Characterization of Fe-Cr alloys using SANS, nanoindentation and ultrasound

C. Heintze, F. Bergner, A. Ulbricht

Forschungszentrum Dresden-Rossendorf,
Dresden, Germany

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Plan

1. Aim and Scope
2. Experiments
   2.1 Materials
   2.2 SANS
   2.3 Nanoindentation
   2.4 Sound velocity measurement
3. Results and Discussion
   3.1 SANS
   3.2 Indentation hardness
   3.3 Elastic properties
   3.4 Irradiation response (selected results)
4. Conclusions

In general:

- Cr steels are candidate materials for Gen IV fission and fusion applications.
- A multiscale modelling approach to the irradiation behaviour of Cr steels requires modelling-oriented experiments.
- Effect of Cr on constitution and properties not yet well understood.

In particular:

- The present set of alloys already basically characterized in [1].
- Distribution in the framework of the EU project GETMAT.
- More complete basic characterization is desirable.
- Unirradiated conditions as reference for irradiation effects.

[1] M. Matijasevic et al., EUROMAT 2005, and M. Matijasevic et al., JNM 377 (2008) 147

Composition

Fe-2.5at%Cr (Alloy 251)
Fe-5at%Cr (Alloy 259)
Fe-9at%Cr (Alloy 252)
Fe-12.5at%Cr (Alloy 253)

} Provided by SCK·CEN, Mol

Composition (wt%)

Alloy	Mn	Si	P	S	Al	Ti	Cr	Ni	O	C	N
251	0.009	0.02	0.013	0.0020	0.003	0.004	2.4	0.044	0.035	0.008	0.0117
259	0.02	0.04	0.011	0.006	0.0033	0.0028	4.6	0.06	0.065	0.02	0.0127
252	0.03	0.09	0.012	0.00066	0.0069	0.0034	8.4	0.07	0.066	0.02	0.0148
253	0.03	0.11	0.05	0.006	0.003	0.0037	11.6	0.09	0.03	0.027	0.0237

Treatment

Normalized (1050°C / 3 h / air cooling)
& tempered (730°C / 4 h / air cooling)

Contact: f.bergner@fzd.de

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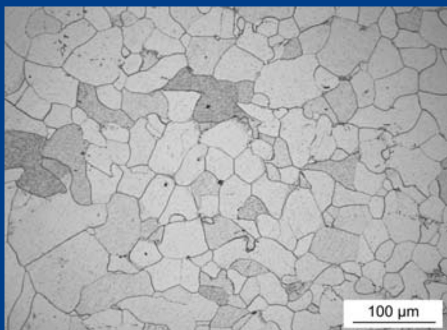
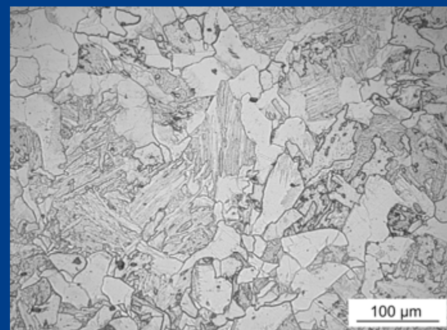
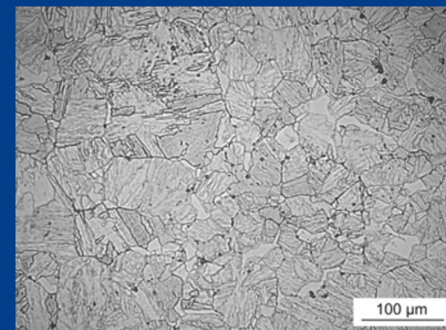
Fe-2.5at%Cr



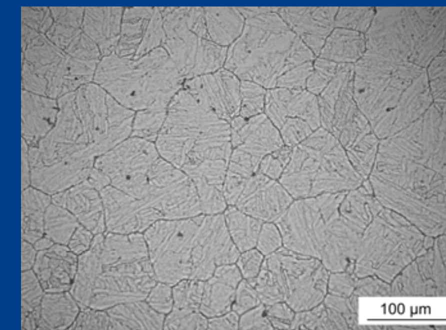
Fe-5at%Cr



Fe-9at%Cr



Fe-12.5at%Cr



Grain size

Ferrite fraction

TEM

Dislocation density, ρ / 10^{13} m^{-2}

Fe-2.5at%Cr	Fe-5at%Cr	Fe-9at%Cr	Fe-12.5at%Cr
1.2	5.8	6.3	5.5

Matijasevic et al., JNM 377 (2008) 147

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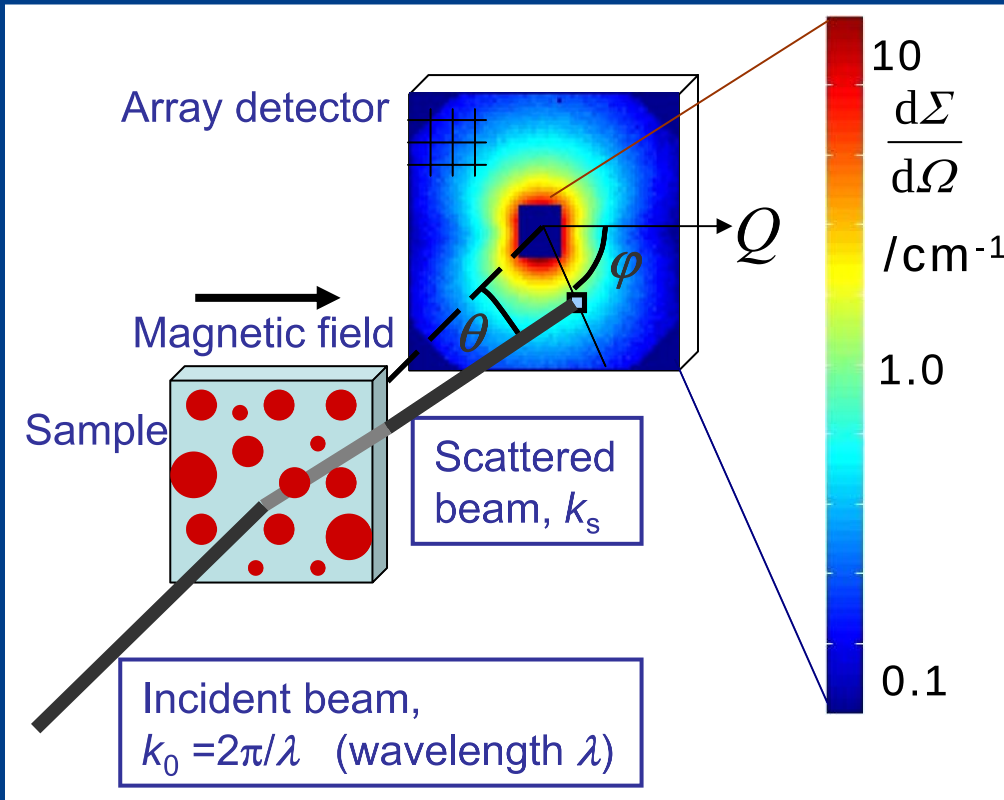
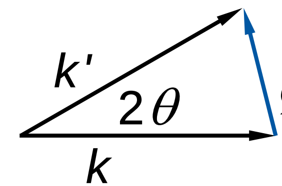
Principle



$$I(Q) = \frac{d\Sigma}{d\Omega}(Q), \quad [I] = \frac{\text{nm}^2}{\text{nm}^3\,\text{sr}} = \text{nm}^{-1}\,\text{sr}^{-1}$$



$$Q = \frac{4\pi}{\lambda}\sin\theta, \quad [Q] = \text{nm}^{-1}$$

$$\frac{d\Sigma}{d\Omega} = \frac{d\Sigma}{d\Omega}_{N} + \frac{d\Sigma}{d\Omega}_{M} \sin^2\varphi$$

$$A = 1 + \frac{d\Sigma}{d\Omega}_{M} \Big/ \frac{d\Sigma}{d\Omega}_{N}$$

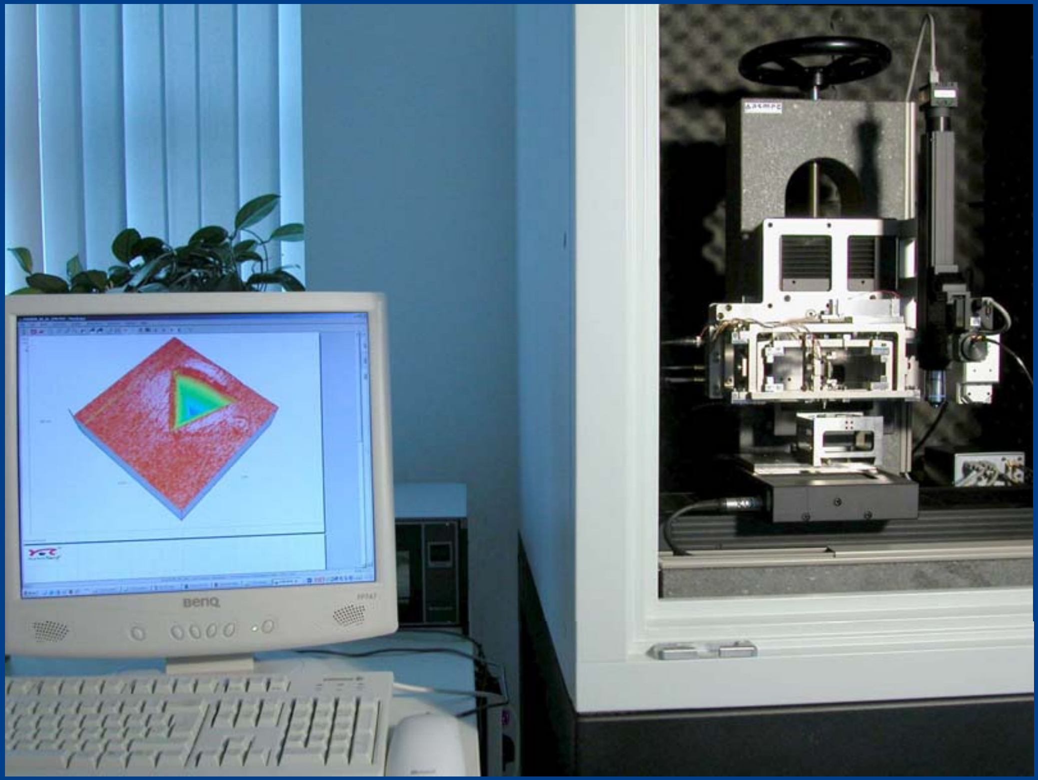
$$\frac{d\Sigma}{d\Omega}_{M(N)}(Q) \xrightarrow{\text{Inverse problem}} \Delta\eta^2_{M(N)}\,c(R)$$

SANS experiments at GKSS Geesthacht, Germany

λ= 0.58 nm; sample-detector distances: 1, 4 and 16 m; Q range: 0.1 - 3 nm^{-1}

Thanks to the local contact, H. Eckerlebe, for technical assistance.

UNAT



<u>Nanomechanics device – UNAT (ASMEC)</u>
- force resolution <100 nN
- displacement resolution <50 pm
- force noise <10 µN
- displacement noise <1 nm
- x-y-z stage
  range 200 mm x 50 mm x 50 mm
  steps 0.5 µm x 0.1 µm x 0.1 µm
- tandem microscope (optical)
- Berkovich indenter
- housing with thermal and electro-magnetic isolation and active vibration damping

<u>AFM Nanite B (Nanosurf)</u>
- built-in or stand-alone
- x-y-z range 110 µm x 110 µm x 20 µm
- contact- and dynamic mode

Pulse-echo technique, 5 MHz, RT

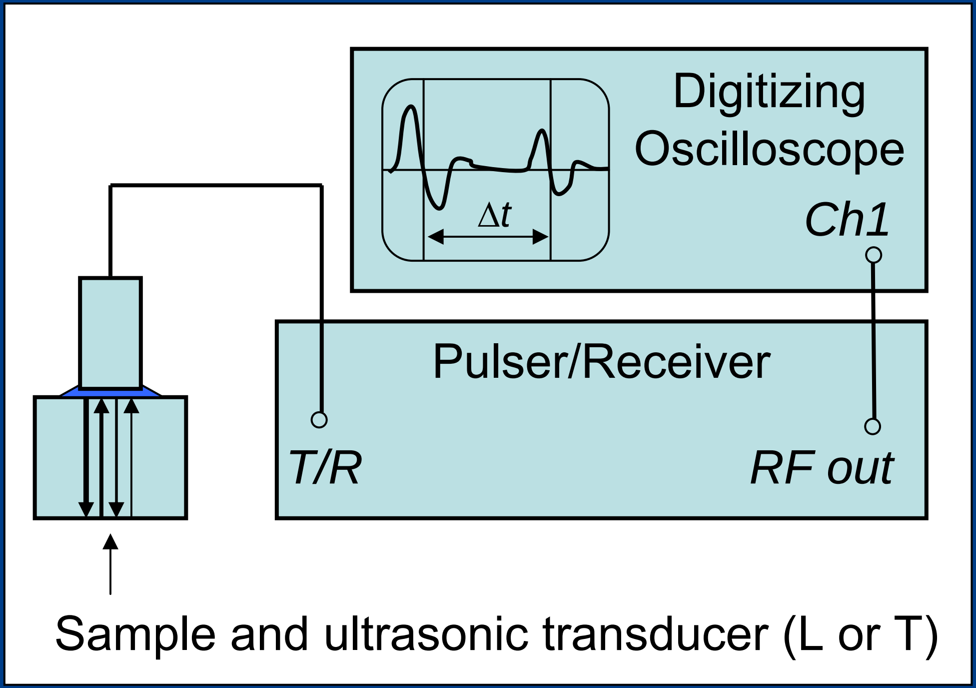
$d \approx 10$ mm, Δt_L, Δt_T → c_L, c_T → ν, E, G, B

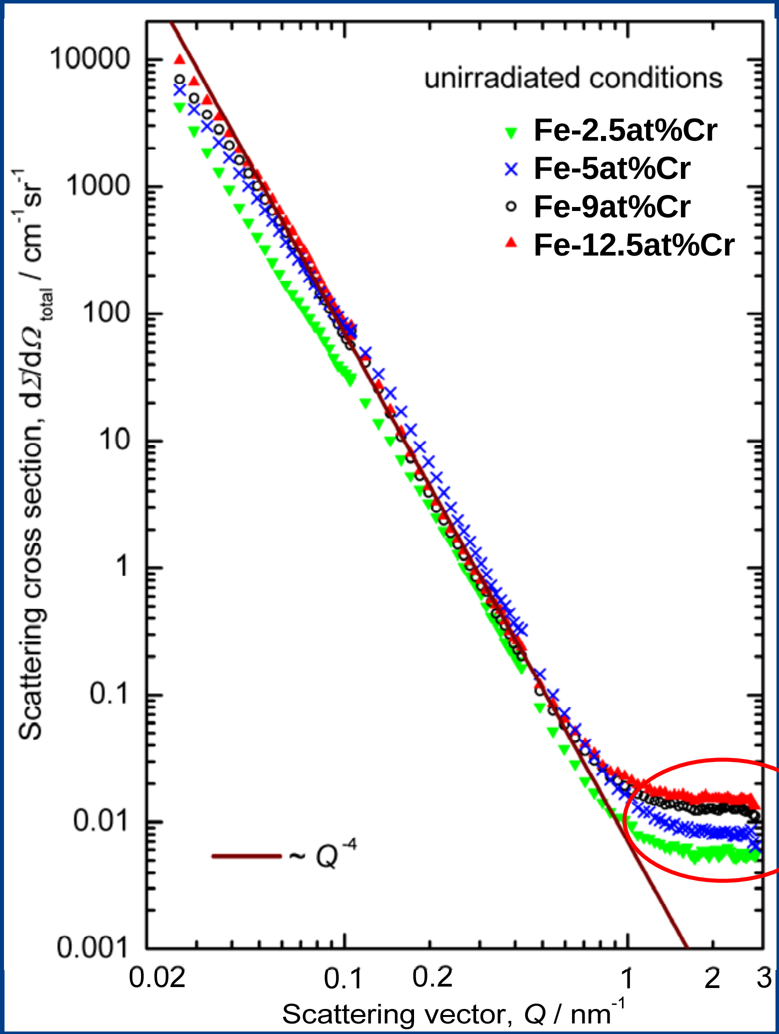
$$\nu = \frac{1 - 2(c_L / c_T)^2}{2 - 2(c_L / c_T)^2}$$

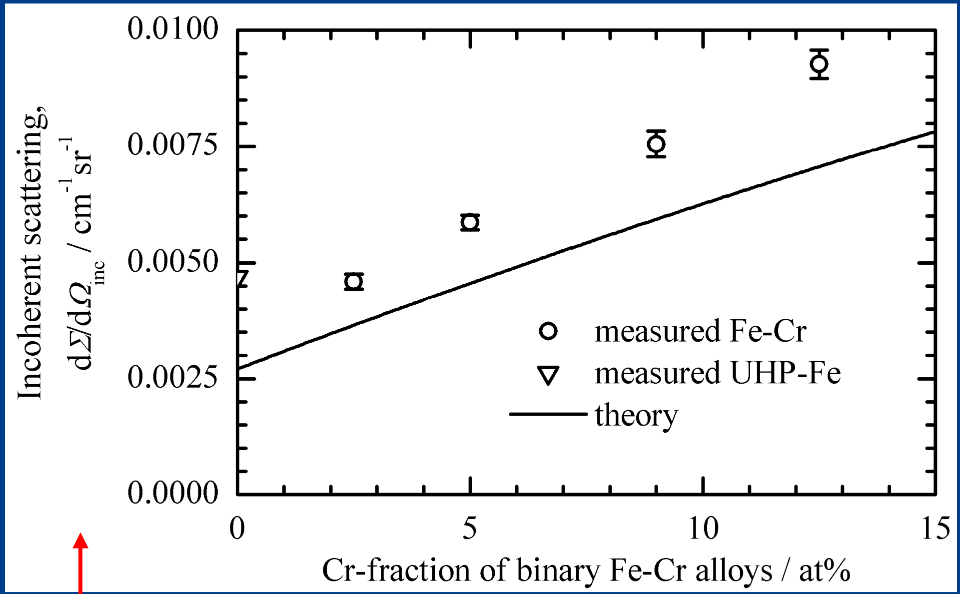
$$G = \rho c_T^2$$

$$E = \rho c_L^2 \frac{(1+\nu)(1-2\nu)}{1-\nu}$$

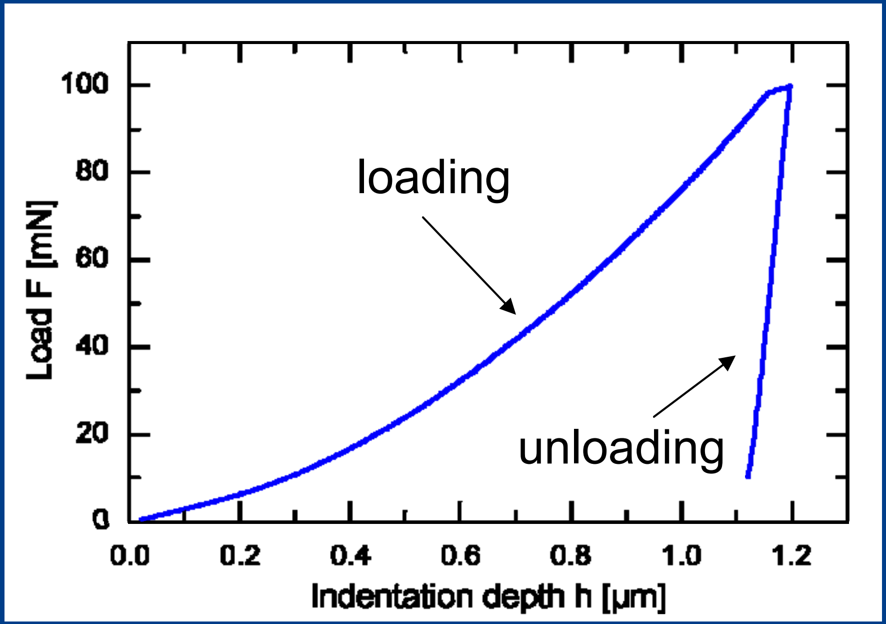
$$B = \rho c_L^2 \frac{1+\nu}{3(1-\nu)}$$



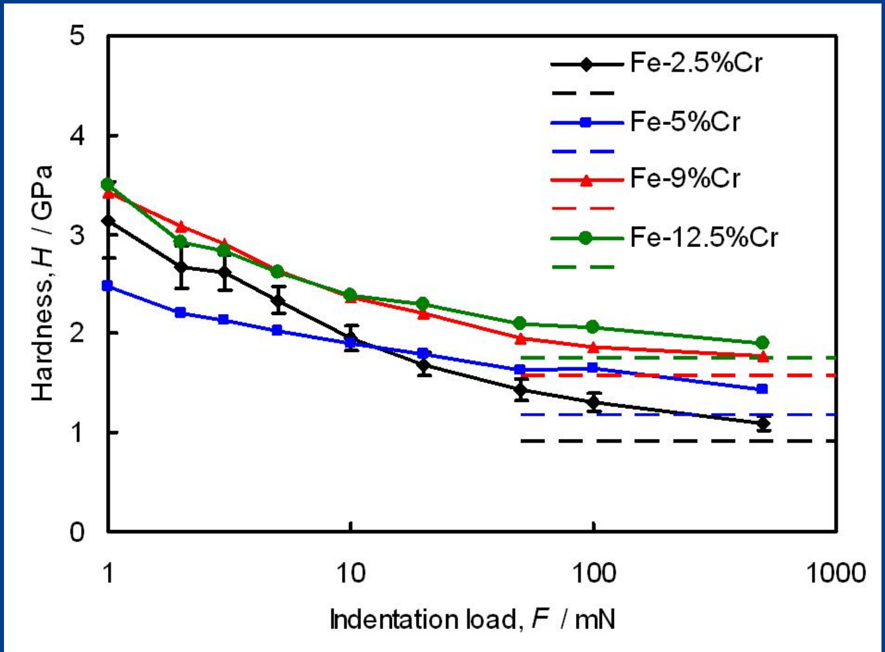




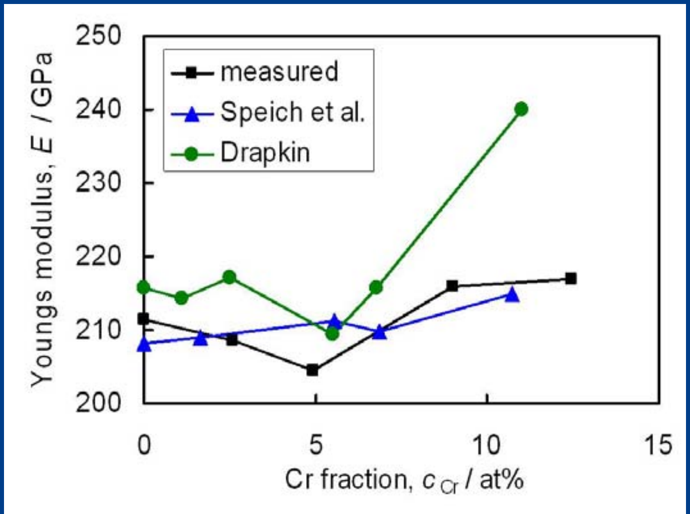
Incoherent scattering:
- mixture of Fe isotopes
- mixture of Cr isotopes
- Fe-Cr solid solution
- impurities and defects

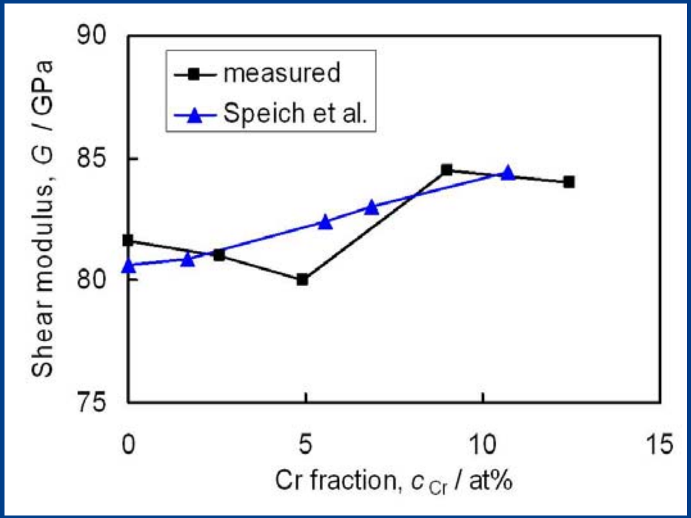


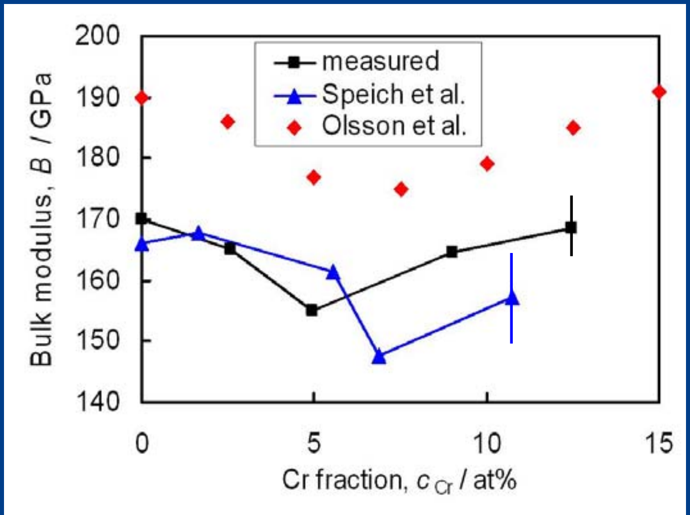
Hardness = $\frac{\text{Load}}{\text{Contact area under load}}$



Main findings:
- indentation-size effect
- macro-hardness is approached
- trend with increasing Cr
- anomaly for 5 at% Cr ?



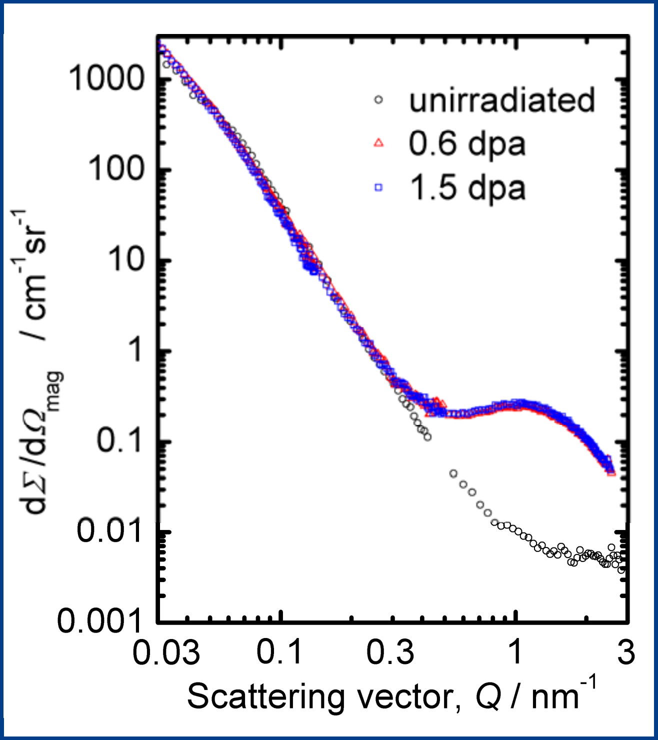


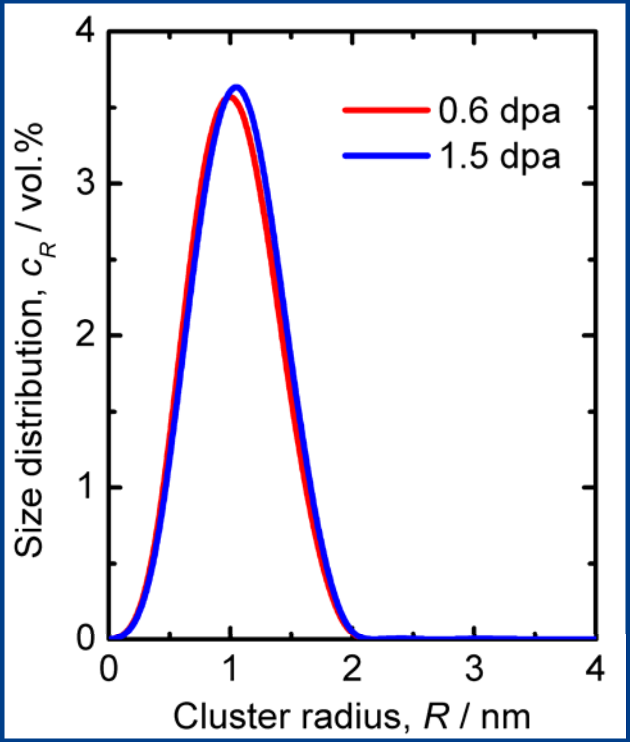


Main findings:

- previous work partly contradictiory
  Speich et al., Met. Trans. (1972)
  Drapkin, Probl. Prochnosti (1973)

  minimum of *E*, *G* and *B* at ~5 at% Cr in agreement with MD calculations
  Olsson et al., JNM 321 (2003) 84
- possible source of the observed hardness anomaly?





SANS

Fe-12.5at%Cr, neutron-irradiation, 300°C, 0.6 dpa, 1.5 dpa

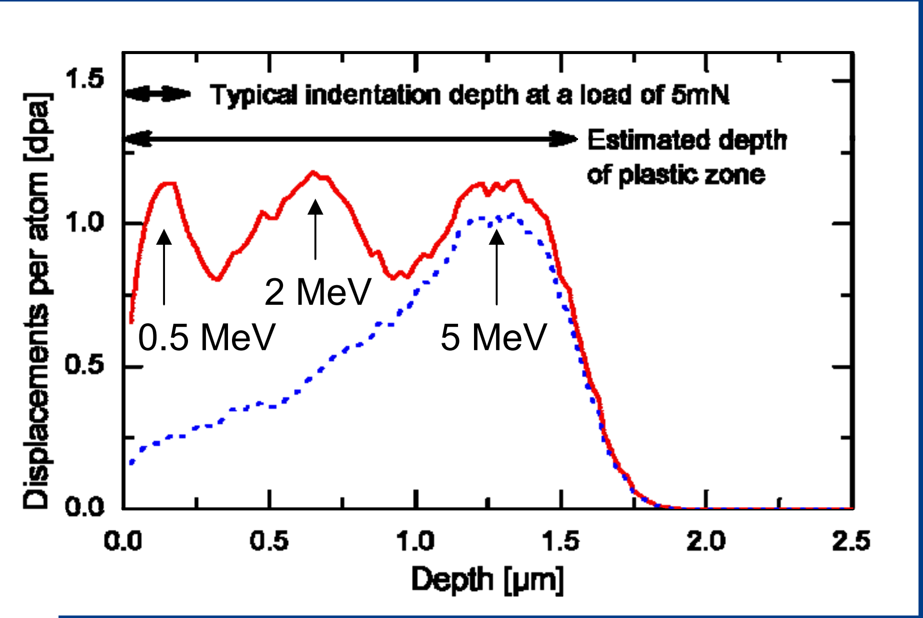
Main findings: (1) saturation, (2) formation of α',
(3) solubility of Cr in Fe at 300°C: (8.8 ± 0.5) at% Cr

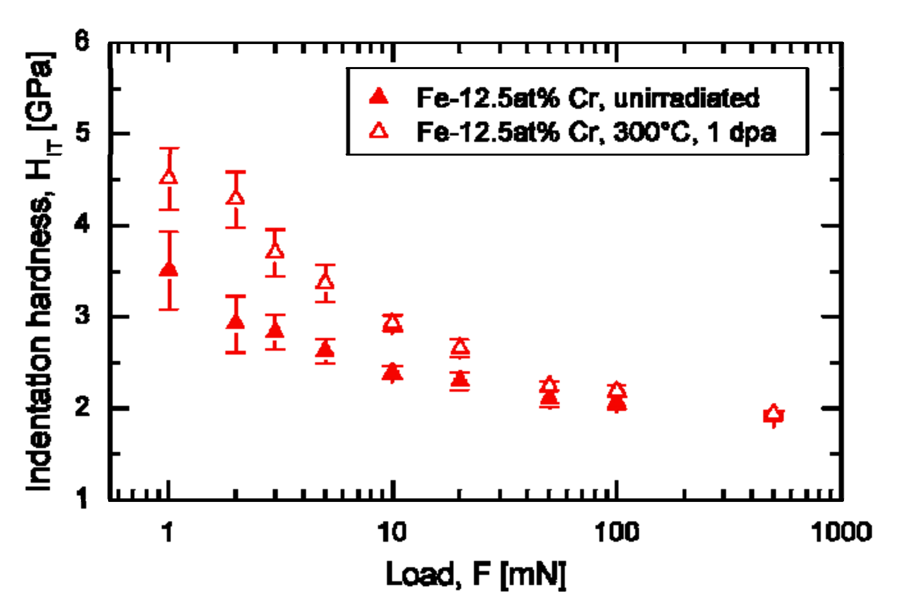
Bergner et al., Scripta Materialia, 61 (2009) 1060

Nanoindentation

Fe-12.5at%Cr
Fe-ion irradiation, 300°C, 1 dpa

three ion energies (0.5, 2, 5 MeV)
vs one single energy (5 MeV)





Main findings:

- accuracy increases with load
- irrad. effect decreases with load (effect of substrate)
- 5 mN: good trade-off
- mechanistic interpretation:

Heintze et al., ICFRM-14, September 7-12, 2009, Sapporo, Japan

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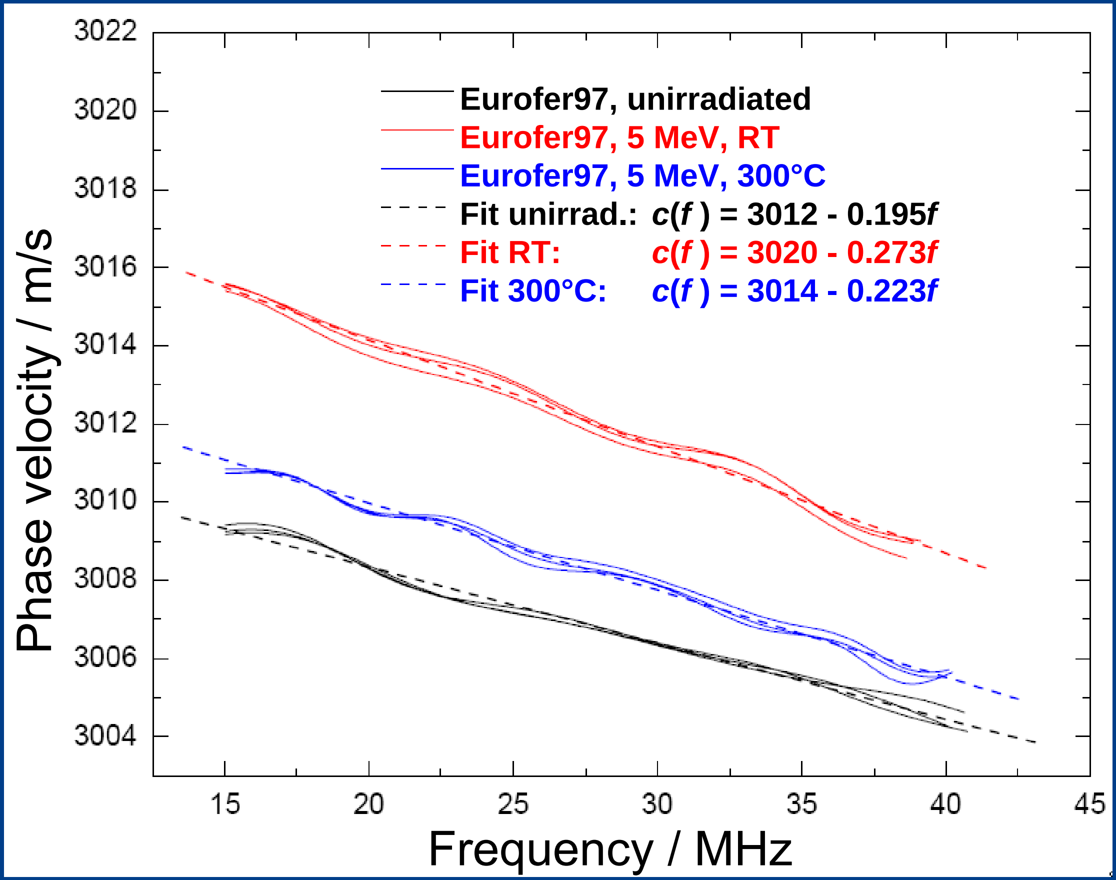

Laser acoustic measurement of surface acoustic wave (SAW) velocity

Eurofer97, Fe-ions, RT and 300°C

→ Irradiation-induced change of SAW velocity dispersion

C. Recknagel, Diploma thesis, TU Dresden, 2007
Thanks to D. Schneider, IWS Dresden, for SAW measurements

As-received (unirradiated) conditions of Fe-Cr alloys with 2.5, 5, 9 and 12.5 at% Cr characterized by means of SANS, nanoindentation and ultrasonics

- Elastic properties and nanohardness exhibit peculiar behaviour at about 5 at%, (but SANS not)

- Effect of irradiation (neutrons, Fe-ions) highlighted by selected applications

Thank you for your attention