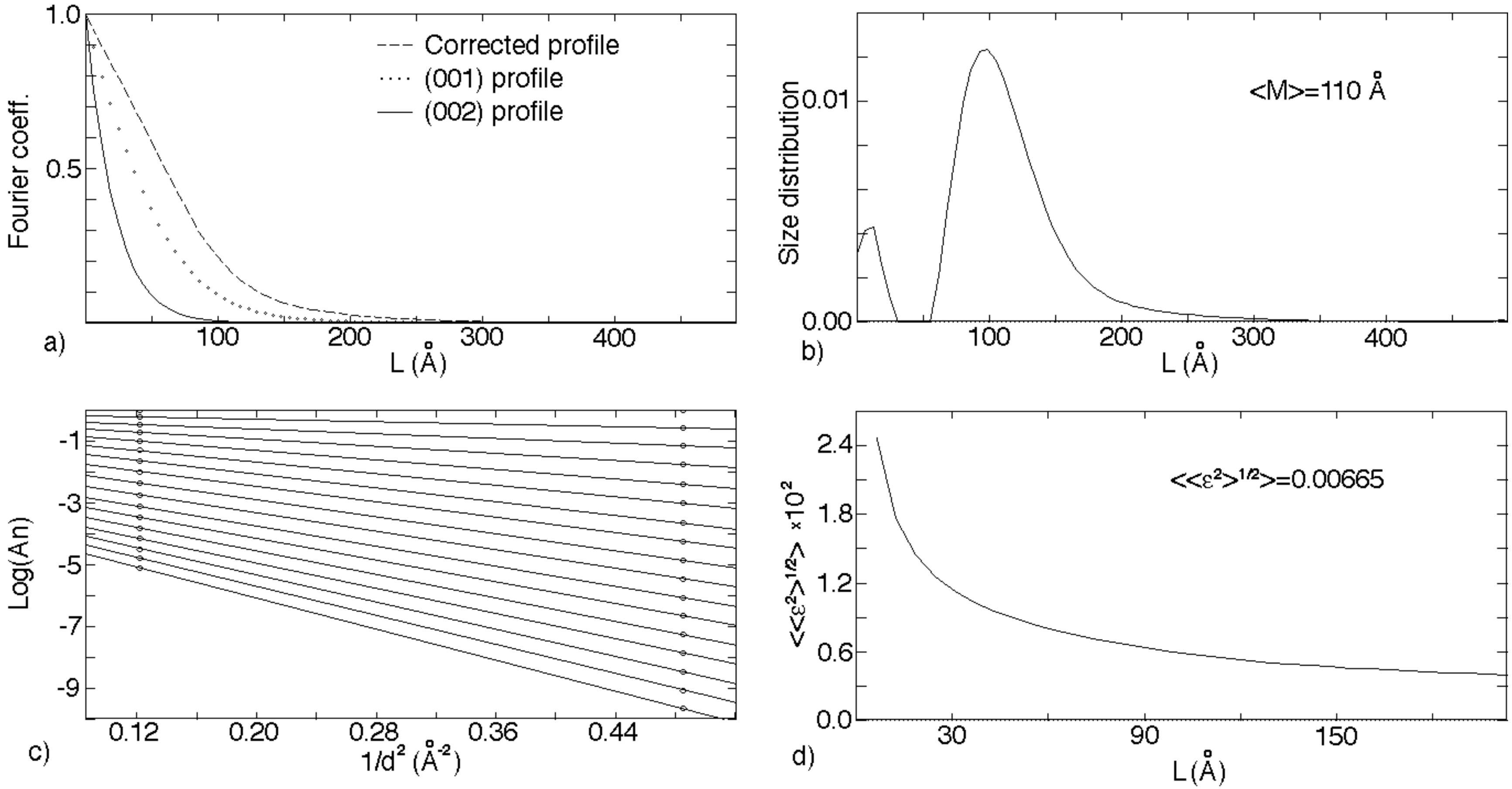
$$\left(\frac{dA_n^S}{dn} \right)_{n \to 0} = -\frac{1}{N_3}$$

Get the profiles for Fourier transform

We need two peaks of different order, same hkl direction

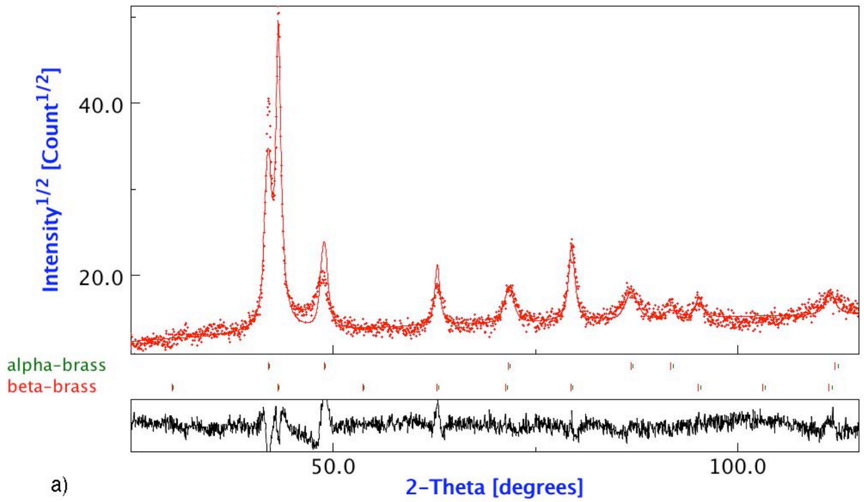


W-A continue.....



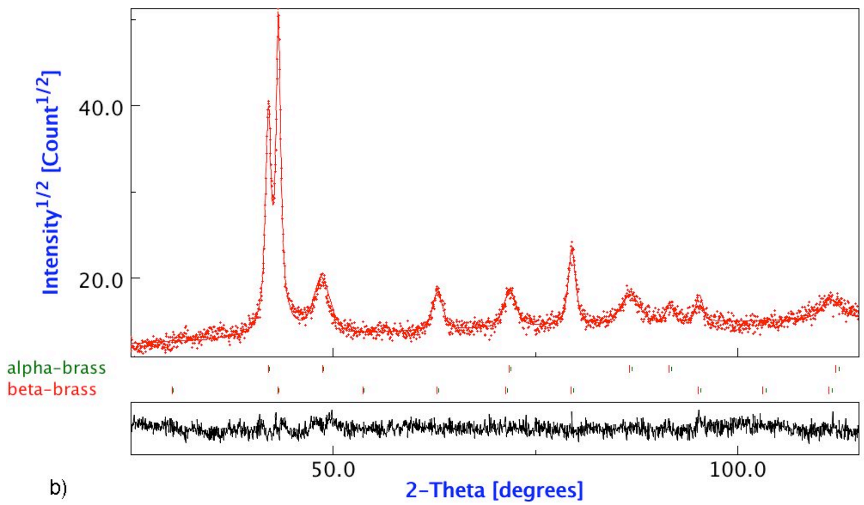
Fourier coeff.
1.0
0.5
Corrected profile
(001) profile
(002) profile
100
200
300
400
L (Å)
a)
Size distribution
0.01
0.00
<M>=110 Å
b)
Log(An)
-1
-3
-5
-7
-9
0.12
0.20
0.28
0.36
0.44
1/d² (Å⁻²)
c)
<<ε²>^1/2> ×10²
2.4
1.8
1.2
0.6
0.0
<<ε²>^1/2>=0.00665
30
90
150
d)

Planar faults



a)

No faults assumed



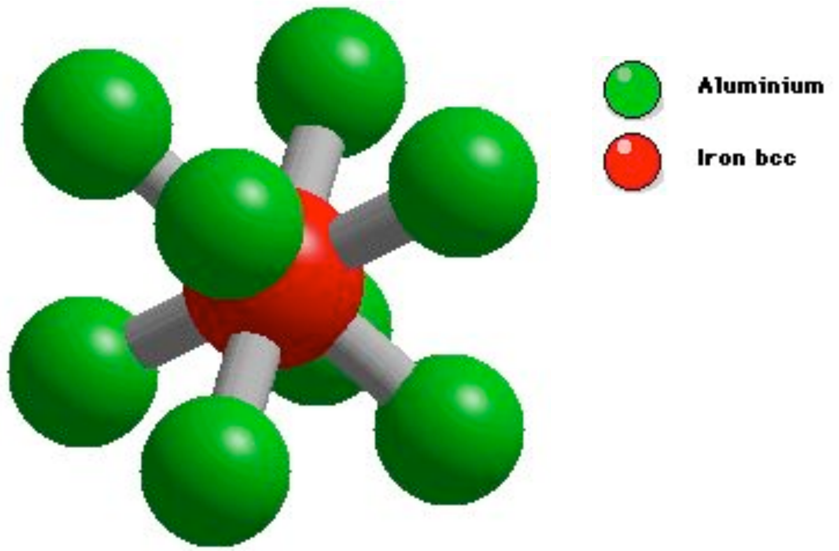
b)

Stacking and deformation faults

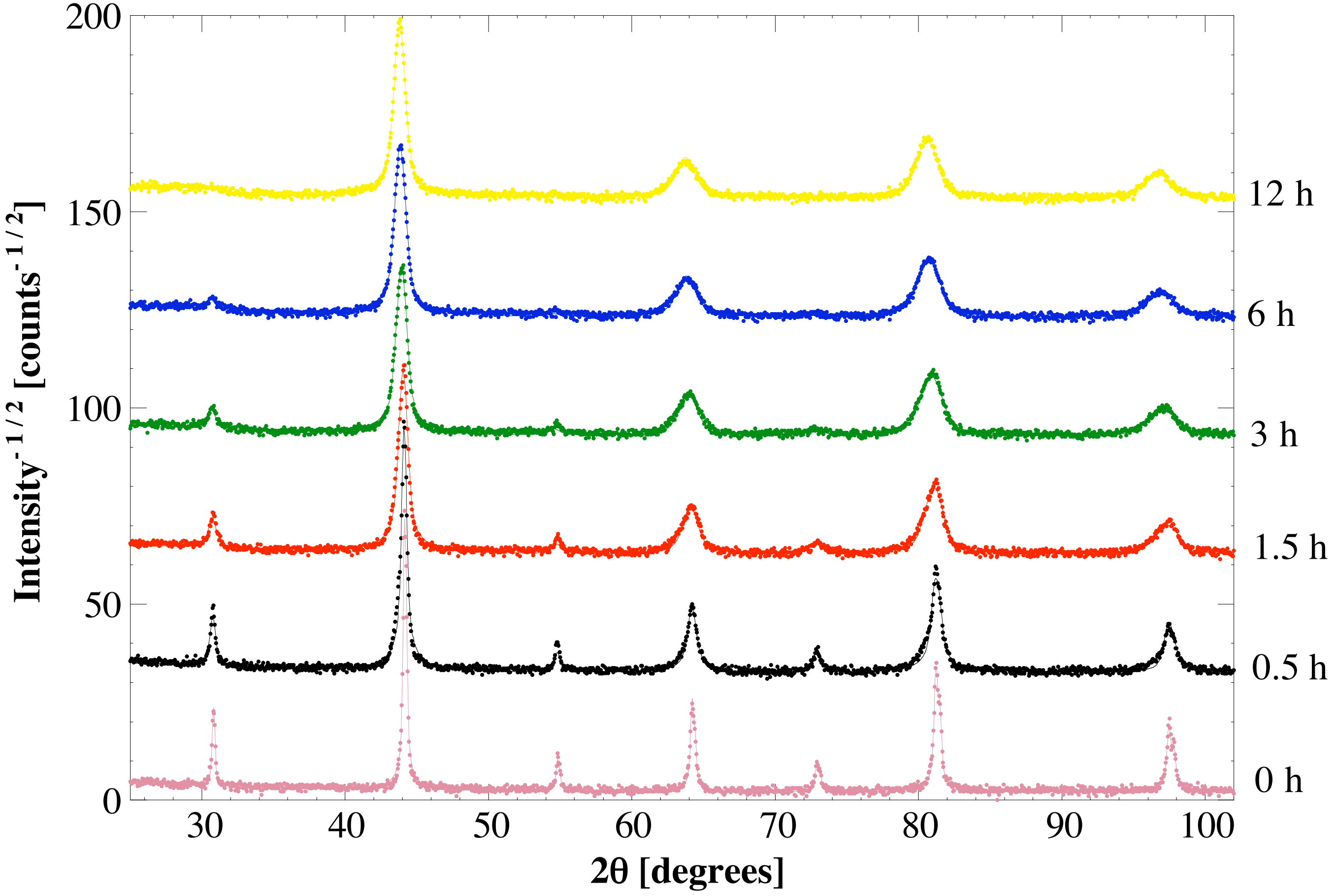
Order-disorder transition in intermetallic

- **Applications:**
  - High temperature structural material
  - Oxidation resistance
  - Blade material for Gas turbine
- **Main problem:**
  - Ductility (forming and shaping)
  - Brittle at low temperature
- **Disordered phase is more ductile**

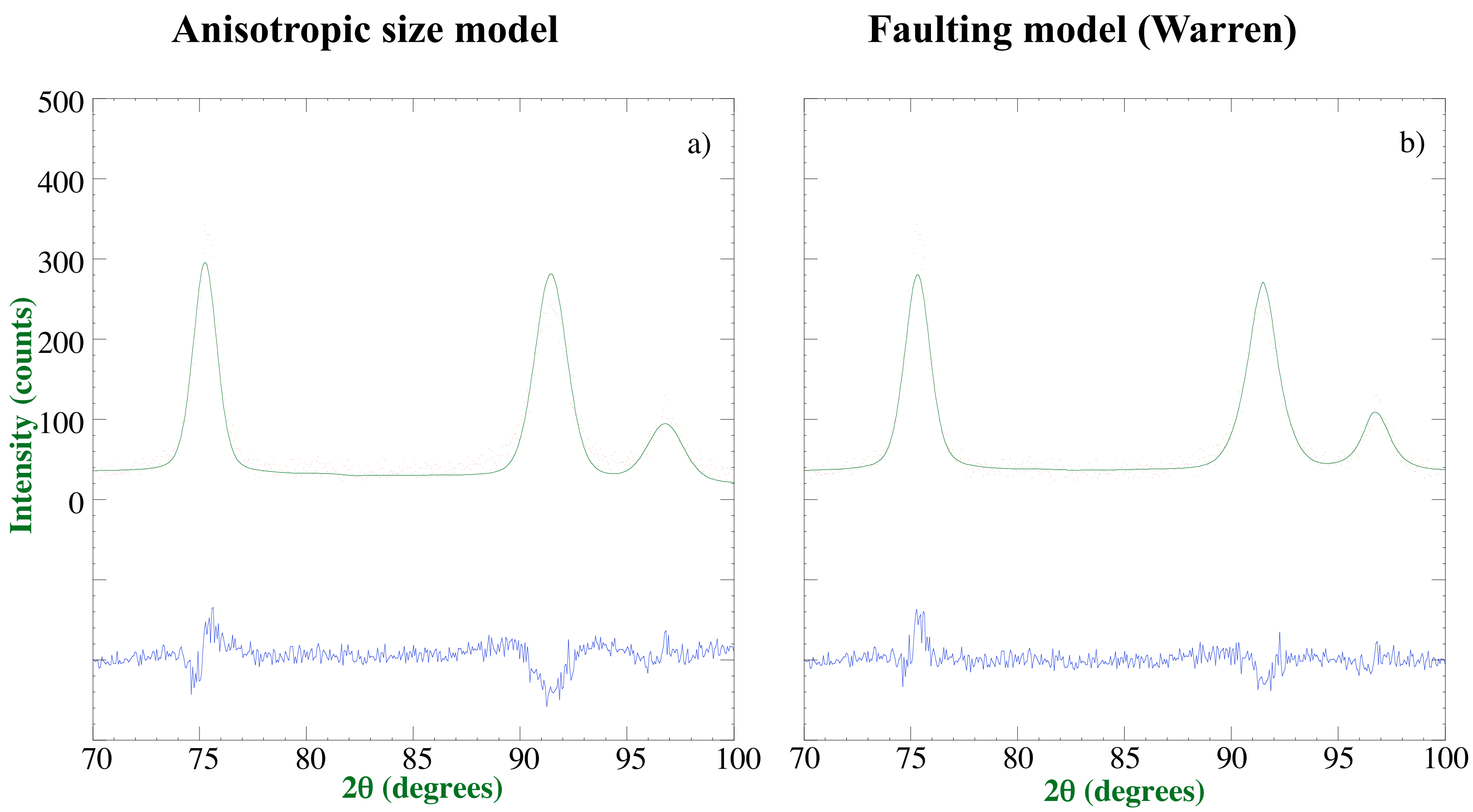
- **Materials:**
  - FeAl, Ni_3Al, NiAl, TiAl...



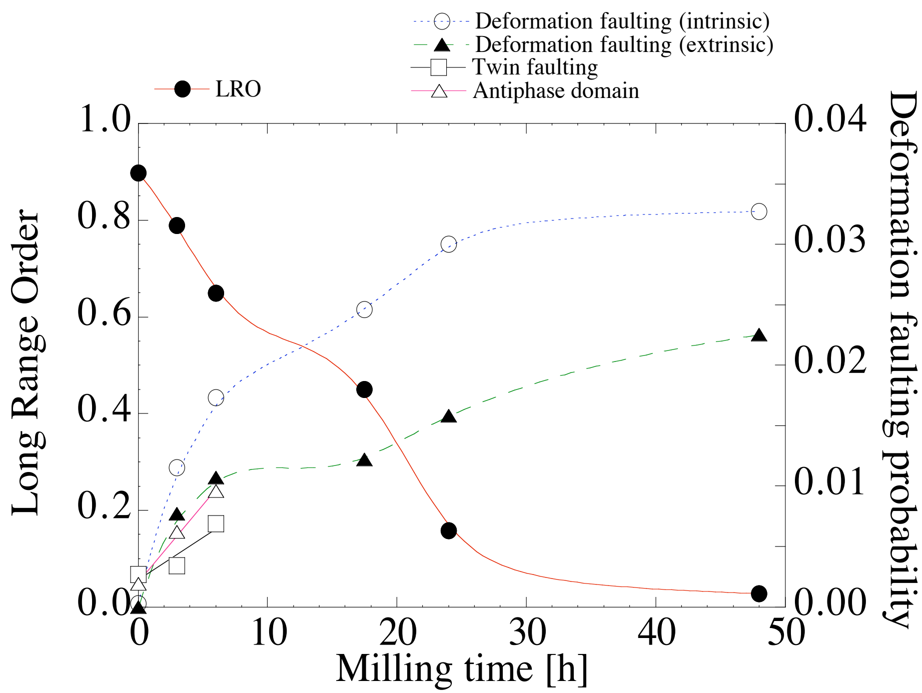
Disordering FeAl by ball-milling

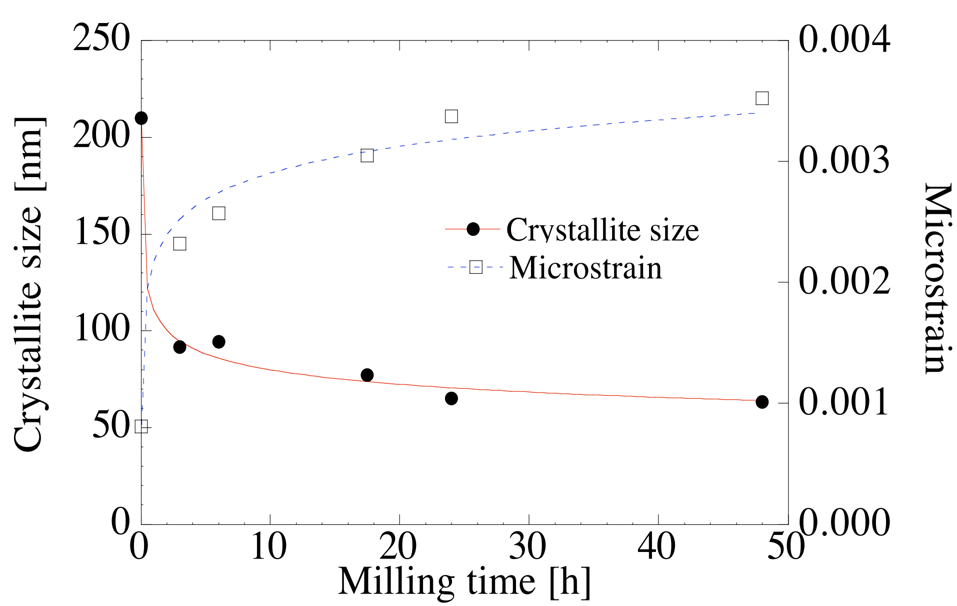


Faults effect

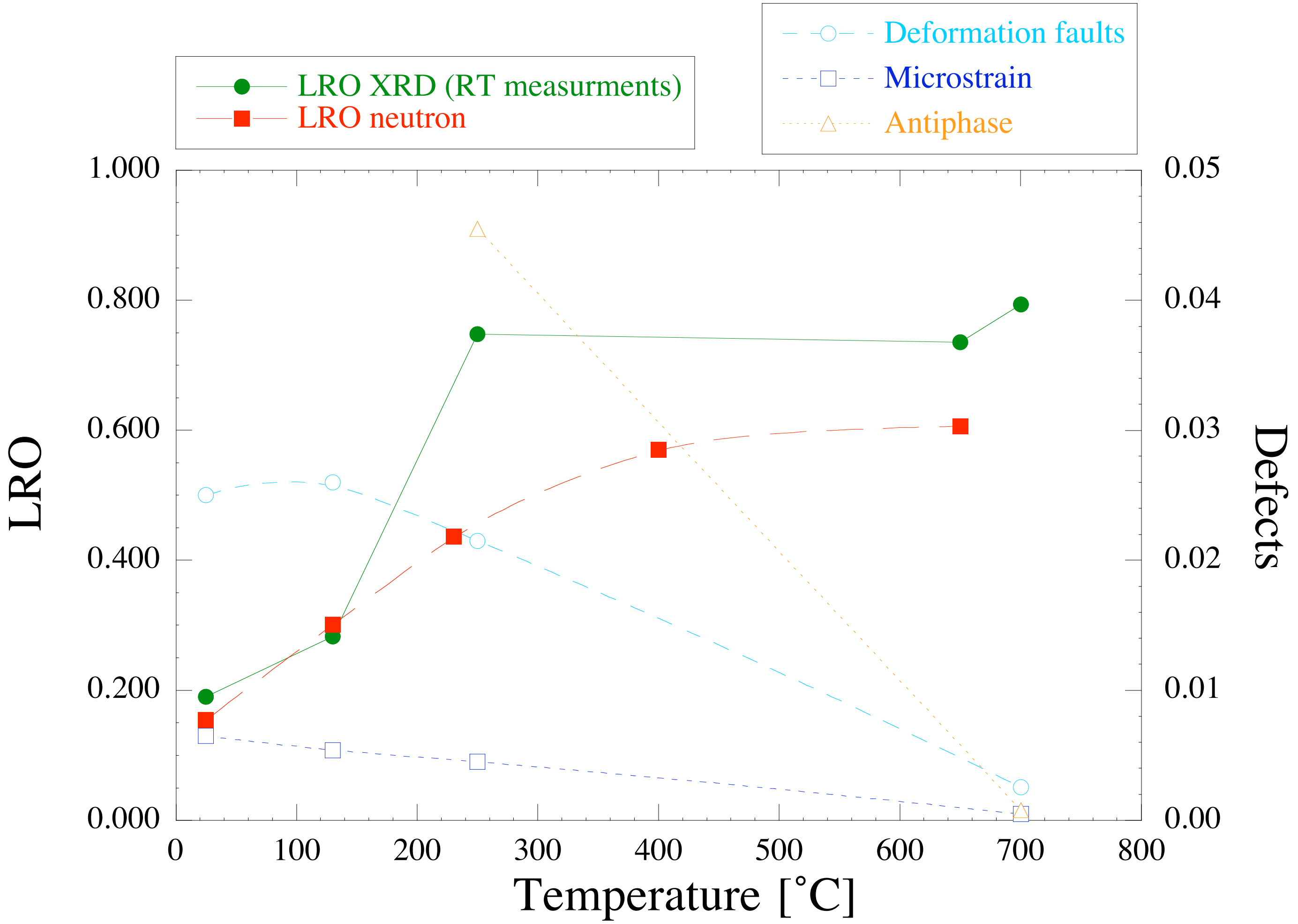


Defect analysis on ball-milled FeAl





FeAl annealing (neutron-XRD meas.)



Quantitative phase analysis

$$I_j^k = I \frac{f_j}{V_j^2} L_k \left|F_{k,j}\right|^2 P_{k,j} A_j$$

- **Methods:**
  - **Polymorph method**
  - **RIR**
  - **Rietveld**
  - **......**

$$\frac{f_1}{f_2} = \frac{I_1^k}{I_2^s} \frac{V_1^2 L_s \left|F_{s,2}\right|^2}{V_2^2 L_k \left|F_{k,1}\right|^2}$$

- **Crystallinity determination**
  - **An external or internal standard is needed**
  - **Traditional methods are based on the change in intensity of the crystallized fraction adding a crystalline standard**
  - **Method without standard: Rietveld + pseudo amorphous model (Le Bail)**

Cermet: Ni-Al_2O_3

- Alumina is quite inexpensive as high temperature structural material
- Lack of toughness

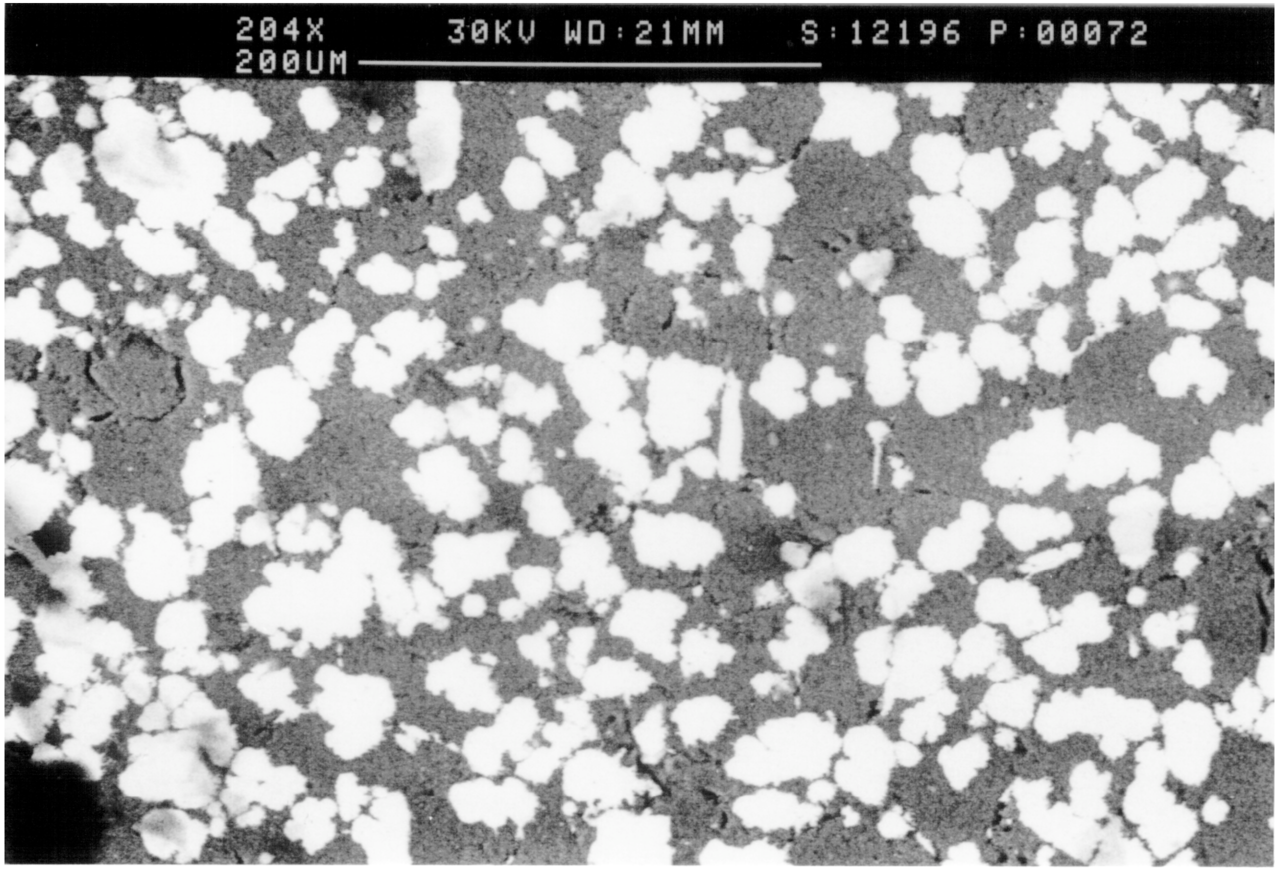
- Ductile metal inclusions
- Ni, Ag, FeAl, NiAl….
- Nickel is very ductile (relatively low temperature applications)

Cermet production (Ni-Al_2O_3)

- **Powder metallurgy**
  - Forming and sintering
  - Cold Isostatic Pressing and sintering
  - Hot Pressing

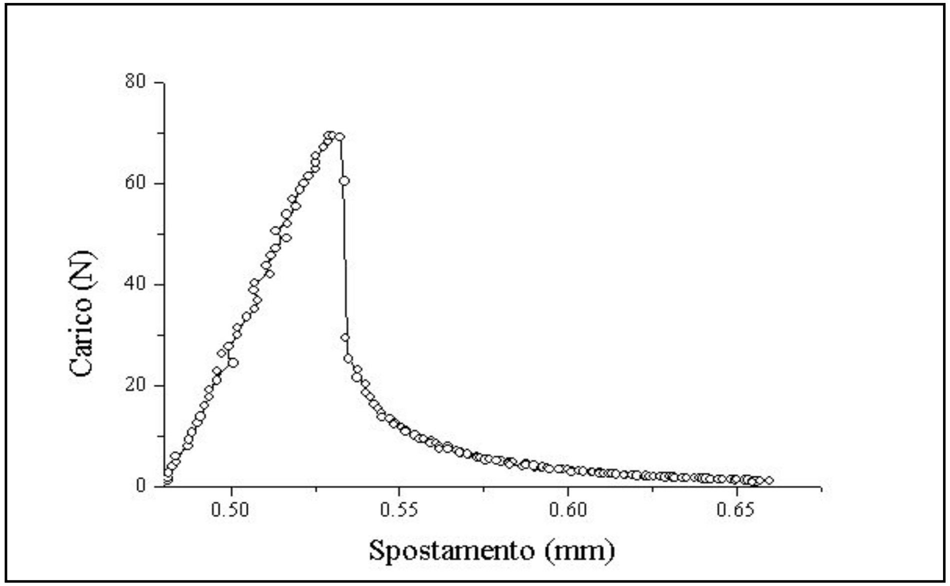
- **Active binder**
  - Binder + powder Al_2O_3 - Ni
  - Polimerization and forming
  - Thermal treatment (decomposition) + sintering

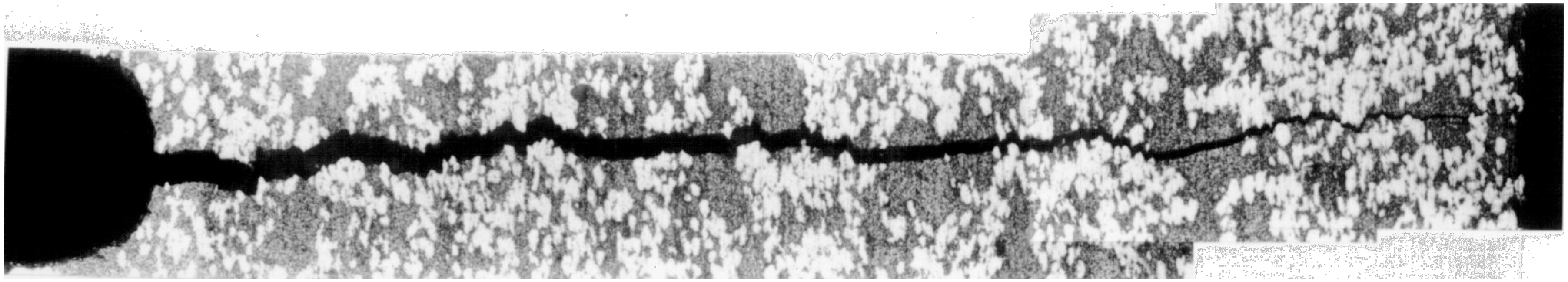
SEM: Al2O3 - 40 % Ni



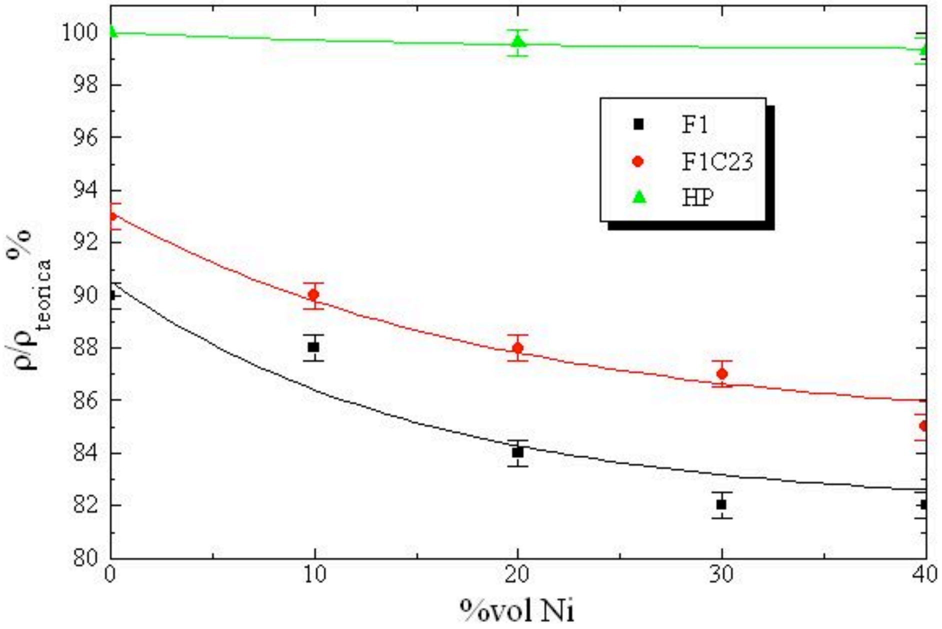
Mechanical properties

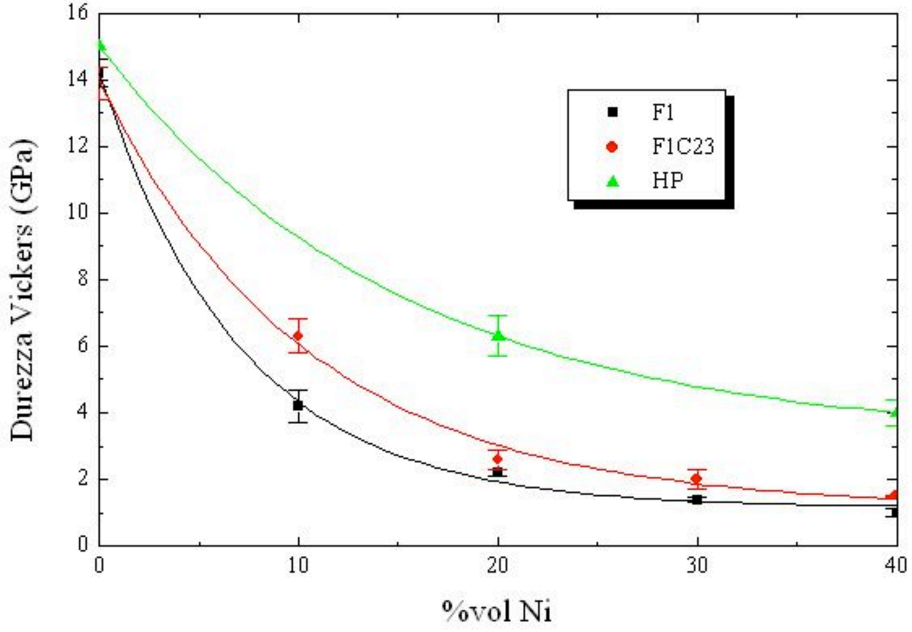
- **Toughness: SENB-S modified**
- **Elastic modulus three point bending**



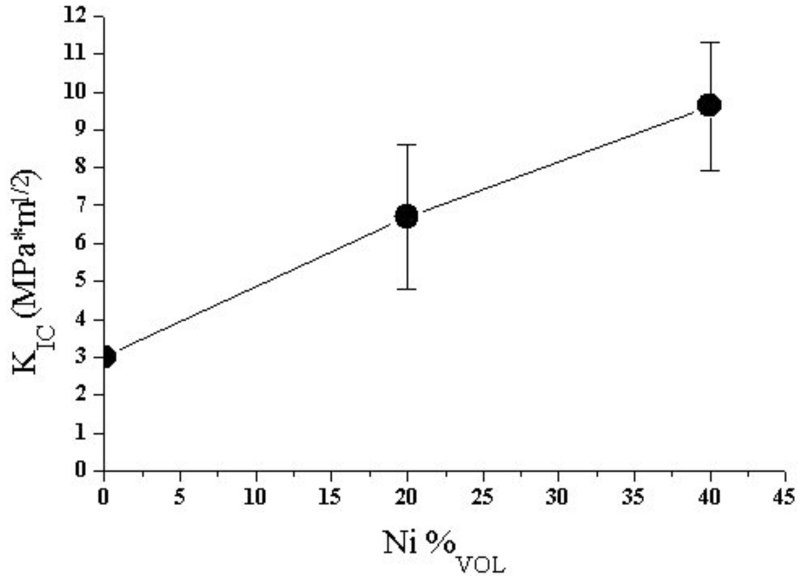


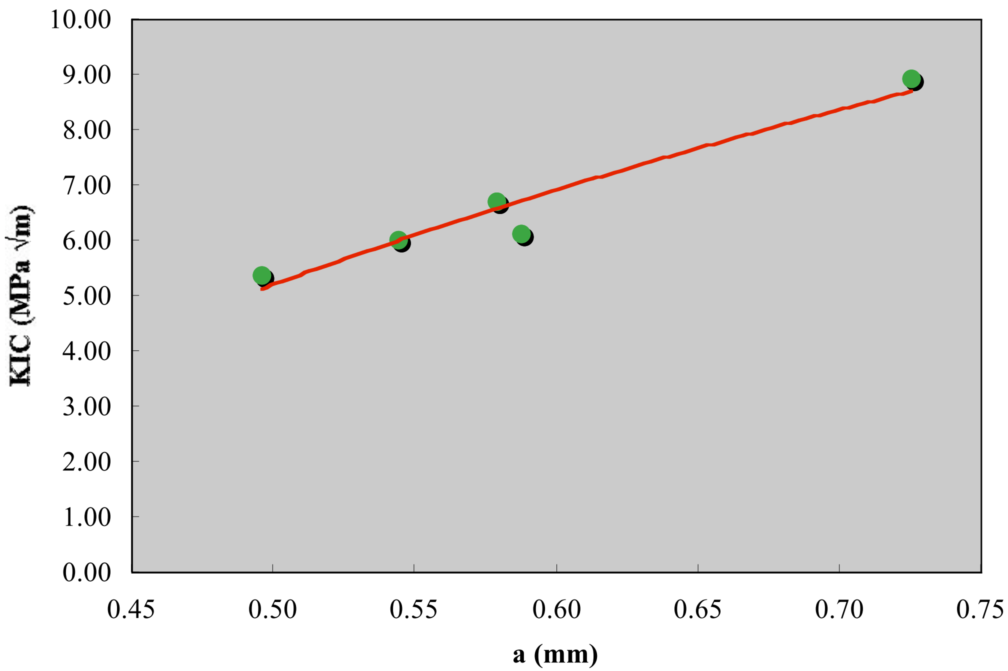
Density and Hardness



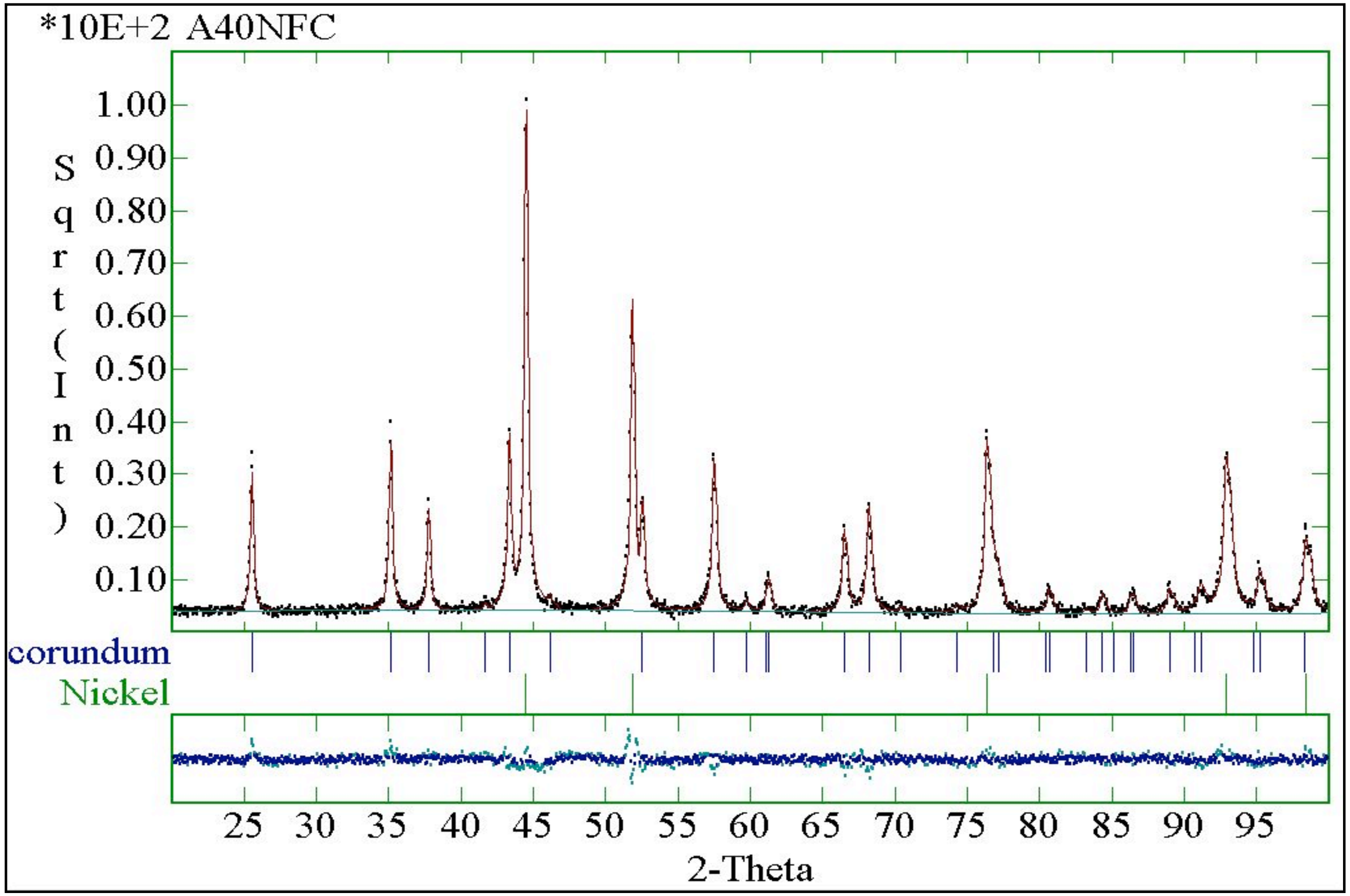


Toughness and R-curve





Quantitative phase analysis



Quantitative phase analysis: problems

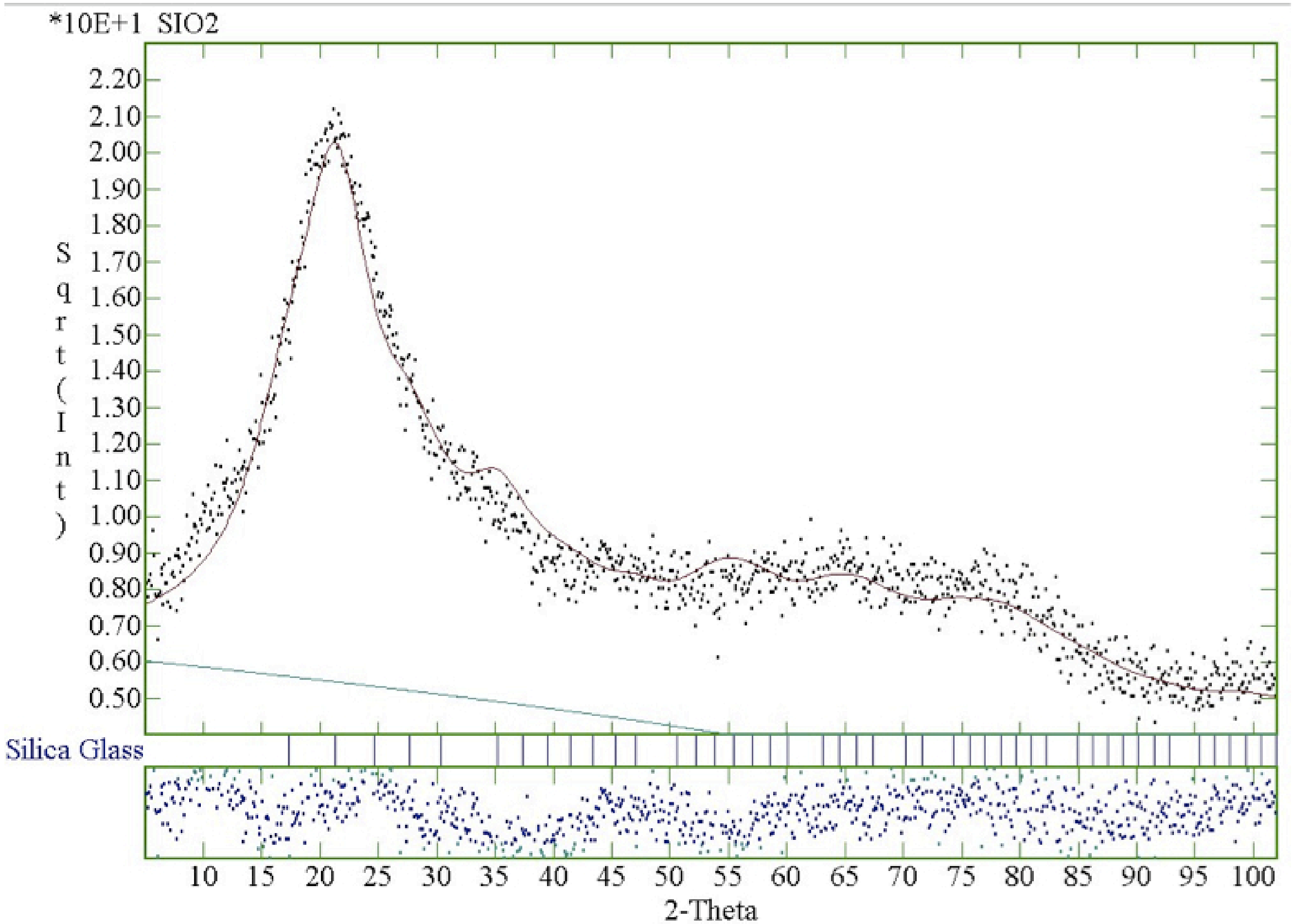
- High contrast in absorption between Ni and alumina

	No Correction		Brindley Cubic Corr.		Brindley Spheric Corr.	
	$\%Al_2O_3$ VOL	%Ni VOL	$\%Al_2O_3$ VOL	%Ni VOL	$\%Al_2O_3$ VOL	%Ni VOL
$Al_2O_3 + 40\%_{VOL}$ Ni Powders	72.1 (2)	27.9 (2)	61.3 (2)	38.7 (2)	63.8 (2)	36.2 (2)
Pure Al_2O_3 F1	100 (0)	0 (0)	100 (0)	0 (0)	100 (0)	0 (0)
$Al_2O_3 + 10\%_{VOL}$ Ni F1	94.7 (1)	5.34 (1)	89.9 (1)	10.1 (1)	91.2 (1)	8.8 (1)
$Al_2O_3 + 20\%_{VOL}$ Ni F1	88.1 (1)	11.9 (1)	79.9 (1)	20.1 (1)	82.0 (1)	18.0 (1)
$Al_2O_3 + 30\%_{VOL}$ Ni F1	80.7 (2)	19.3 (2)	70.4 (2)	29.6 (2)	72.9 (2)	27.1 (2)
$Al_2O_3 + 40\%_{VOL}$ Ni F1	70.1 (3)	29.9 (2)	59.2 (3)	40.8 (2)	61.7 (3)	58.1 (2)
Pure Al_2O_3 F1C23	100 (0)	0 (0)	100 (0)	0 (0)	100 (0)	0 (0)
$Al_2O_3 + 10\%_{VOL}$ Ni F1C23	91.4 (1)	8.6 (1)	84.6 (1)	15.4 (1)	86.4 (1)	13.6 (1)
$Al_2O_3 + 20\%_{VOL}$ Ni F1C23	88.2 (1)	11.8 (1)	79.8 (1)	20.2 (1)	82.0 (1)	18.0 (1)
$Al_2O_3 + 30\%_{VOL}$ Ni F1C23	79.8 (2)	20.2 (2)	69.4 (2)	30.6 (2)	71.9 (2)	28.1 (2)
$Al_2O_3 + 40\%_{VOL}$ Ni F1C23	70.6 (3)	29.4 (3)	60.4 (3)	39.6 (3)	62.9 (3)	37.1 (3)
$Al_2O_3 + 20\%_{VOL}$ Ni HP (b)	88.4 (1)	11.6 (1)	80.4 (1)	19.6 (1)	82.4 (1)	17.6 (1)
$Al_2O_3 + 20\%_{VOL}$ Ni HP (w)	88.4 (1)	11.6 (1)	80.3 (1)	19.7 (1)	82.4 (1)	17.6 (1)
$Al_2O_3 + 20\%_{VOL}$ Ni HP (Mo)	89.7 (1)	10.3 (1)	78.8 (1)	21.2 (1)	81.4 (1)	18.6 (1)
$Al_2O_3 + 40\%_{VOL}$ Ni HP (b)	73.8 (2)	26.2 (2)	63.1 (2)	36.9 (2)	65.6 (2)	34.4 (2)
$Al_2O_3 + 40\%_{VOL}$ Ni HP (w)	70.2 (2)	29.8 (2)	59.2 (2)	40.6 (2)	61.9 (2)	38.1 (2)
$Al_2O_3 + 40\%_{VOL}$ Ni HP (Mo)	76.1 (3)	23.9 (2)	61.5 (3)	38.5 (2)	64.7 (3)	35.3 (2)

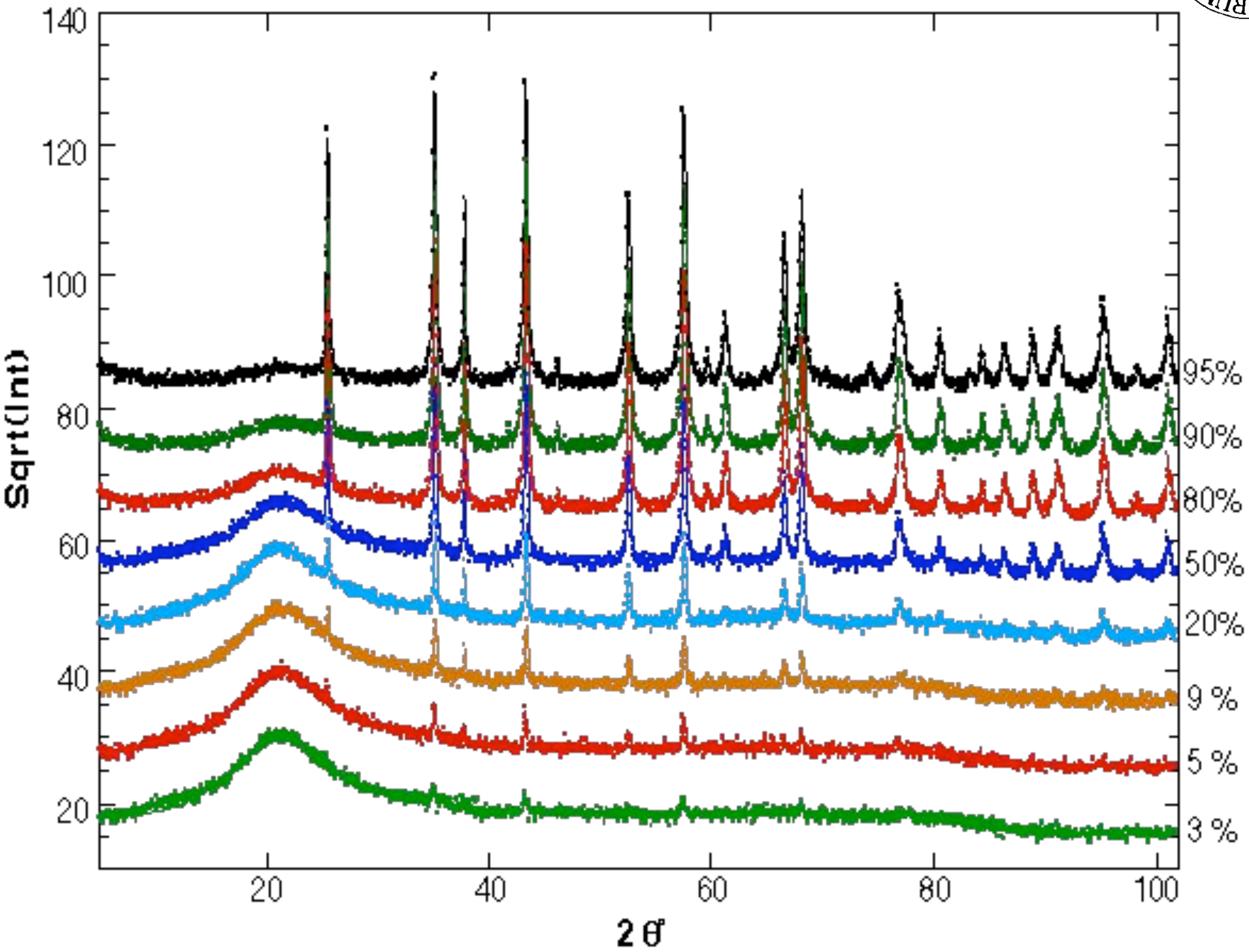
Use neutron

Pseudo-amorphous approximation

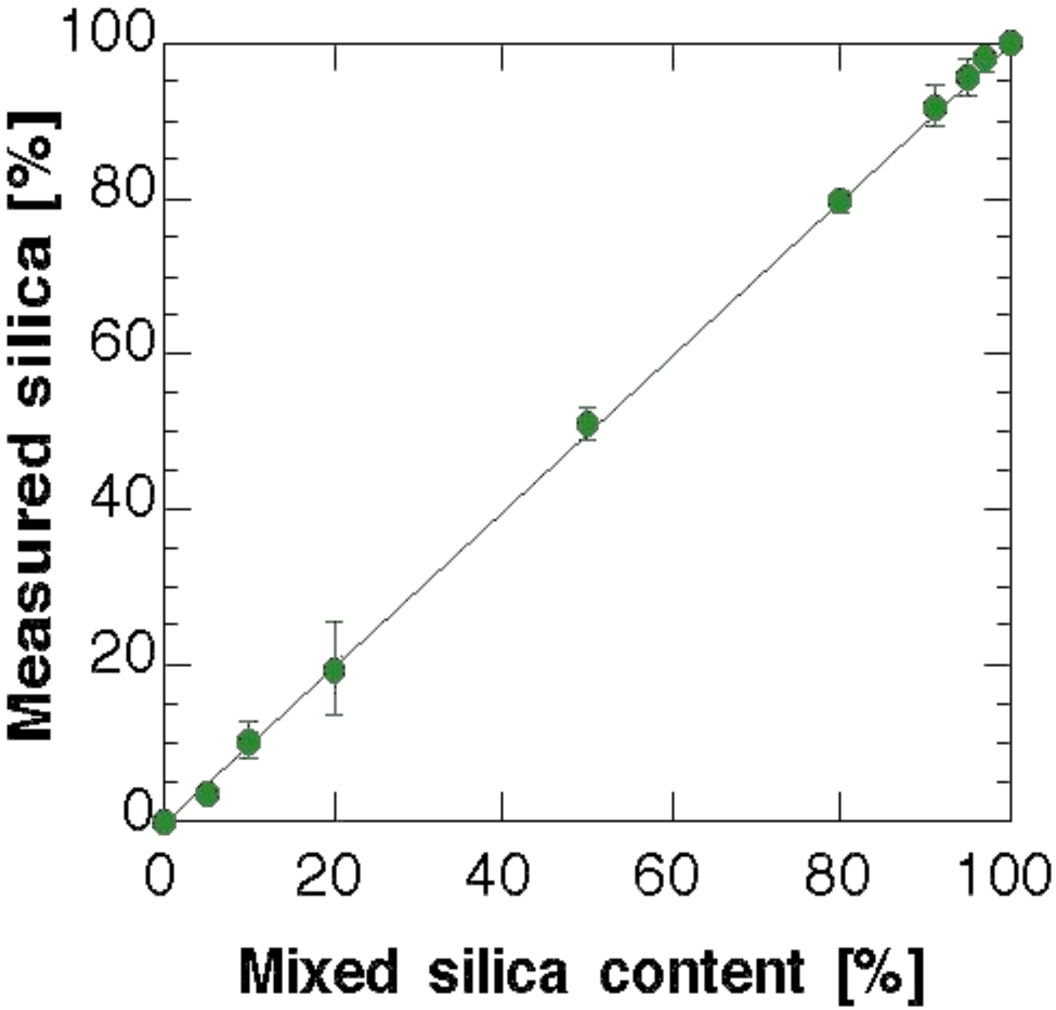
- Le Bail, A., *J. Non-Cryst. Solids*, 183, pp. 39-42, 1995



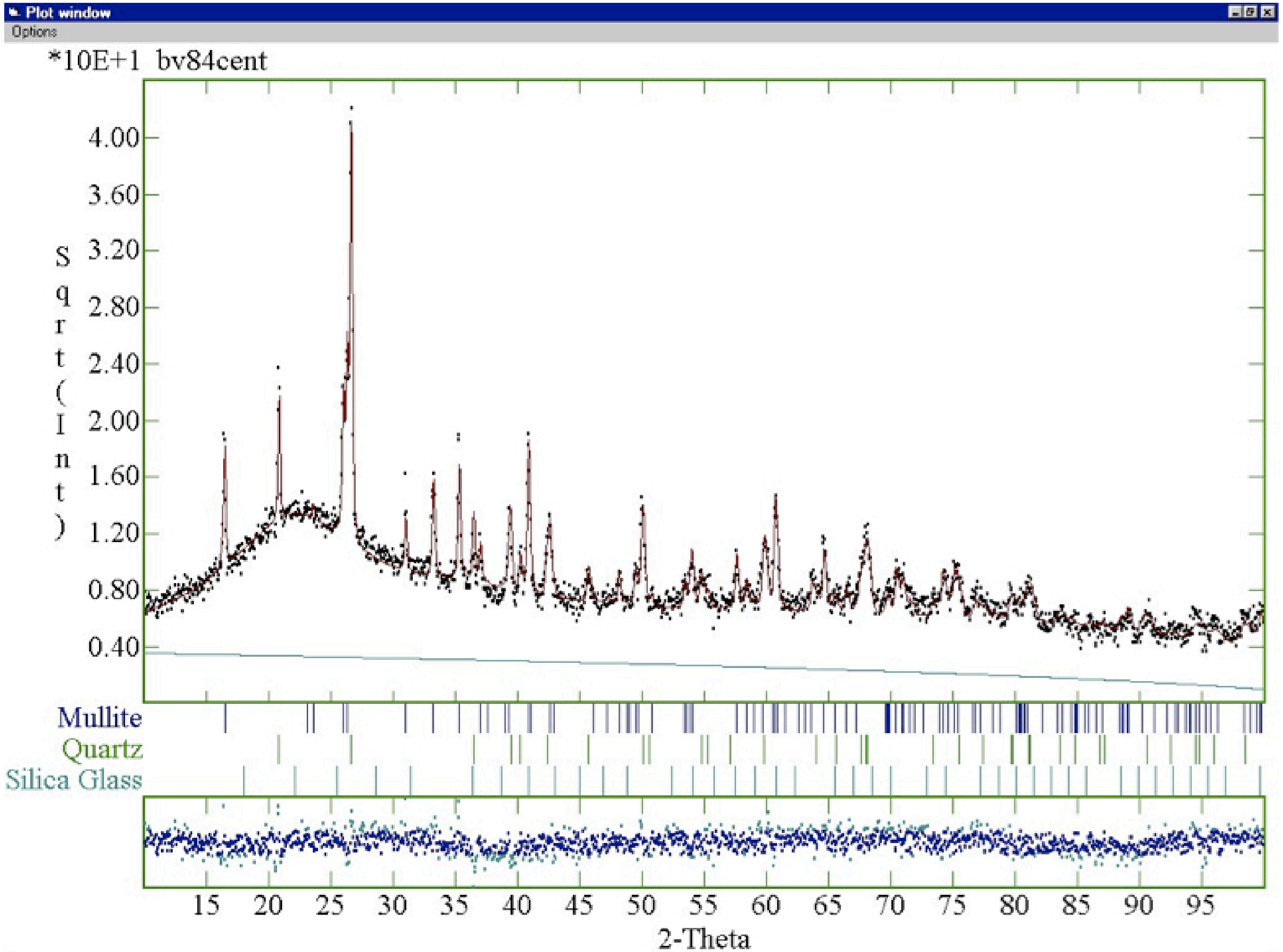
Corundum+amorphous silica mixed



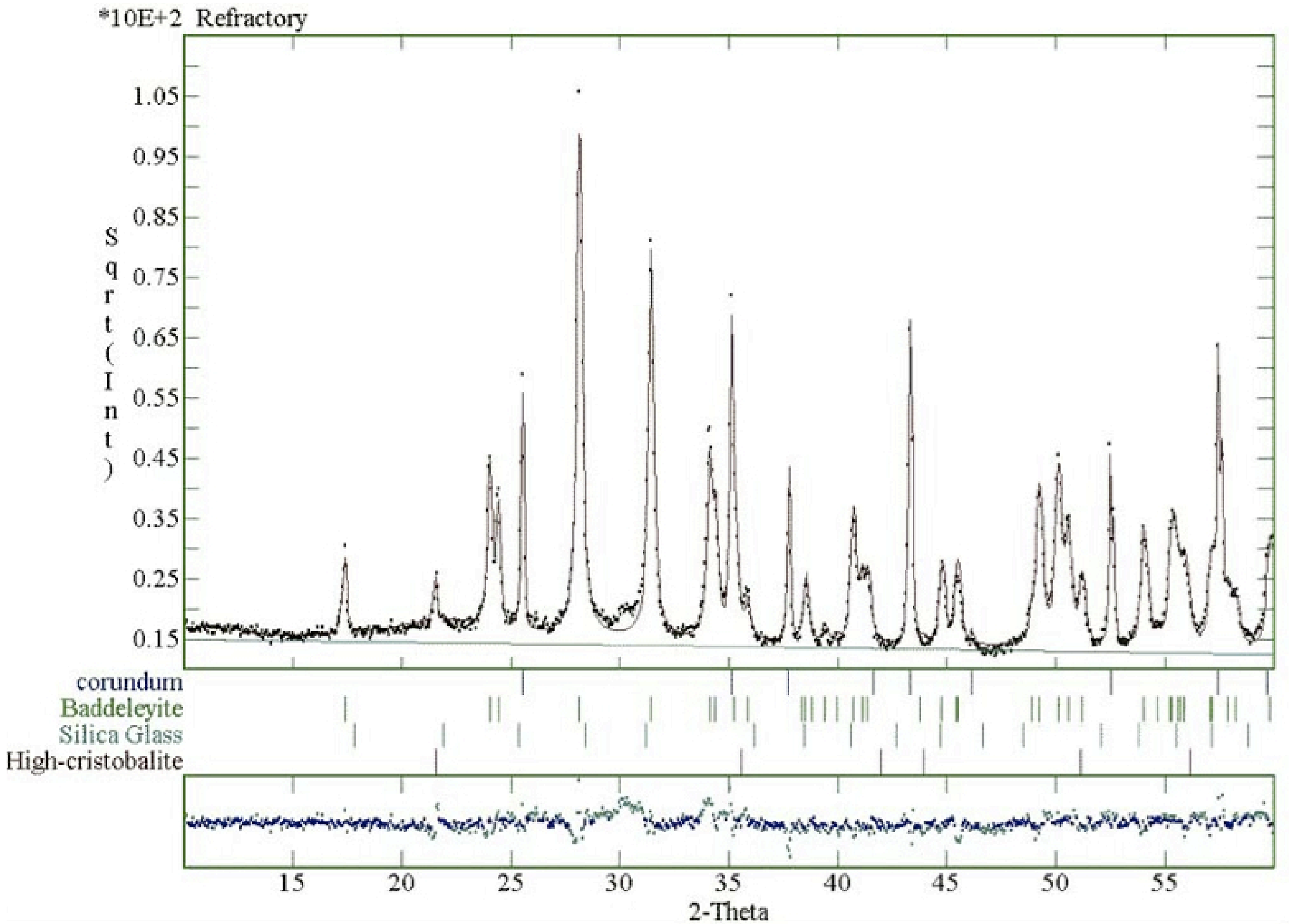
Result of the Rietveld analysis using the pseudo-amorphous approx.



Sanitary ware (>60% silica glass)



Ceramics for high temperature



Results

	Sanitary-ware		Refractory material
	Monochromator	Filtered radiation	Filtered radiation
Mullite	24.2(5)	20.5(7)	-
Quartz Low	13.3(2)	15.1(10)	-
Silica Glass	62.5(26)	64.4(6)	11(1)
Corundum	-	-	49(4)
Baddeleyite	-	-	39(1)
High Cristobalite	-	-	1(0.2)