Application of a three-feature dispersed-barrier hardening model to neutron-irradiated FeCr alloys

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HZDR
HELMHOLTZ
ZENTRUM DRESDEN
ROSSENDORF

Outline

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2 Model

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- Materials and Irradiations
- Tensile tests
- TEM
- APT
- SANS

4 Best rms fit, results and discussion

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Introduction

Industrial purity Fe-Cr alloys fabricated by SCK-CEN, irradiated in the BR2 reactor

Tensile tests performed at SCK-CEN → Yield stress as function of neutron exposure, Cr and test temperature and first TEM.

EU FP7 project GETMAT →
SANS performed by HZDR at GKSS Geesthacht
APT performed by and at Uni de Rouen/CNRS
TEM performed by and at CIEMAT … + others

Three kinds of irradiation-induced nanofeatures detected and quantified:
a‘ phase particles,
NiSiPCr enriched clusters,
interstitial dislocation loops.

Resulting question: Are the observed features potentially sufficient to explain the observed hardening?

Application of a three-feature dispersed-barrier hardening model.
Work in progress !!!

Model

Random distribution of barriers of type x, number density N_x and diameter d_x.
BKS model (*Bacon, Kocks, Scattergood , Phil. Mag. 1973*):

$$\Delta\sigma_{\mathrm{y}x} = f(N_x, d_x) \stackrel{\mathrm{def}}{=} \alpha_x \frac{M\mu b}{2\pi l}\left[\ln\left(\frac{l}{b}\right)\right]^{-1/2}\left[\ln\left(\frac{d'}{b}\right)\right]^{3/2}$$

$$l = \frac{1}{\sqrt{N_x d_x}} \qquad d' = \frac{d_x l}{d_x + l}$$

M = 3.06	Taylor factor.
μ = 84000 MPa	shear modulus.
b = 0.248 nm	Burgers vector.
→ α_x	unknown dimensionless obstacle strength.

Simplified version (periodic array of obstacles, no self-stress of the dislocation):

$$\Delta\sigma_{\mathrm{y}x} = f_{\mathrm{S}}(N_x, d_x) \stackrel{\mathrm{def}}{=} \alpha_{x\mathrm{S}} M\mu b\sqrt{N_x d_x}$$

Model

Superposition rules:

(1) Linear superposition of pre-existing barriers and irradiation-induced barriers:

→ implicitly expressed by the use of $\Delta\sigma_y = \sigma_{y,i} - \sigma_{y,u}$

(2a) Linear superposition of barriers of different kinds:

$$\Delta\sigma_\mathrm{y} = \alpha_\mathrm{A}\, f(N_\mathrm{A}, d_\mathrm{A}) + \alpha_\mathrm{L}\, f(N_\mathrm{L}, d_\mathrm{L}) + \alpha_\mathrm{C}\, f(N_\mathrm{C}, d_\mathrm{C})$$

(2b) Square superposition of barriers of different kinds:

$$\left(\Delta\sigma_\mathrm{y}\right)^2 = [\alpha_\mathrm{A}\, f(N_\mathrm{A}, d_\mathrm{A})]^2 + [\alpha_\mathrm{L}\, f(N_\mathrm{L}, d_\mathrm{L})]^2 + [\alpha_\mathrm{C}\, f(N_\mathrm{C}, d_\mathrm{C})]^2$$

Model

List of assumptions (incomplete):

(1) Uniform and random distributions of barriers (observed non-uniformity smeared out).
(2) A number of three distinguished families of barriers.
(3) Families represented by two parameters: Number density and mean size (size distributions ignored).
(4) Values of M, μ and b (crystallographic anisotropy and variety of slip systems ignored).
(5) Underlying superposition law.
…

(far too many)

Input parameters

Materials and irradiations:

Composition (wt%)

	Mn	Si	P	S	Al	Ti	Cr	Ni	O	C	N	V
Fe2.5Cr	0.009	0.02	0.013	0.0020	0.003	0.004	2.4	0.044	0.035	0.008	0.0117	0.001
Fe5Cr	0.02	0.04	0.011	0.006	0.0033	0.0028	4.6	0.06	0.065	0.02	0.0127	0.001
Fe9Cr	0.03	0.09	0.012	0.00066	0.0069	0.0034	8.4	0.07	0.066	0.02	0.0148	0.002
Fe12Cr	0.03	0.11	0.05	0.006	0.003	0.0037	11.6	0.09	0.03	0.027	0.0237	0.002

Austenitization: 1050 °C/1 h, tempering: 730 °C/4 h, air cooling

Neutron irradiation in the BR2 reactor:

- Irradiation temperature 300 °C.
- Neutron flux was 9 x 10^{13} cm^{-2} s^{-1} .
- Neutron exposure 0.06 dpa, 0.6 dpa, 1.5 dpa (nominal).

More details:

M. Matijasevic, A. Almazouzi, JNM 377 (2008) 147.

Input parameters

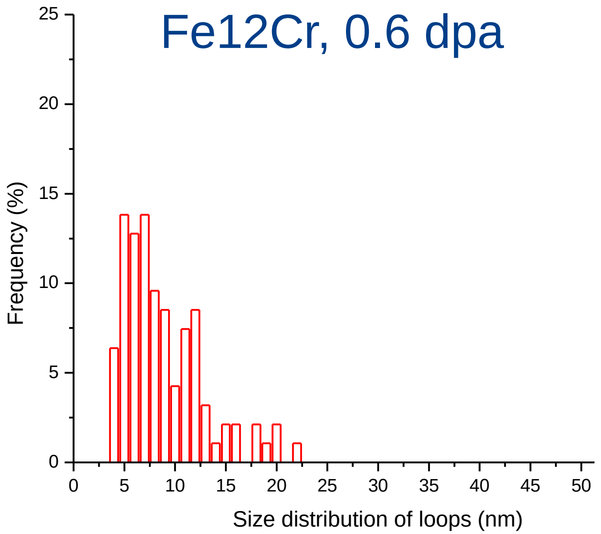
Overview of methods applied so far (PAS not included):

wt% Cr	dpa	TEM	APT	SANS	Tensile tests
2.4	0.06	X		X	X
	0.6	XX	X	X	X
	1.5	X		X	X
4.6	0.06	X		X	X
	0.6	XX	X	X	X
	1.5	X		X	X
8.4	0.06	XX		X	X
	0.6	XX	X	X	X
	1.5	XX		X	X
11.6	0.06			X	X
	0.6	XX	X	X	X
	1.5	X		X	X

Work in progress

Input parameters

TEM



Performed at CIEMAT Madrid
TEM: JEOL model JEM-2010 operating at 200 keV
(see talk by *M. Hernández Mayoral, this workshop*)

Findings:

- Non-uniform spatial distributions of loops observed for Fe5Cr, Fe9Cr and Fe12Cr.

→ Average density of loops calculated, treated as if they were uniformly distributed.

- Different families of loops not distinguished here.
- Mean diameter of loops measured from both BF images and WBDF images.
- Reasonable agreement between both measures and with earlier results reported by *Matijasevic et al., JNM 377 (2008) 147* (within a factor of 2).

→ Size obtained in BF contrast taken as reference case here.

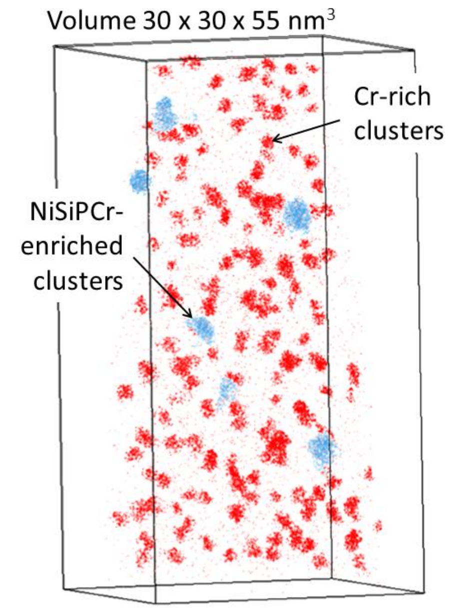
Input parameters

APT

- Energy Compensated Tomographic Atom Probe (ECoTAP)
- Energy Compensated Wide-Angle TAP (ECoWATAP)
- Laser Assisted Wide-Angle TAP (LAWATAP)

Performed at Université et INSA de Rouen/CNRS
(see e.g. *Kuksenko et al., JNM 432 (2013) 160*)

Fe12Cr, 0.6 dpa



Findings:

- Cr-rich clusters identified as α' for Fe9Cr and Fe12Cr.
- NiSiPCr-enriched clusters for each Cr content.

→ Total number density and average size of NiSiPCr-enriched clusters

- Narrow zones free of clusters around grain boundaries.

…

(many more)

Input parameters

SANS

Performed by HZDR at SANS-2 facility of GKSS Geesthacht, λ = 0.58 nm, ^3He-detector, 128 x 128 cells, saturation magentic field (see e.g. *Heintze et al., JNM 409 (2011) 106*)

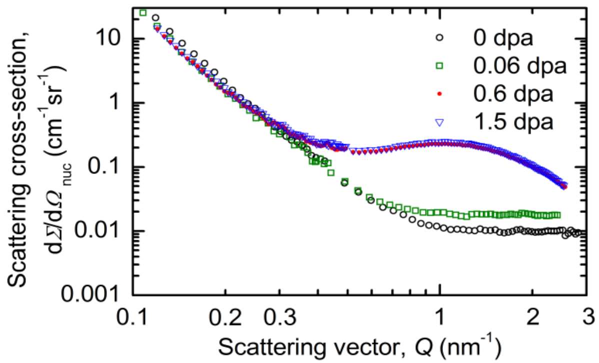
Findings:

- α'-phase nanoparticles clearly identified for Fe9Cr and Fe12Cr.

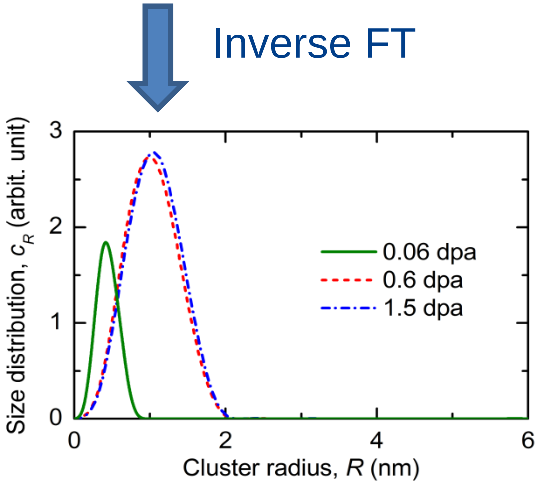
→ Total volume fraction, mean size and number density calculated, statistically reliable average over a macroscopic volume, reasonable agreement with APT (within a factor of 2).

- Other minor detections in Fe2.5Cr, Fe5Cr and Fe9Cr might be attributed to NiSiPCr-enriched clusters and/or Cr-C-enriched loops.

Fe12Cr

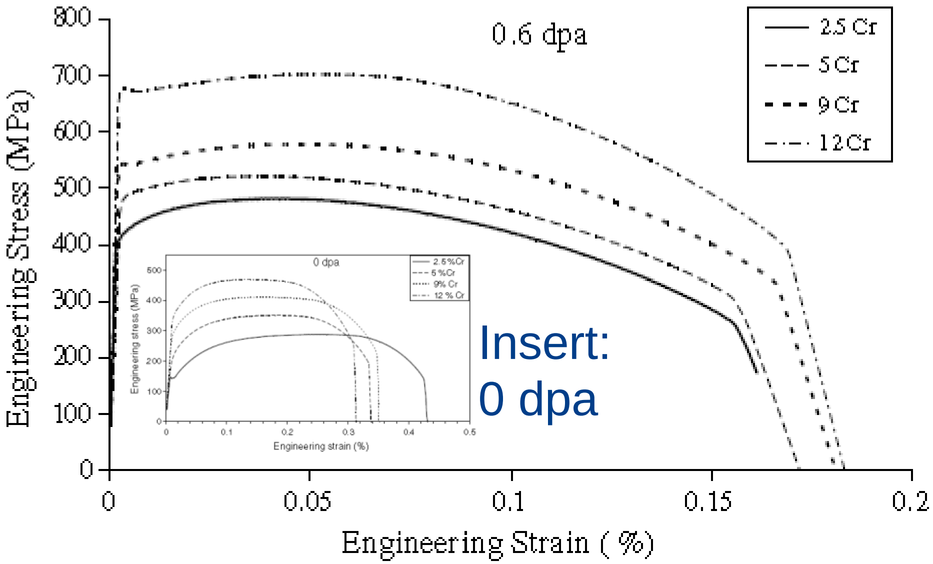


Inverse FT



Input parameters

Engineering stress-strain curves at RT



Tensile tests

Performed at SCK·CEN using miniaturized round tensile bars. (see *Matijasevic et al., JNM 377 (2008) 147*)

Findings:

→ Yield stress increase measured as a function of Cr and neutron exposure.

- In some cases yield point phenomenon observed.

Input parameters

Summary

			A		L		C	
Alloy specification			α' phase particles		Dislocation loops		NiSiPCr-rich clusters	
Cr (wt %)	Exposure (dpa)	$\Delta\sigma_y$ (MPa)	d_A (nm)	N_A (10^{16} cm^{-3})	d_L (BF) (nm)	N_L (10^{16} cm^{-3})	d_C (nm)	N_C (10^{16} cm^{-3})
2.4	0.6	277	-	0	13.2	0.44	3.6	13
4.6	0.6	283	-	0	8.2	0.52	4.4	13
8.4	0.6	255	1.82	25	6.5	0.13	3.3	24
11.6	0.6	327	1.66	990	7.4	0.18	3.2	11

Over-determined system of linear equations (four equations, three unknowns, 16 measured quantities)

Reference case: (BKS model, linear superpos., BF contrast)

$$\begin{pmatrix} 0 & 236 & 402 \\ 0 & 167 & 496 \\ 258 & 64 & 506 \\ 1564 & 87 & 325 \end{pmatrix} \begin{pmatrix} \alpha_A \\ \alpha_L \\ \alpha_C \end{pmatrix} = \begin{pmatrix} 277 \\ 283 \\ 255 \\ 327 \end{pmatrix}$$

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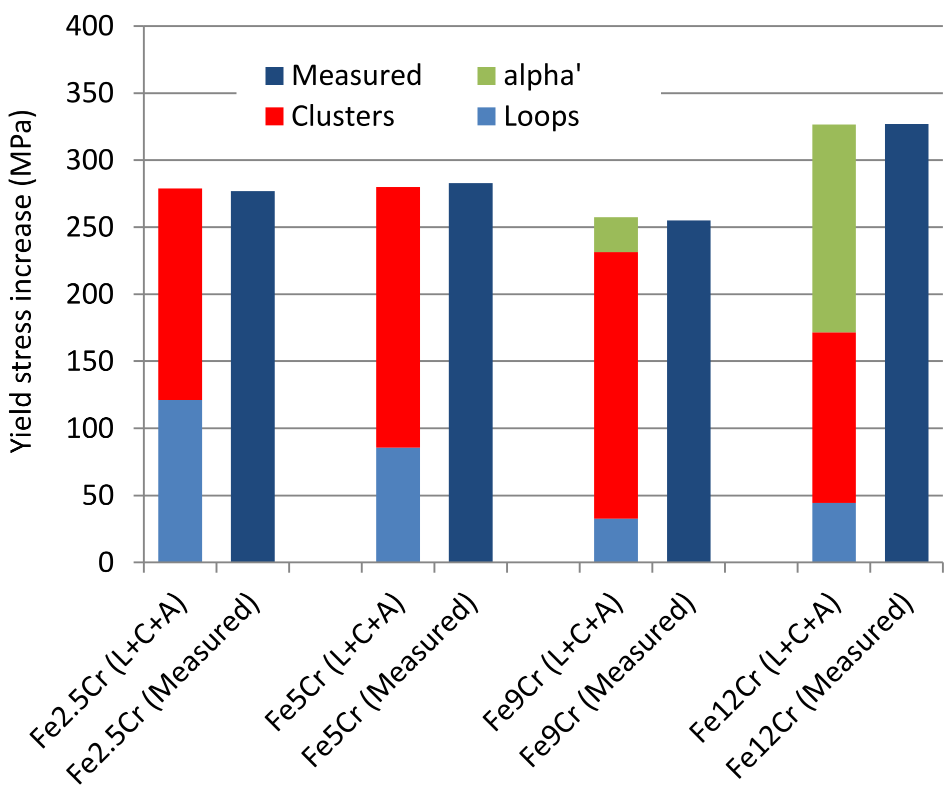
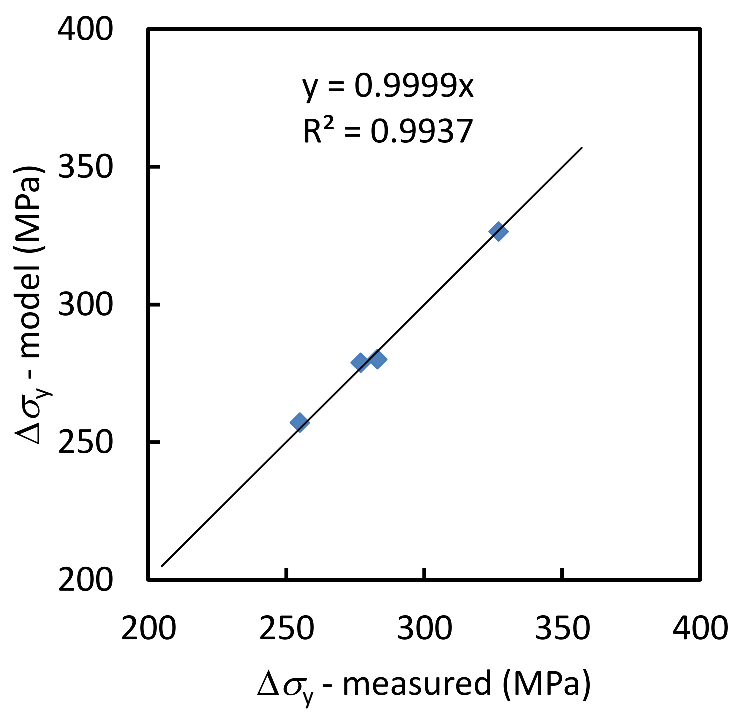
Best rms fit

Results: Reference case

Superposition / contrast	Obstacle strength			
	α_A	α_L	α_C	R^2
Linear / BF	0.099 ± 0.003	0.513 ± 0.026	0.392 ± 0.010	0.994





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Discussion

Reference case:

- The three-feature dispersed-barrier hardening model is sufficient to reproduce the measured yield stress increase within the range of measuring errors.
- The fitted model suggests that the dislocation loops are the strongest obstacles and the α'-phase particles are the weakest obstacles to dislocation glide.
- However, each of the families of obstacles does contribute significantly to the yield stress increase of the set of Fe-Cr alloys.
- Absolute values of the obstacle strength must not be overstressed.

Best rms fit

Results: Reference case and selected options

Superposition / contrast	Obstacle strength			
	α_A	α_L	α_C	R^2
Linear / BF (Reference case)	0.099 ± 0.003	0.513 ± 0.026	0.392 ± 0.010	0.994
Square / BF	0.177 ± 0.001	0.835 ± 0.032	0.489 ± 0.006	0.993
Linear / WBDF	0.096	0.599	0.431	0.64
Old set of data				
Linear / BKS	0.103 ± 0.004	0.755 ± 0.058	0.334 ± 0.019	0.991
Square / BKS	0.178 ± 0.001	1.085 ± 0.063	0.462 ± 0.007	0.986
Linear / Orowan	0.018 ± 0.003	0.425 ± 0.084	0.076 ± 0.019	0.89
Square / Orowan	0.033 ± 0.001	0.533 ± 0.059	0.125 ± 0.004	0.91

Discussion

Comparison of selected options (work in progress):

- The quality of fit does not depend significantly on the rule of superposition, i.e. the present set of data and the applied method cannot distinguish between linear and square superposition.
- The use of loop sizes derived from BF images gives much better „predictions“ than WBDF. This might be an indication for the TEM analysis but must not be overstressed, because the result may be biased by the assumptions
- The BKS model gives much better fits than the Orowan model, whichever superposition rule is used. This might be an indication that dislocation self-interaction and randomness of obstacles (both not included in the Orowan model) do matter.

Conclusion

(1) The three-feature dispersed-barrier BKS-type hardening model is sufficient to reproduce the measured yield stress increase within error for neutron-irradiated Fe2.5Cr, Fe5Cr, Fe9Cr and Fe12Cr.

(2) TEM, APT, SANS and tensile tests are suitable to provide the required set of input data.

(3) However, the goodness of fit does not mean that a fourth family of obstacles could not exist and be operative.

(4) All model options tested so far agree in the ranking of obstacle strengths according to: Dislocation loops > NiSiPCr-enriched clusters > α‘ particles. This ranking corresponds to expectation. However, the absolute values of the obstacle strength must not be overstressed.

Outlook

An increase of the degree of over-determination of the system of equations can be reached by adding the irradiation conditions (0.06 dpa, 1.5 dpa) not yet fully analyzed (work in progress).

The number of available materials and irradiation conditions is sufficient to evaluate a four-feature model.

The propagation of measuring errors is not yet included in the analysis, the given errors are just fit errors (work in progress).

Consideration of the source hardening model versus (or in combination with) the dispersed-barrier hardening model.