Review of IVAR Results

G. R. Odette
And
Many UCSB Co-workers and Other Collaborators
Mechanical & Environmental Engineering Department
Materials Department
University of California Santa Barbara

ASTM E10 Meeting

February 6, 2006

ATTACHMENT 3

Outline

- IVAR
- Flux, composition and heat treatment effects in Cu bearing steels
- Temperature effects
- Microstructural characterization
- Late blooming phases
- Low Cu steels - flux and composition dependence
- Other IVAR Activities
- Implications to analysis of the PRED

Experimental Knowledge Base

Alloys

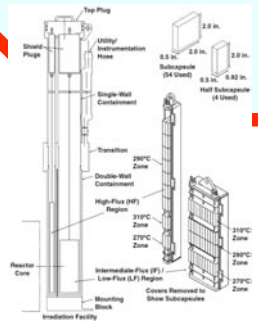
very large special alloy & split melt MV matrices > 100 alloy conditions in tailored specimen configurations

Cu	Ni	Mn
0.0	0.0	1.6
0.0	0.8	1.6
0.0	1.6	1.6
0.0	0.8	0.0
0.05	0.8	1.6
0.1	0.8	1.6
0.2	0.0	1.6
0.2	0.8	1.6
0.2	1.6	1.6
0.3	0.8	1.6
0.4	0.0	1.6
0.4	0.8	1.6
0.4	1.6	1.6
0.4	0.8	0.0
0.4	0.8	0.8

Example: Cu-Ni-Mn

Irradiations

controlled single - combined variable IVAR irradiation (ϕ, ϕt,T) for properties, nano structures & mechanisms (also e-& p+ beams, other reactors) ≈ 3000 conditions



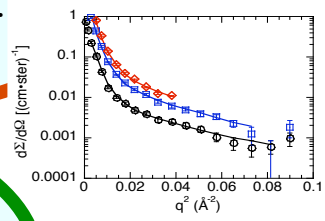
IVAR

Testing

automated small specimen hardness and tensile testing

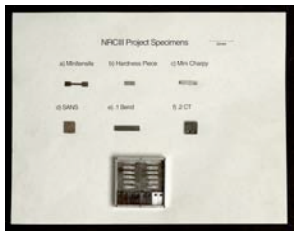
Characterization

complementary nano-characterization: TEM, SANS, ASAXS, 3DAP, PAS, RSC, PIA, .



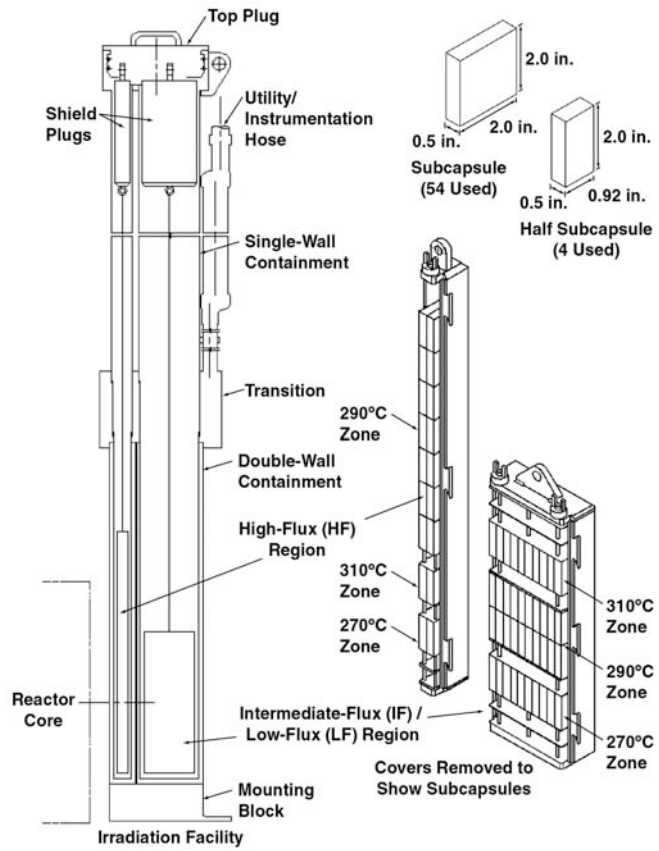
Specimens

small specimen fracture & testing & micromechanics characterization



IVAR

- IVAR was conceptually designed by UCSB and ORNL built & operated by ORNL at the U.Michigan Ford Research Reactor
- Irradiations at controlled temperatures (270 to 310°C) and ϕ (5×10^{10}-10^{12} n/cm^2-s) to over-lapping ranges of ϕt (0.004-4 $\times 10^{19}$)
- 54 interchangeable sub-capsules each containing a large number of small specimens
- Total of 80 irradiated capsules
- $T < \pm 5°C$ and $\phi t \approx \pm 7\%$



IVAR Objective - Approach

- Map with *'high'* accuracy and resolution the combined effects of embrittlement variables: T, ϕ, ϕt, Cu, Ni, Mn, P,... product form, heat treatment... -- small specimen tests and microstructural characterization
- Study mechanisms and phenomena like late blooming MNP phases as well as deformation and fracture micromechanics
- Split melt model alloys/steels plus commercial & program steels
- Multiple-purpose & types of specimens provide various mechanical properties and micro(nano)structural database
- Tensile matrix total of 2050 material-irradiation condition matrix points with ≈ 1800 evaluated to date - providing new and unprecedented insight on embrittlement - including combined effects of variables and basic mechanisms

Alloys

- CM /L series RPV steels 31 compositions + large HT matrix
- Model alloys - 40
- Commercial and program alloys CW- series 17
- BE, KAERI and CRIEPI alloys
- Example

CM-series

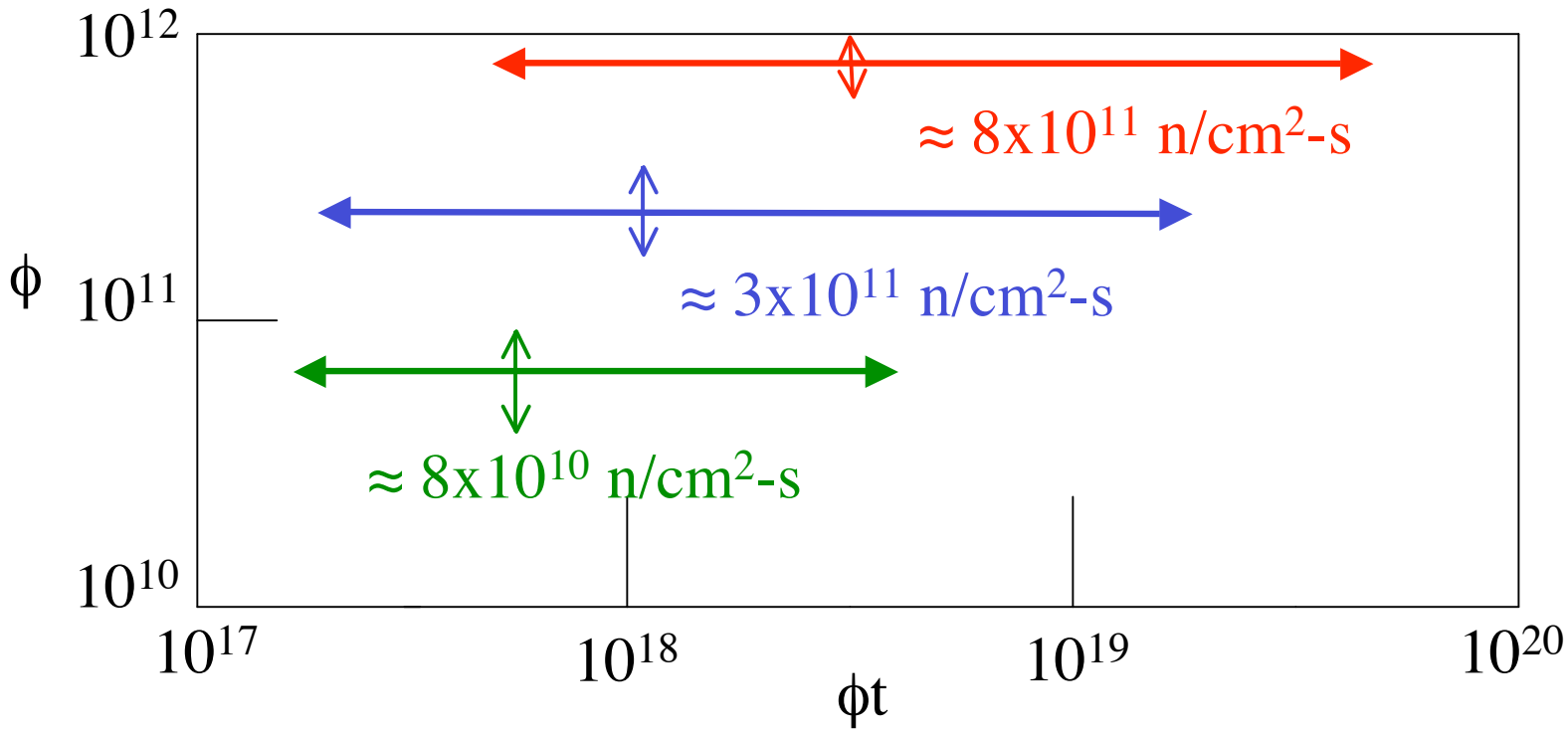
Cu	Ni	Mn
0.0	0.0	1.6
0.0	0.8	1.6
0.0	1.6	1.6
0.0	0.8	0.0
0.05	0.8	1.6
0.1	0.8	1.6
0.2	0.0	1.6
0.2	0.8	1.6
0.2	1.6	1.6
0.3	0.8	1.6
0.4	0.0	1.6
0.4	0.8	1.6
0.4	1.6	1.6
0.4	0.8	0.0
0.4	0.8	0.8

CW-series

Alloy	C	Mn	P	S	Si	Cr	Ni	Mo	Cu	V	HT*
AW	0.08	1.69	0.014	0.013	0.45	0.14	0.63	0.40	0.21	--	1
BW	0.09	1.63	0.018	0.009	0.54	0.10	0.69	0.40	0.28	--	2
CW	0.08	1.30	0.009	0.010	0.37	0.08	0.62	0.31	0.06	--	3
62W	0.08	1.61	0.020	0.007	0.59	0.12	0.60	0.39	0.23	0.01	4
63W	0.10	1.65	0.016	0.011	0.63	0.10	0.69	0.43	0.30	0.01	5
65W	0.08	1.45	0.015	0.015	0.48	0.09	0.60	0.39	0.22	0.006	6
67W	0.08	1.44	0.011	0.012	0.50	0.09	0.69	0.39	0.27	0.007	7
73W	0.10	1.56	0.005	0.005	0.45	0.25	0.60	0.58	0.31	--	8
Midland W	0.08	1.61	0.017	0.007	0.62	0.10	0.57	0.41	0.27	0.004	9
HSST02 P	0.23	1.55	0.009	0.014	0.20	0.04	0.67	0.53	0.14	0.003	10
A302B P	TBD										11
A508 P	TBD										12
JRQ P	0.18	1.40	0.019	0.004	0.25	0.12	0.82	0.50	0.14	0.003	13
WP W	0.06	1.43	0.011	0.005	0.50	0.05	1.65	0.39	0.04		14
WG W	0.04	1.21	0.008	0.007	0.60	0.09	1.71	0.35	0.24		14
WV W	0.04	1.36	0.010	0.010	0.38	0.05	1.66	0.41	0.56		14
EPRI C W	0.16	1.55	0.005	0.009	0.17	0.04	0.60	0.44	0.35		15

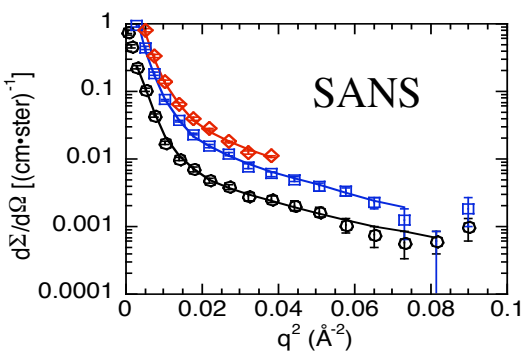
IVAR Irradiation Variables

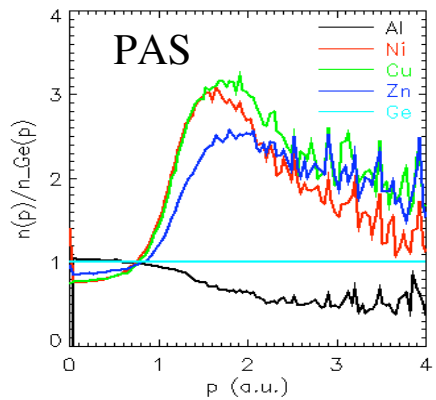
- Overlapping ϕt in three ϕ ranges at 270, 290 & 310 C
- Focus on tensile and SANS matrix
- Other studies include: a) extensive evaluation of heat treatment & Cu pre-precipitation effects; b) effects of 270 to 290°C and 290 to 270°C temperature changes; c) and an extensive fracture mechanics/micromechanics matrix



Specimens and PI Characterization

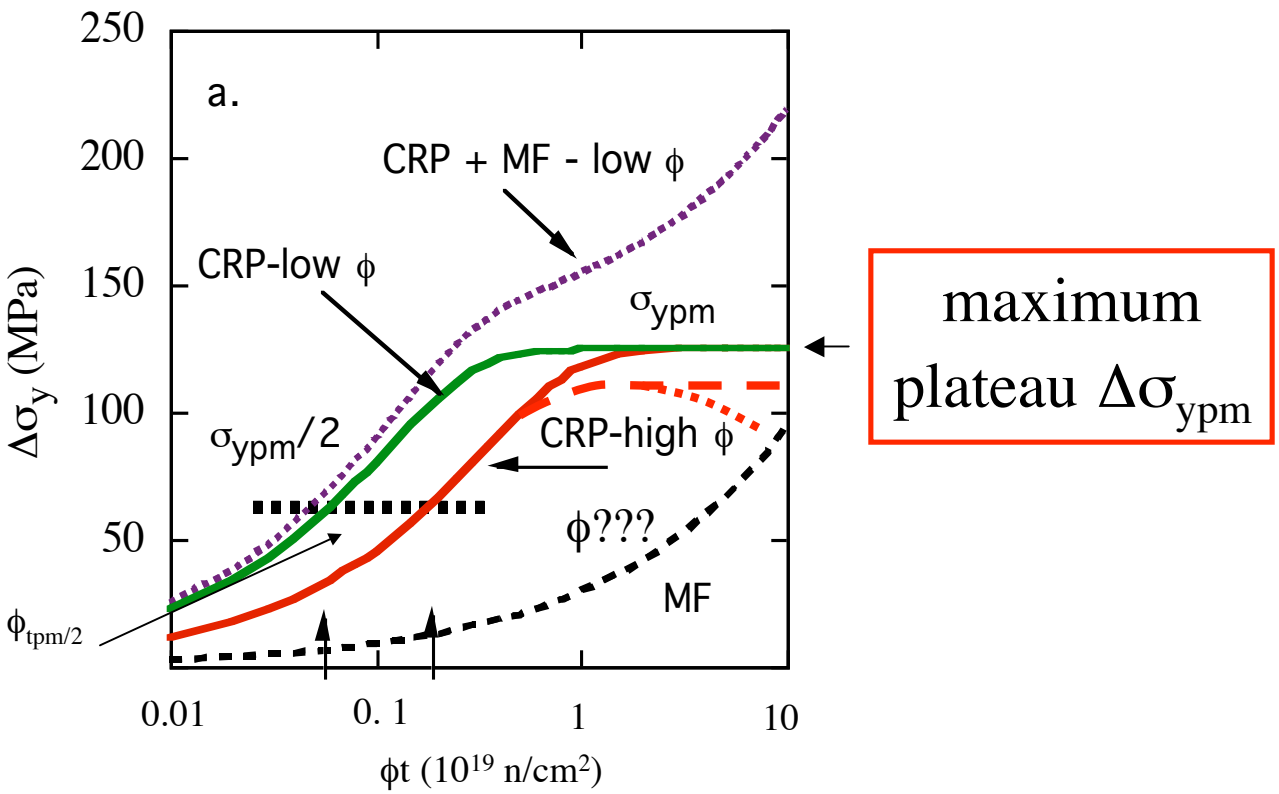
- Subsized tensile semi-automated testing with minimum redundancy of 2 - $\Delta\sigma_y$ to ±15MPa (average varies with alloy & capsule) and rich deformation database
- Tensile specimens also used for combined resistivity-Seebeck coefficient (RSC) measurements to track precipitation by evolution of Cu, Ni and Mn in solution
- Extensive use of microhardness
- Comprehensive SANS coupon matrix also used for PAS and sliced for APT wires





Flux Effects - Framework

- Hardening due to copper rich precipitates (CRP) and matrix features (MF): $\Delta\sigma_y = \Delta\sigma_{yp} + \Delta\sigma_{mf}$
- CRP -- $\phi t_{pm/2}$ @ $\Delta\sigma_{ypm}/2$



0.4% Cu, 1.3% Ni Plate

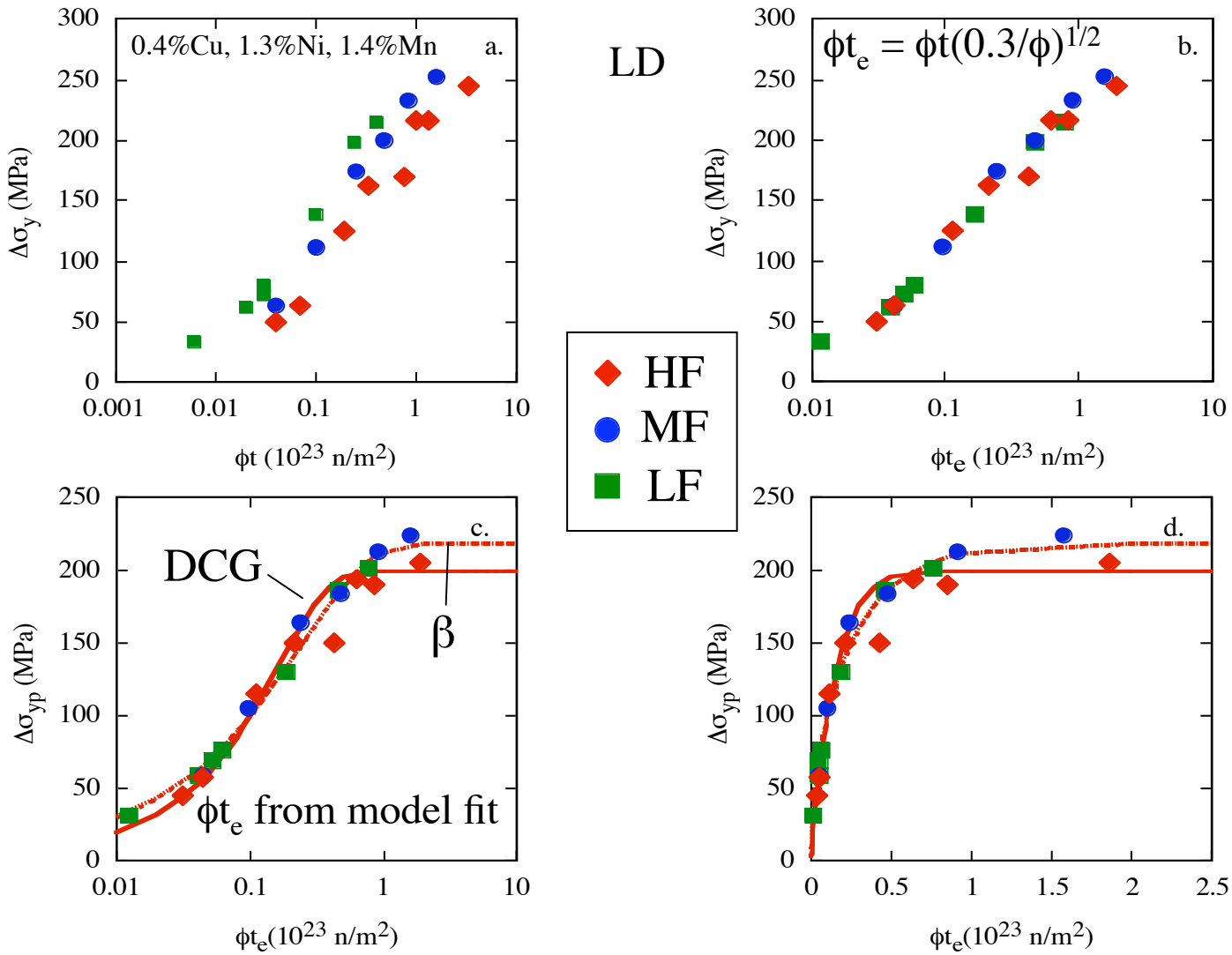
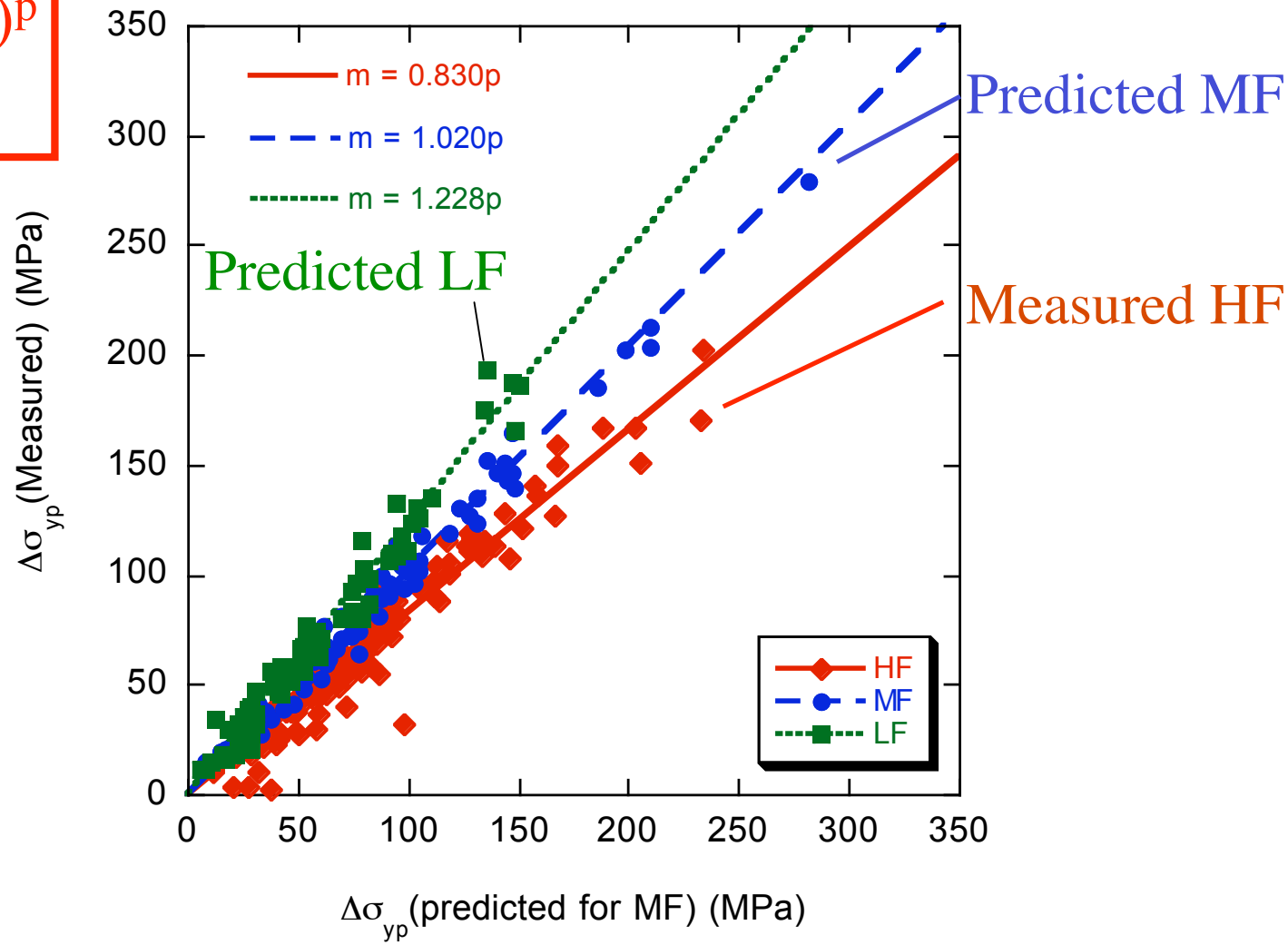


Figure A15 LD

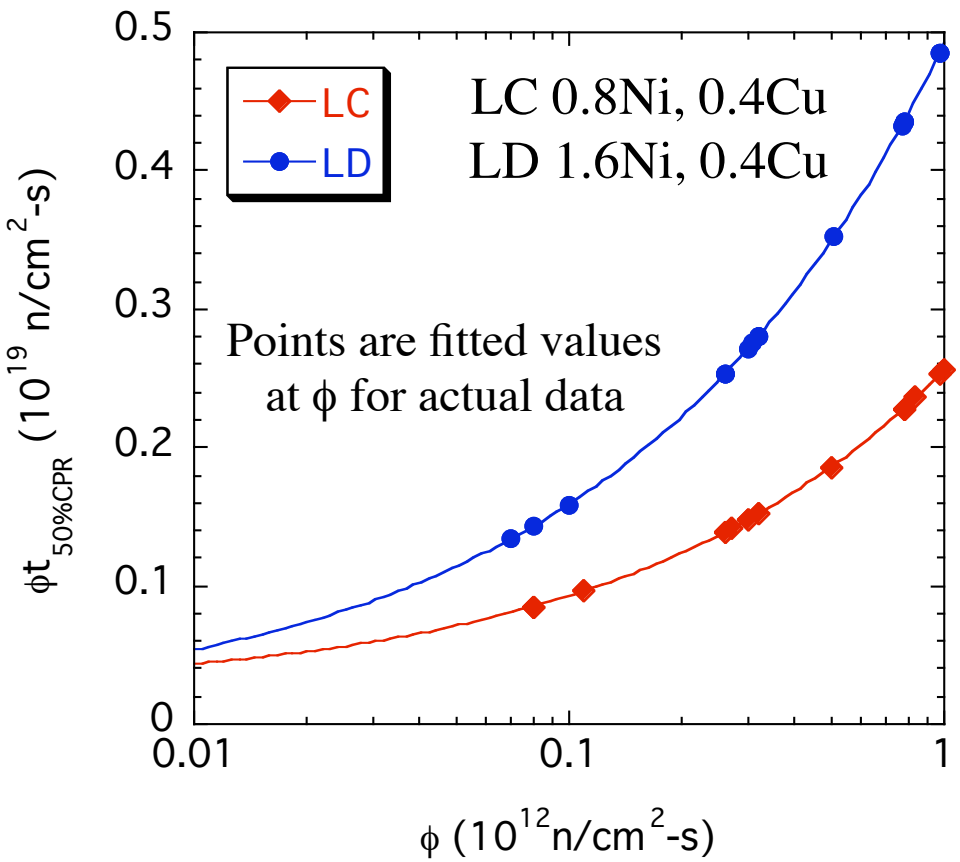
Measured vs Predicted w/o ϕ-Effect

$$\phi t_e \approx \phi t(\phi r/\phi)^p$$
$$p_{av} \approx 0.4$$



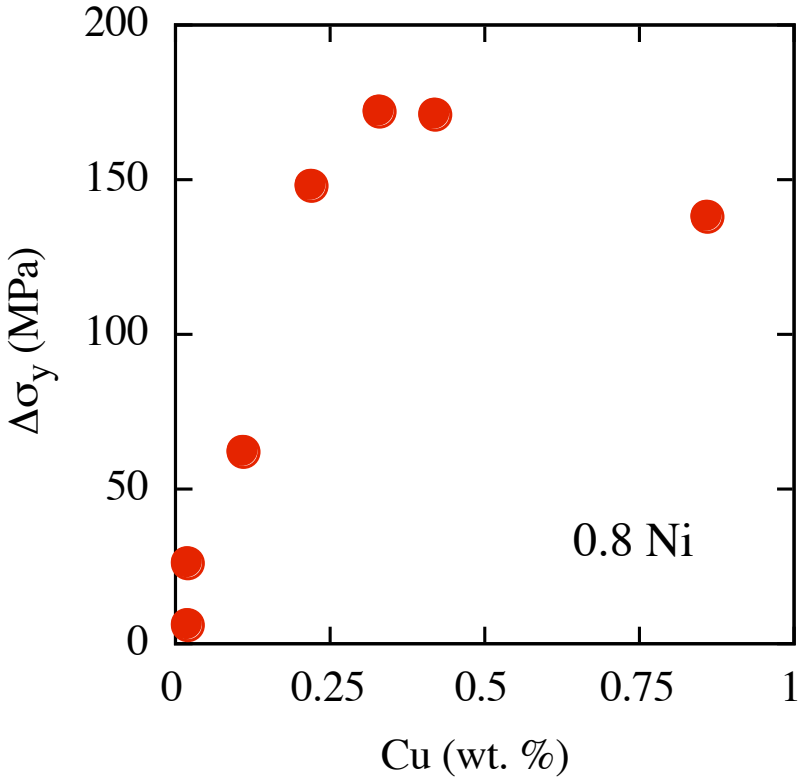
Mechanism Based Extrapolations

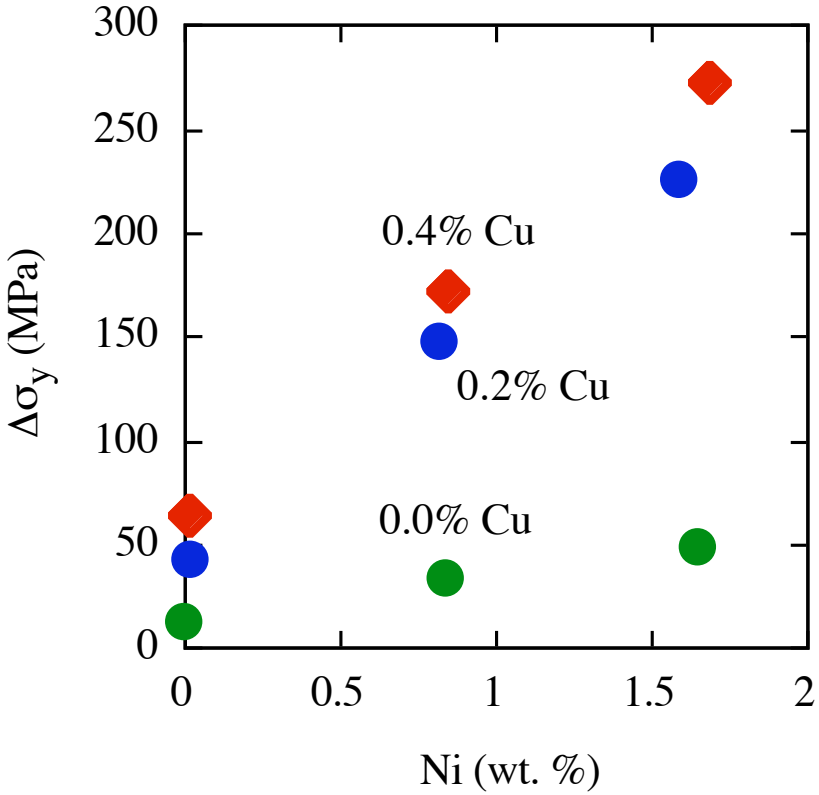
Known functional form for recombination allows physical extrapolation ϕ effects fits shown here as $\phi t_{pm/2}$ - depends on alloy and composition

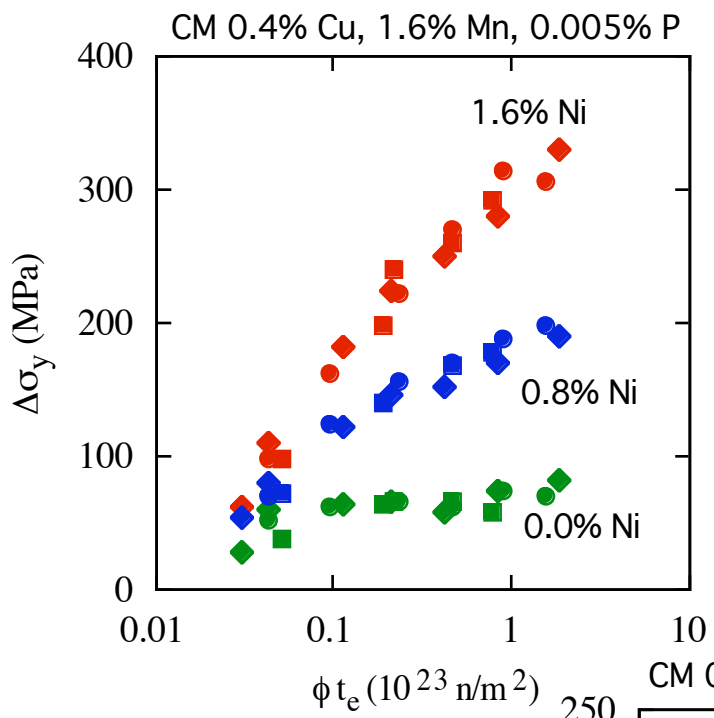


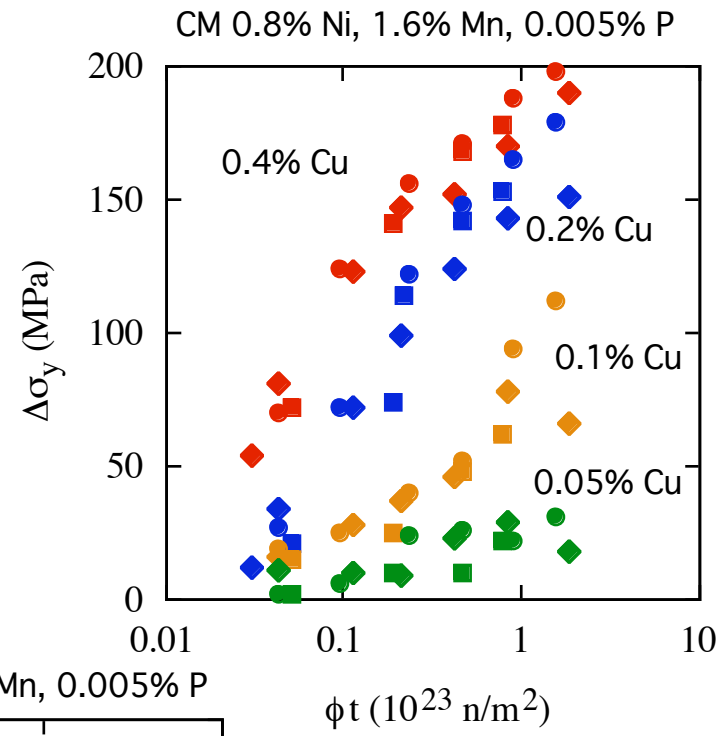
Composition

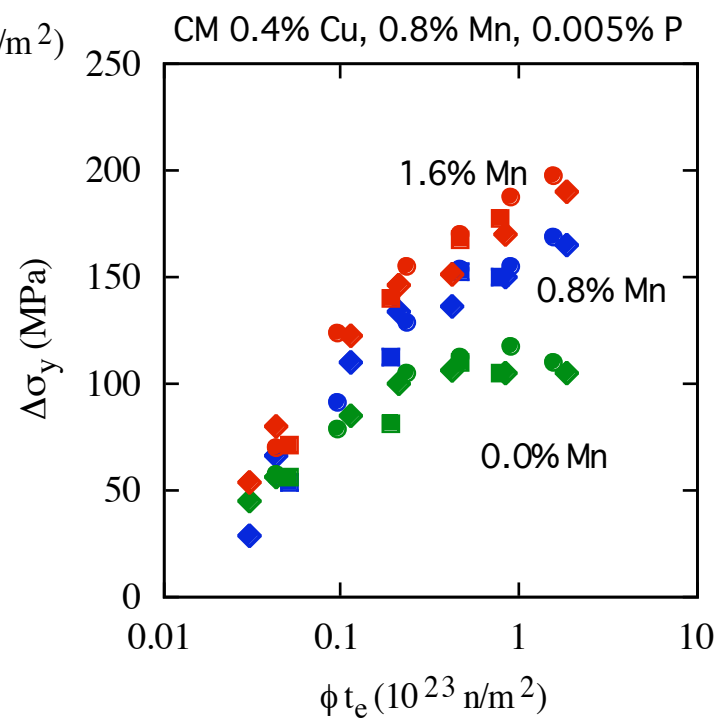
• *Supersaturated* Cu precipitates into *nanoscale* Cu-Ni-Mn coherent transition phases leading to $\Delta\sigma_y$ - hence - ΔT (T4 capsule)







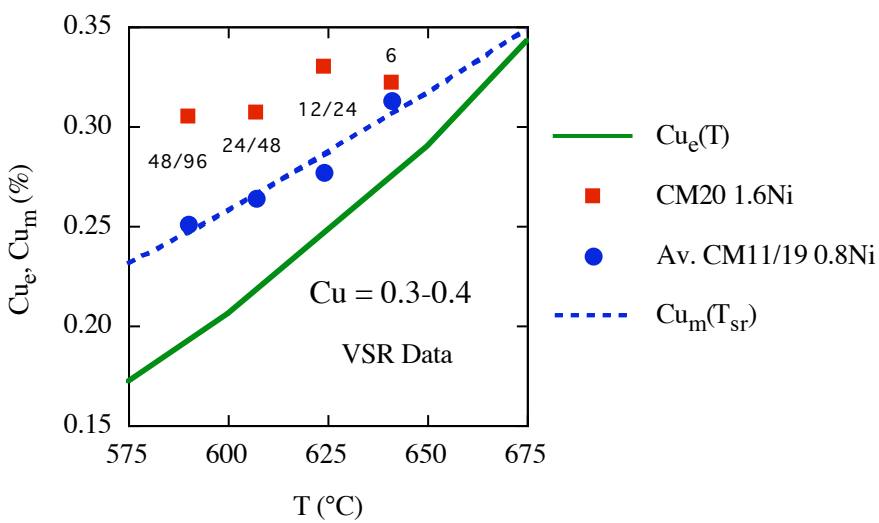


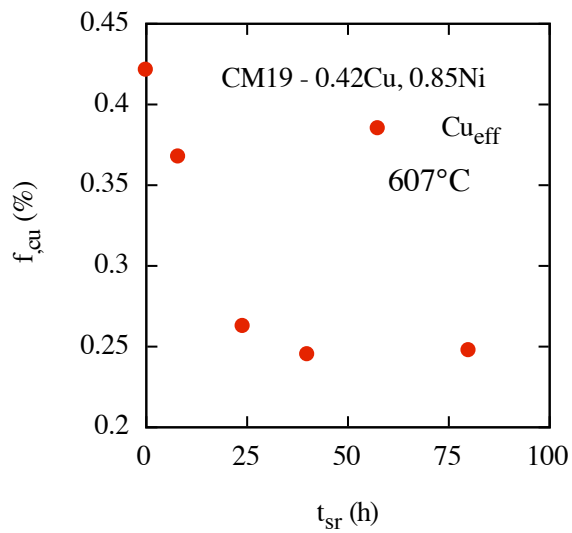


Composition Effects on $\Delta\sigma_y(\phi t_e)$

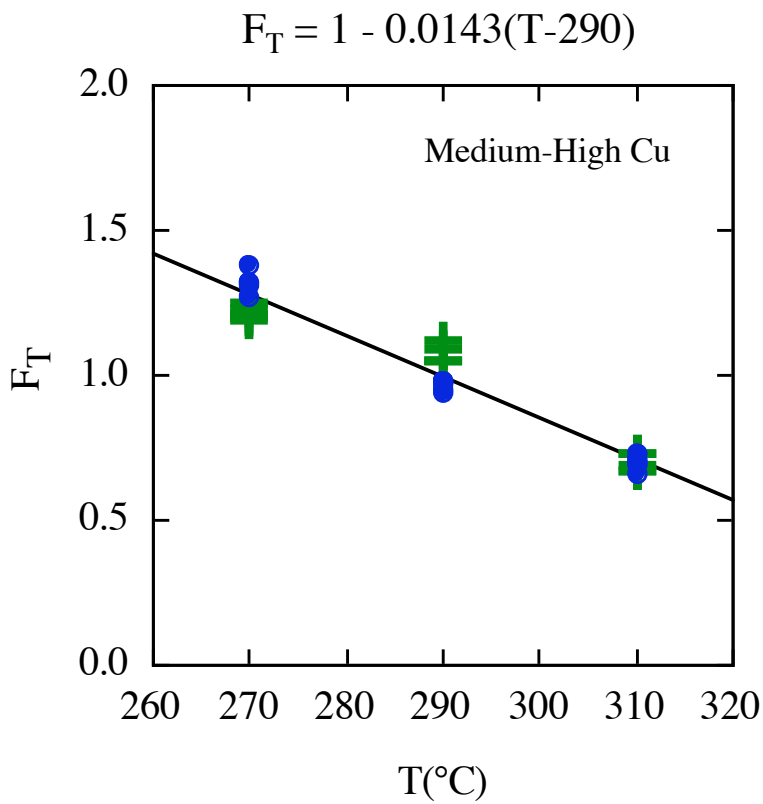
Heat Treatment

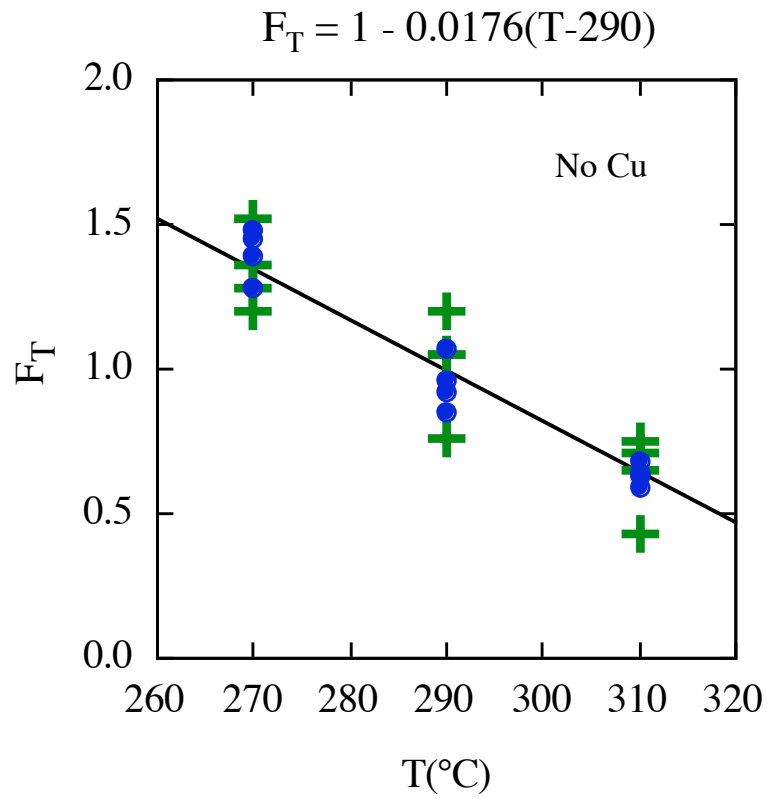
- Extensive study of HT T_{ht}-t_{ht} on $\Delta\sigma_y$, and the CRP/MNP microstructure and the 'effective' Cu in solution following various tempering and stress relief heat treatments -> Cu_{eff} or maximum Cu_m
- Developed $Cu_{eff}(T,t)$ model - high Ni increases Cu_{eff}



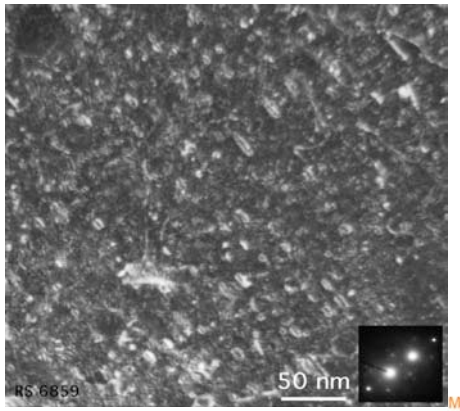


T- Dependence



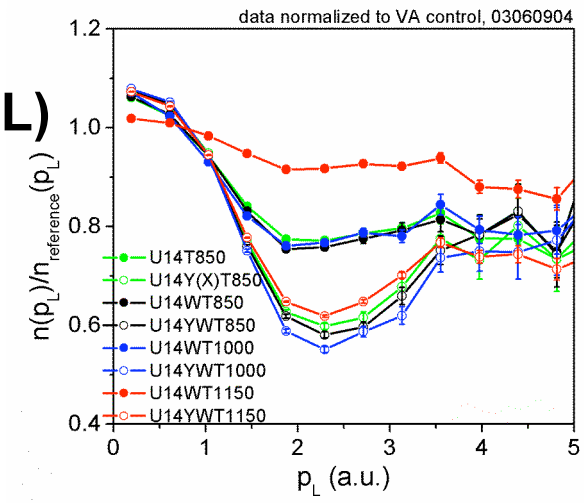


Characterization Techniques

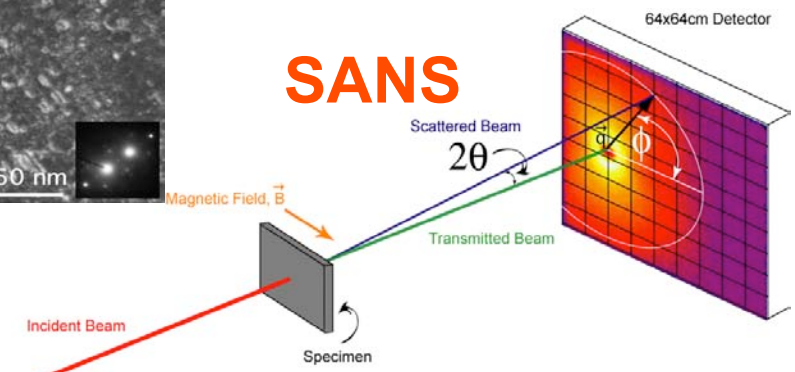


TEM

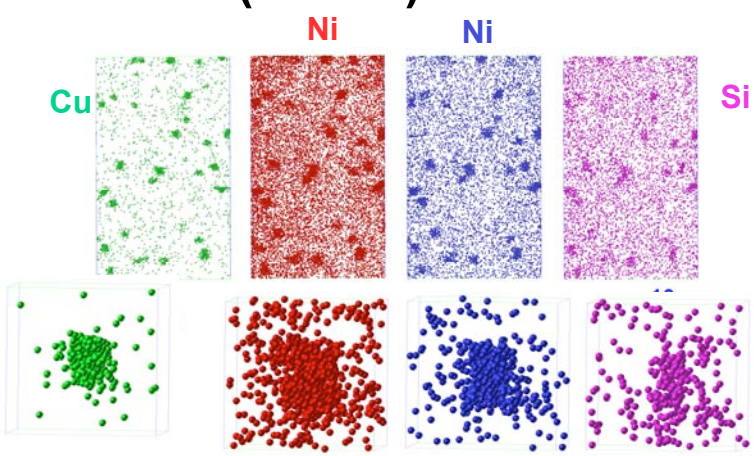
PAS (UCB/LLNL)



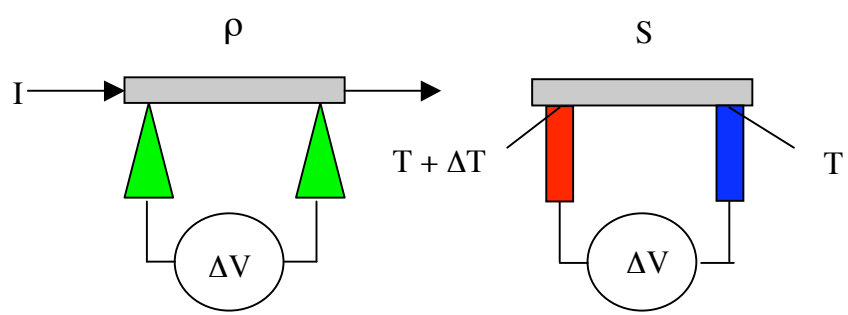
SANS



APT (ORNL)

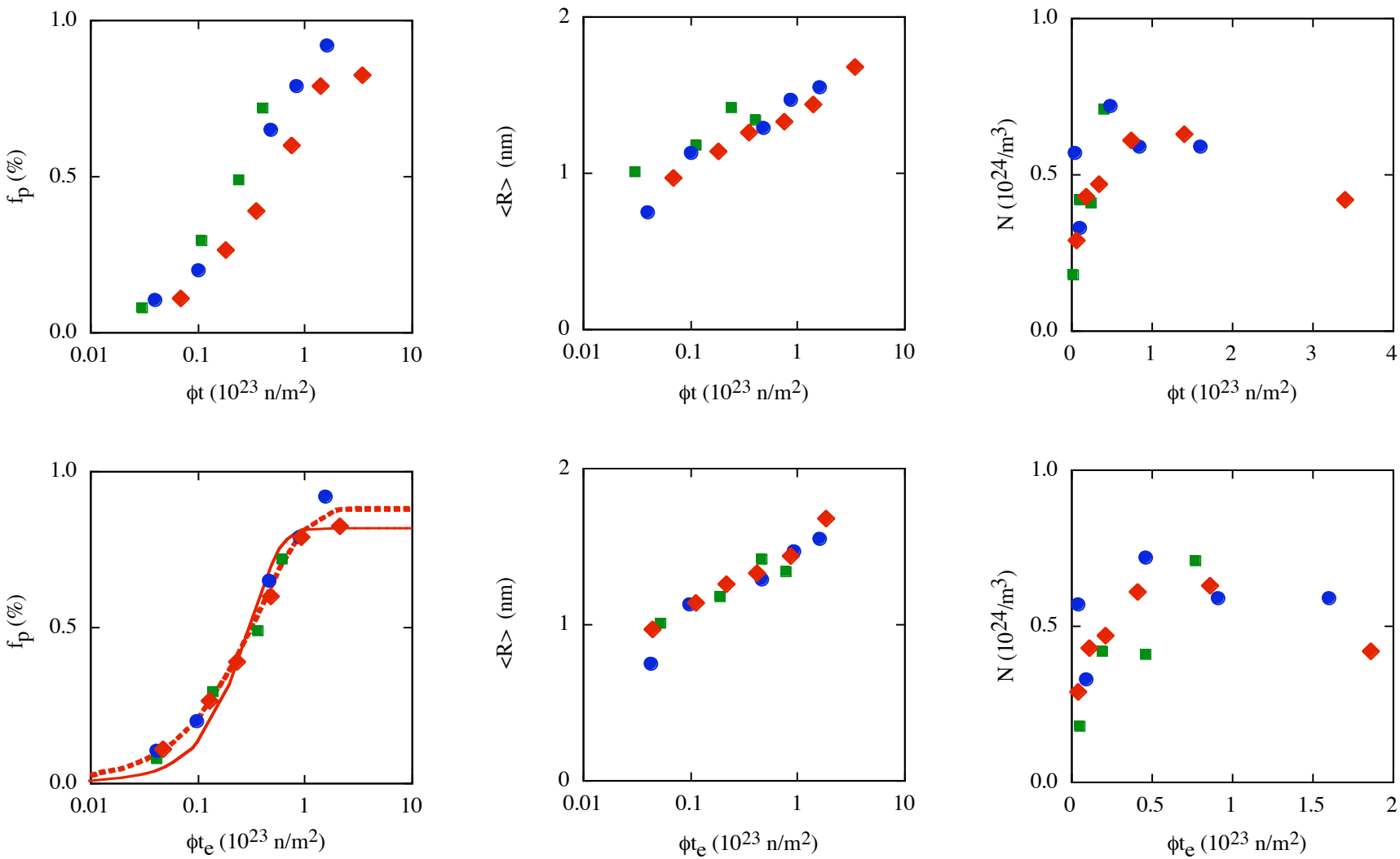


RSC

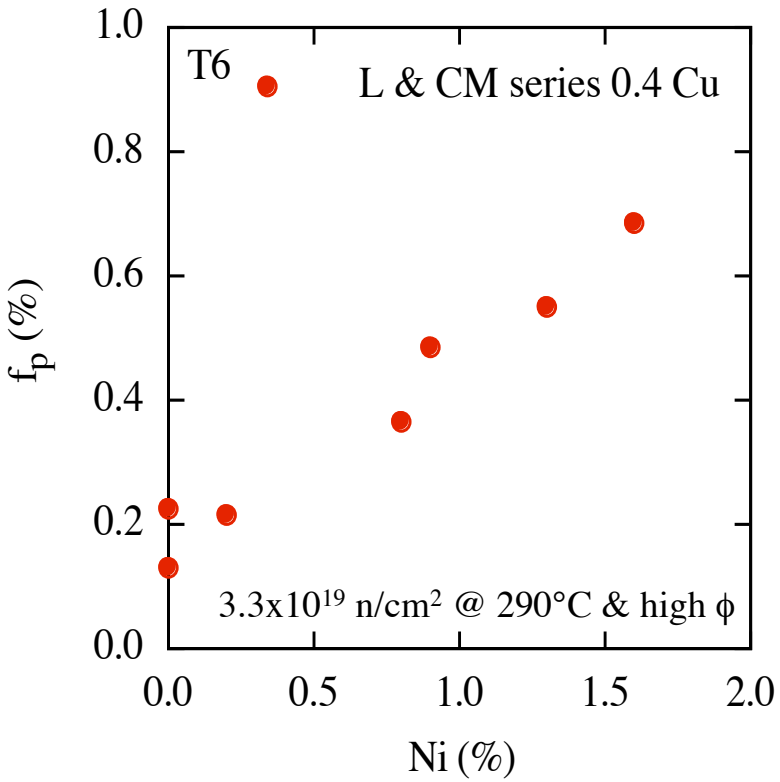


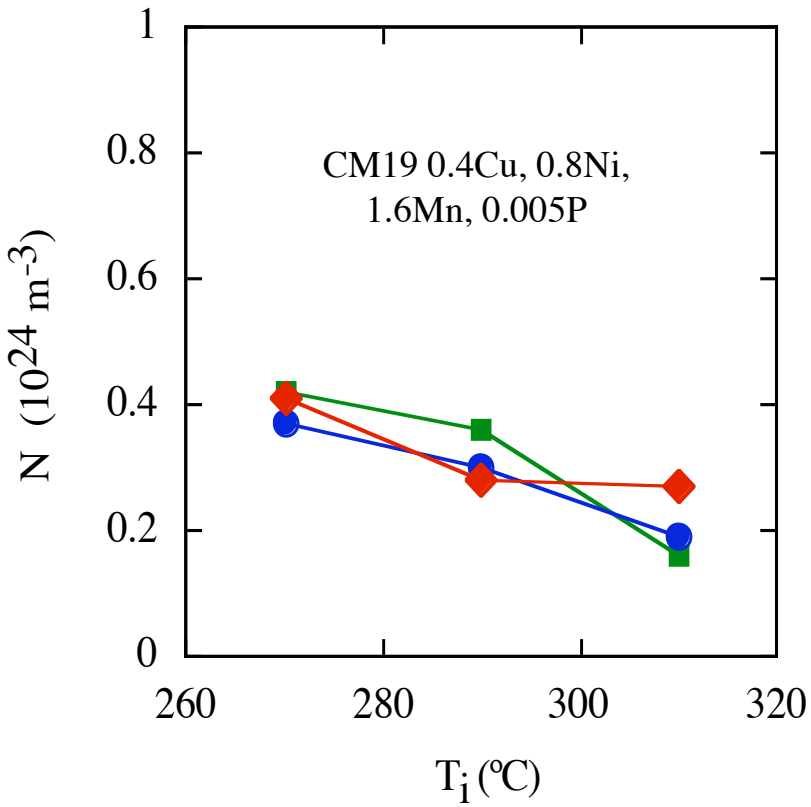
SANS:

CM20 0.4Cu, 1.6Ni, 1.6Mn, 0.005P



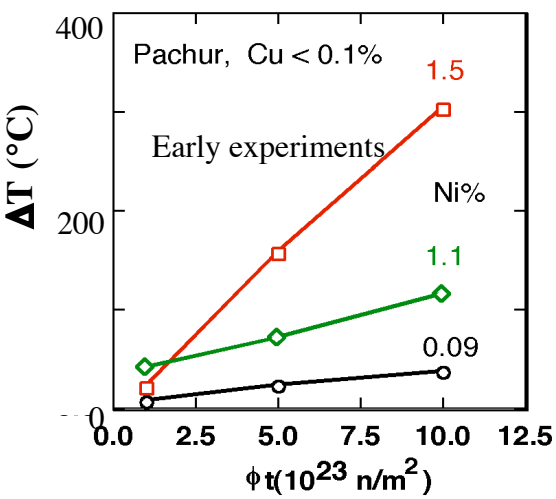
Composition and Temperature

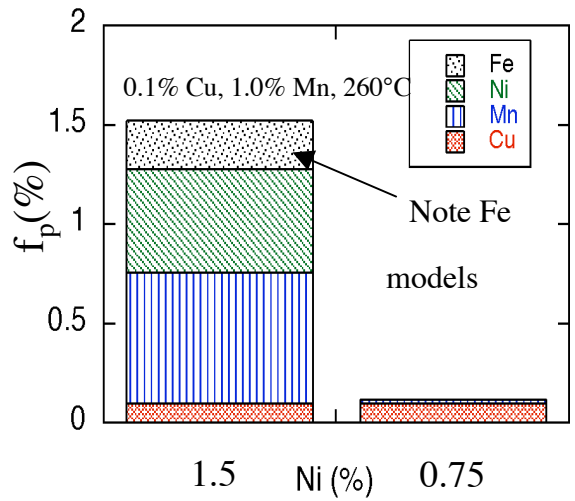


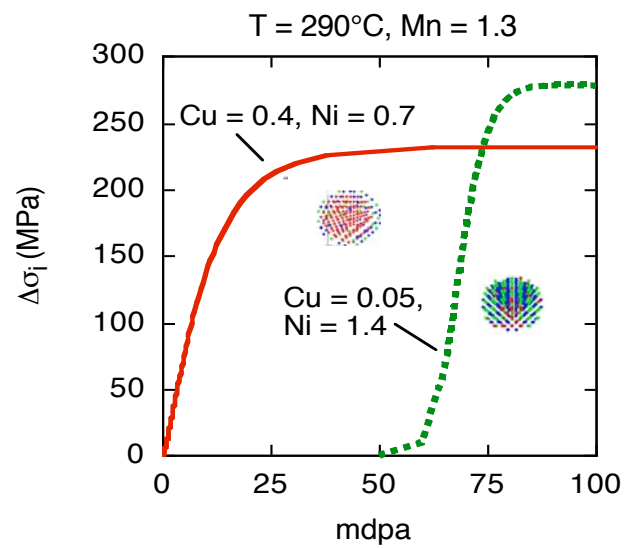


Late Blooming Phases?

- Model predicts *large volume fraction f_p of Mn-Ni(-Si-P) phases at low-no Cu -> ΔT ???*
- Cluster dynamics -> slow nucleation rates at low Cu - some Cu is a good catalyst - but how much needed???
- *Nucleation* is the rate limiting step
- *Late blooming phases (LBP)* grow rapidly at high ϕt???



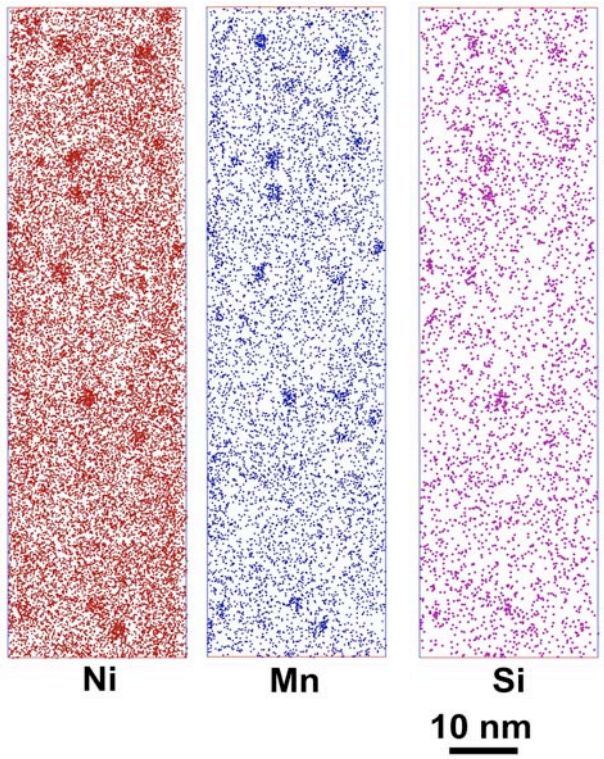


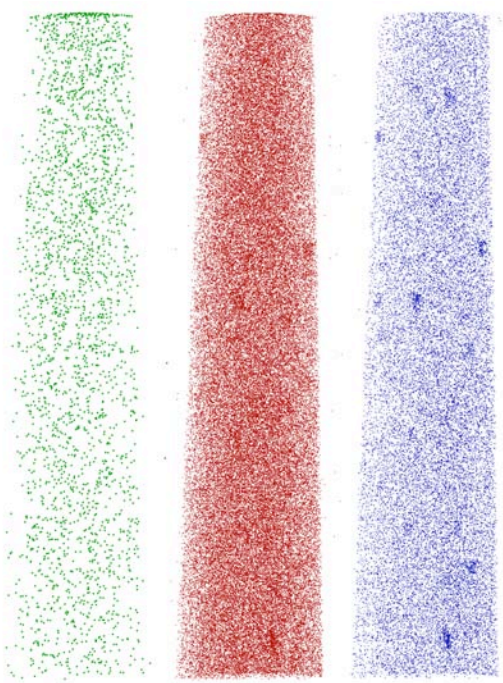


LBP

Mn-Ni-Si (Cu) precipitates found in CM6 and <u>all</u> low Cu and Cu free OV alloys as well as by SANS, PAS-OEMS and RSC

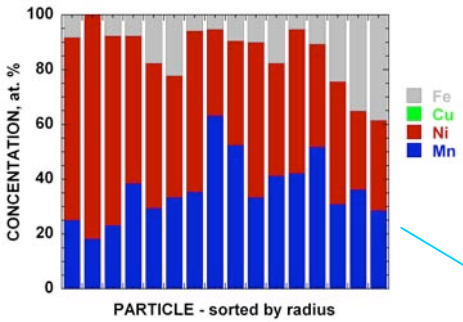
CM6

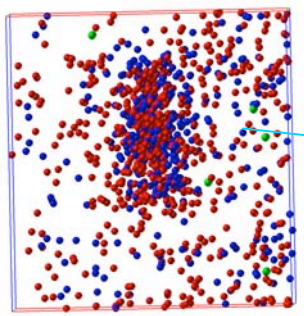


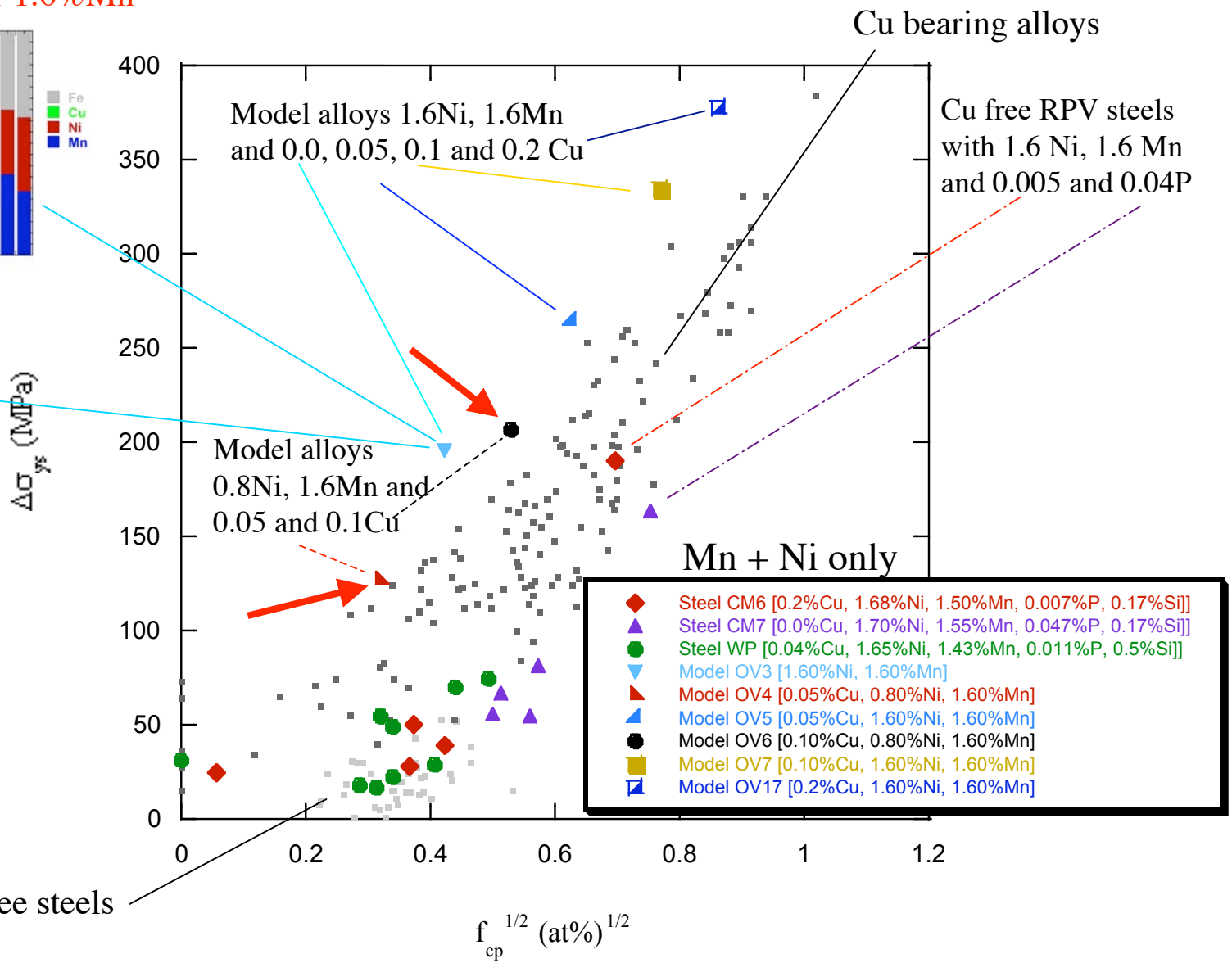


OV3

Fe-0% Cu-1.6% Ni-1.6%Mn







Other low, Cu-free steels

MD Trends in the IVAR Database: Preliminary Results

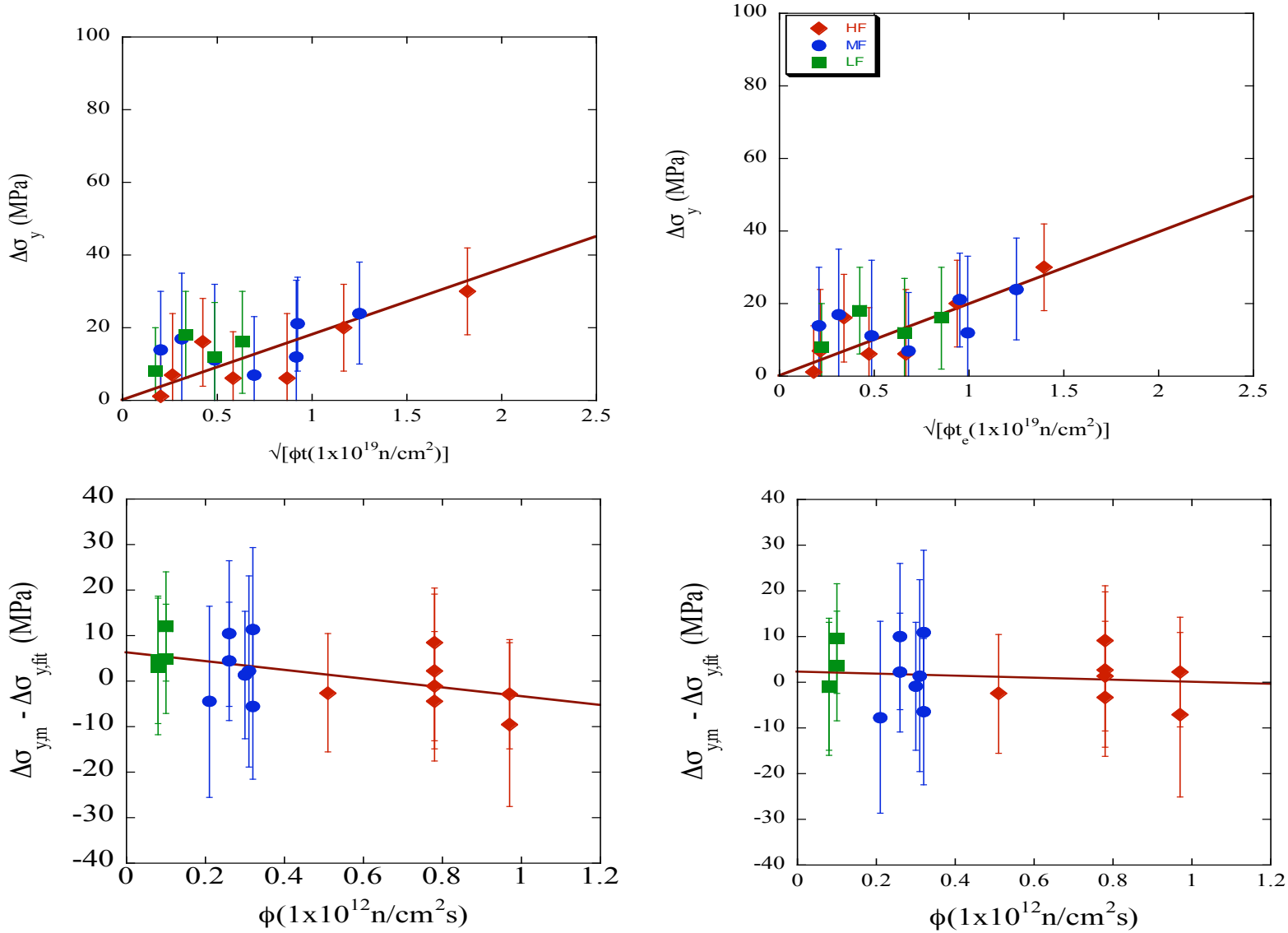
- Detailed assessment of low and Cu free IVAR data at 290°C
- Consistent trend observed that lower flux data shifted to lower fluence
- Use effective fluence $\phi t_e = \phi t(\phi t_r/\phi t)^p$ with fit parameter p
- Compare residual trends with ϕt_r verses ϕt
- Examples of

  - base compositions (≈ 0.8Ni, 1.6Mn, 0.005P)

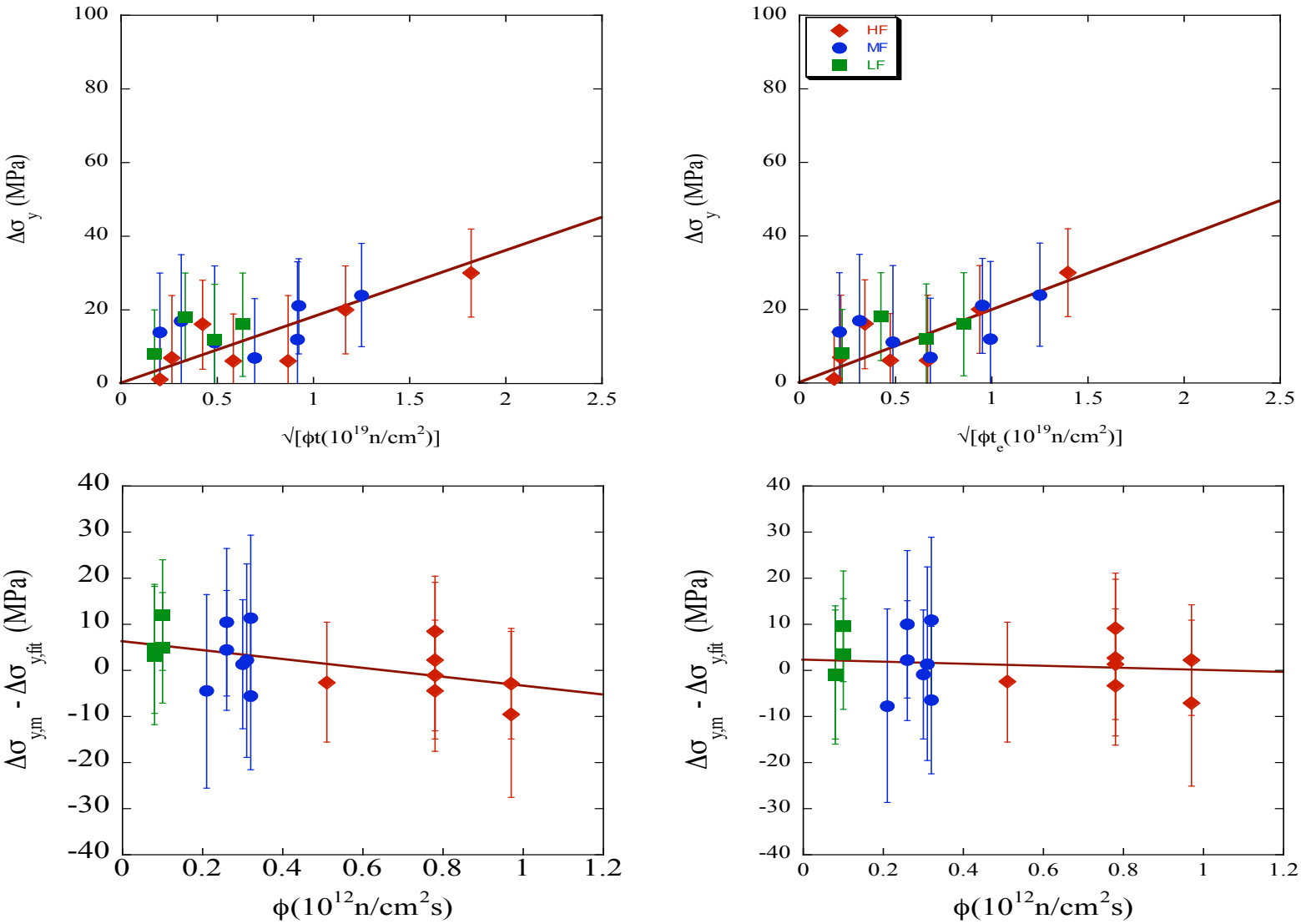
  -high sensitivity alloys (1.6Ni, 0.0025 & 0.040P, 0.1Cu)

  - low sensitivity alloys (0.0Ni, 0.0 &0).8Mn
- Cross plot slopes - interactions observed but not shown

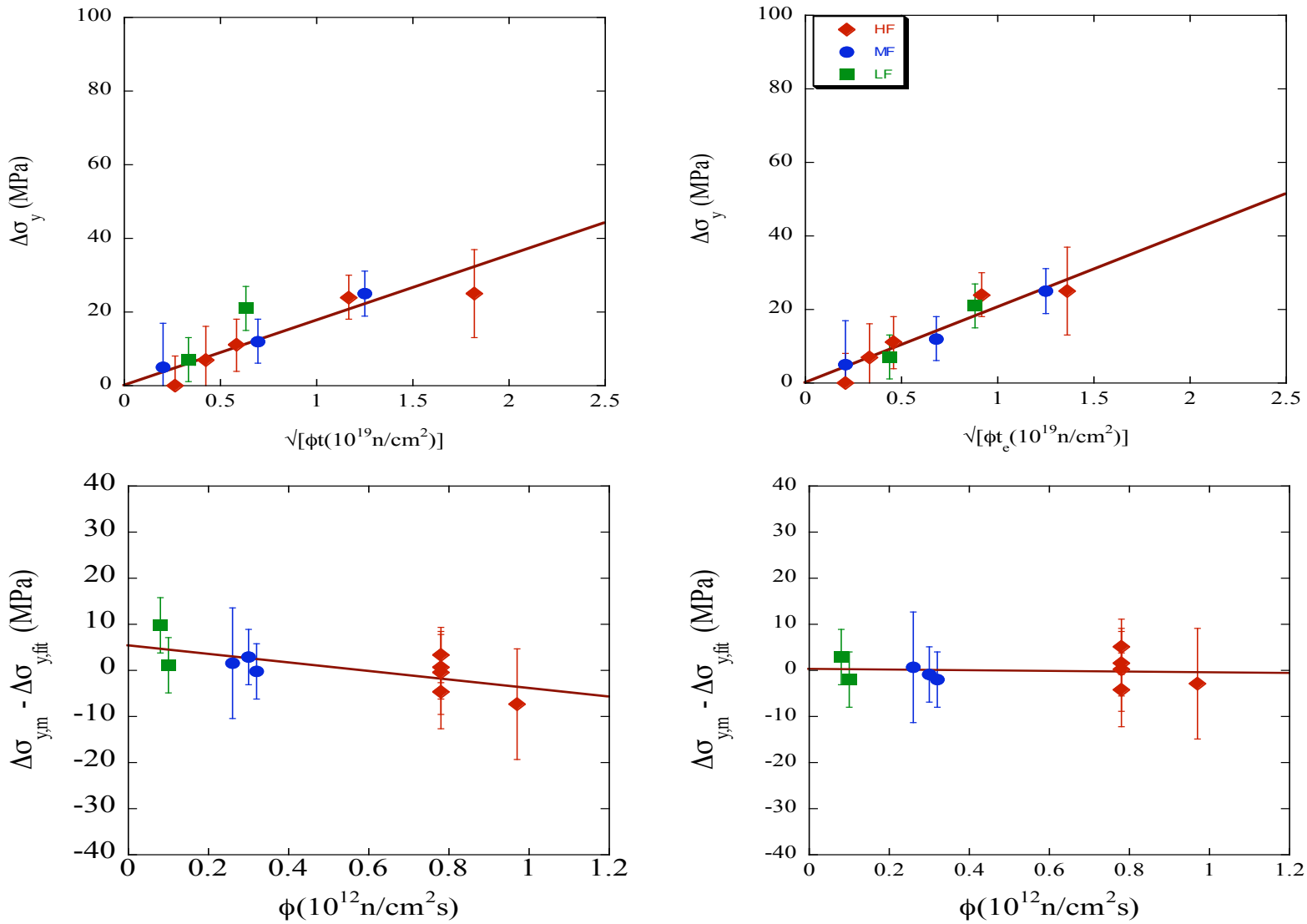
Cu Free Baseline 1



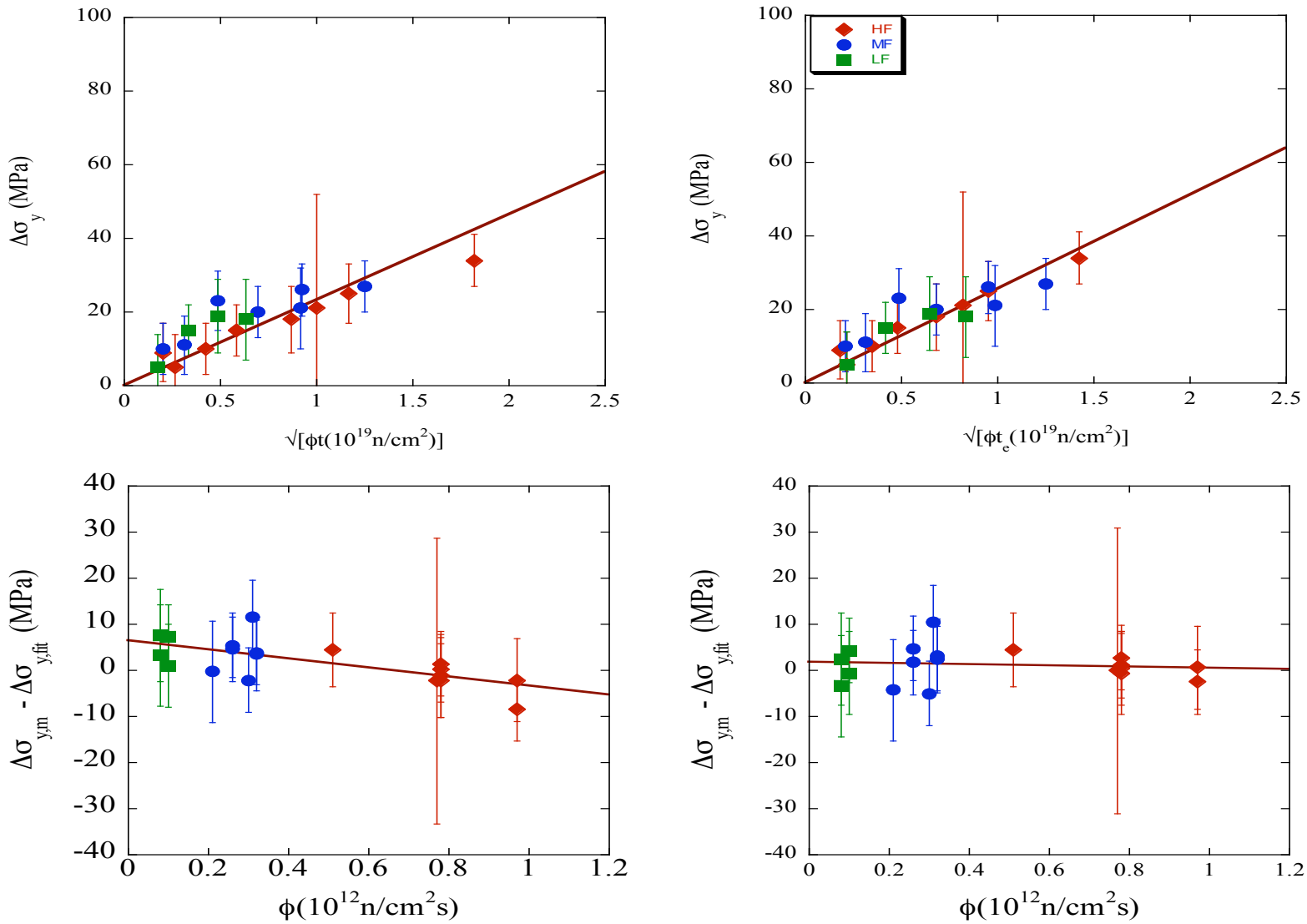
Cu Free Baseline 1



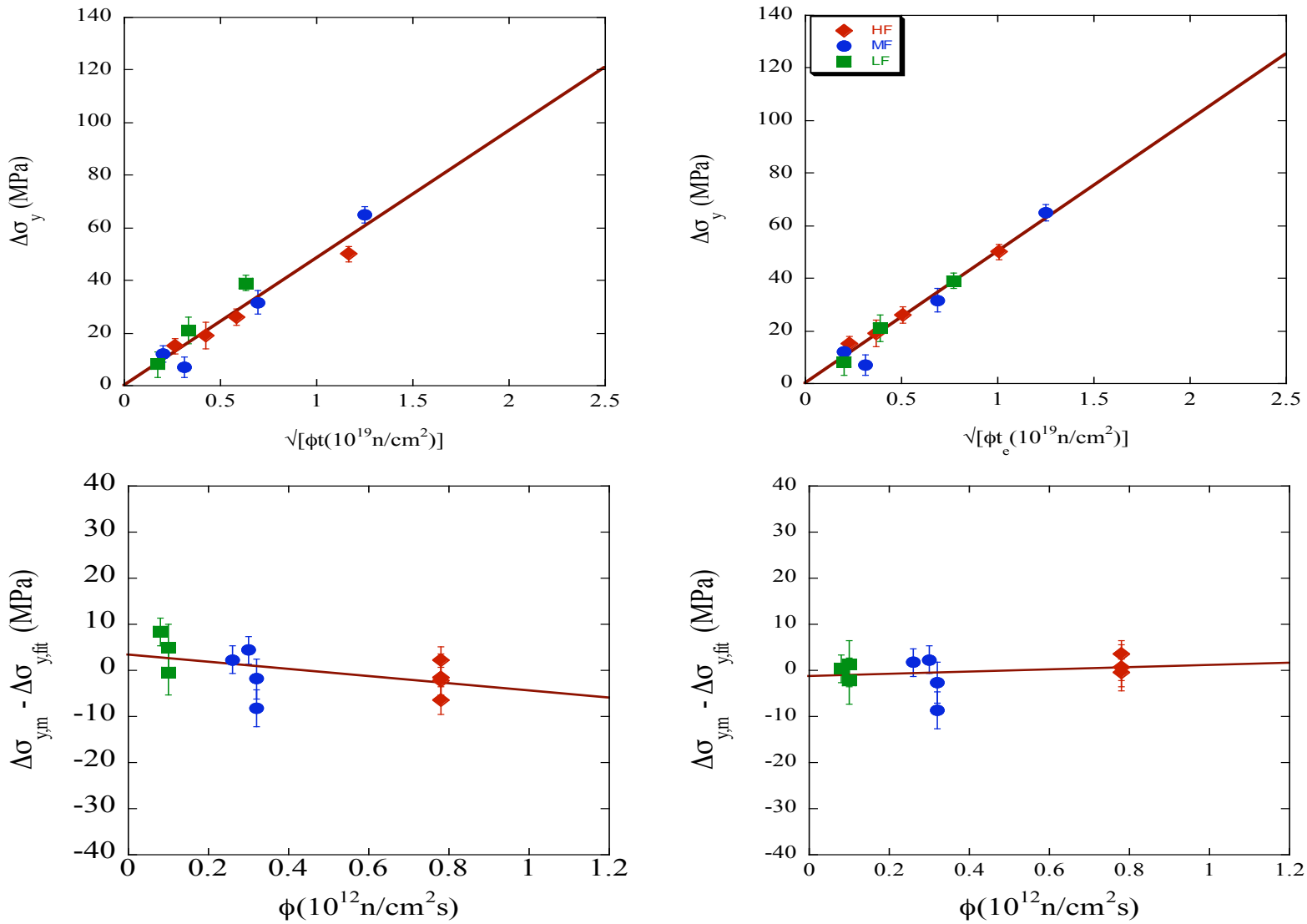
Cu Free Baseline 2



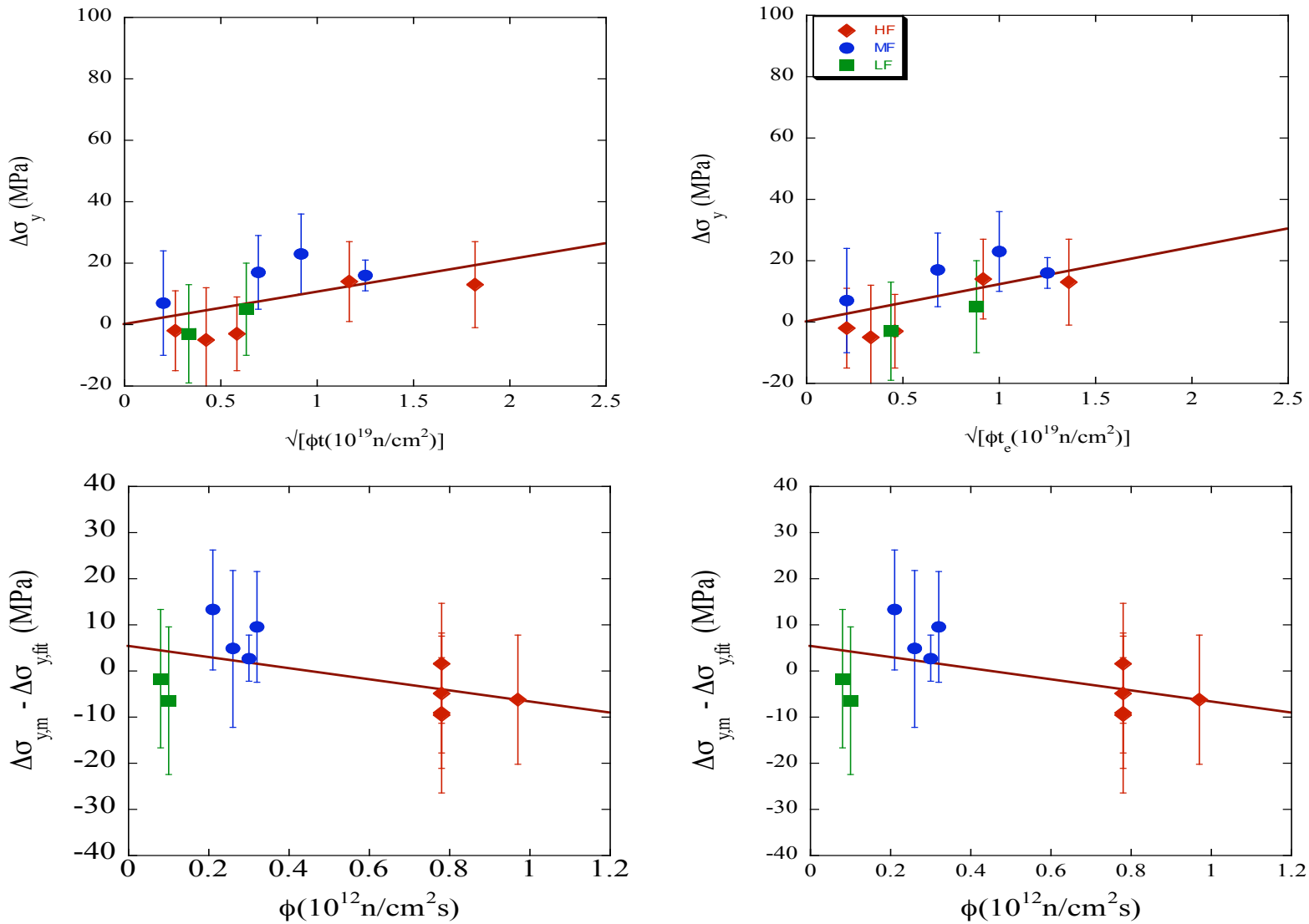
Cu Free Baseline 3



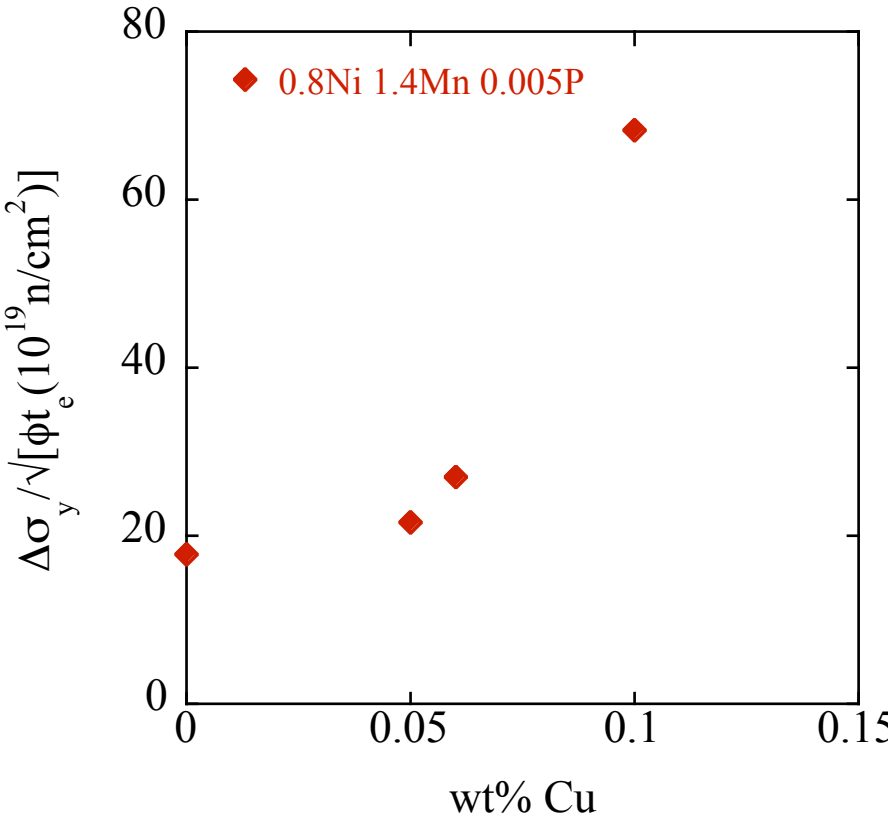
High Sensitivity 1

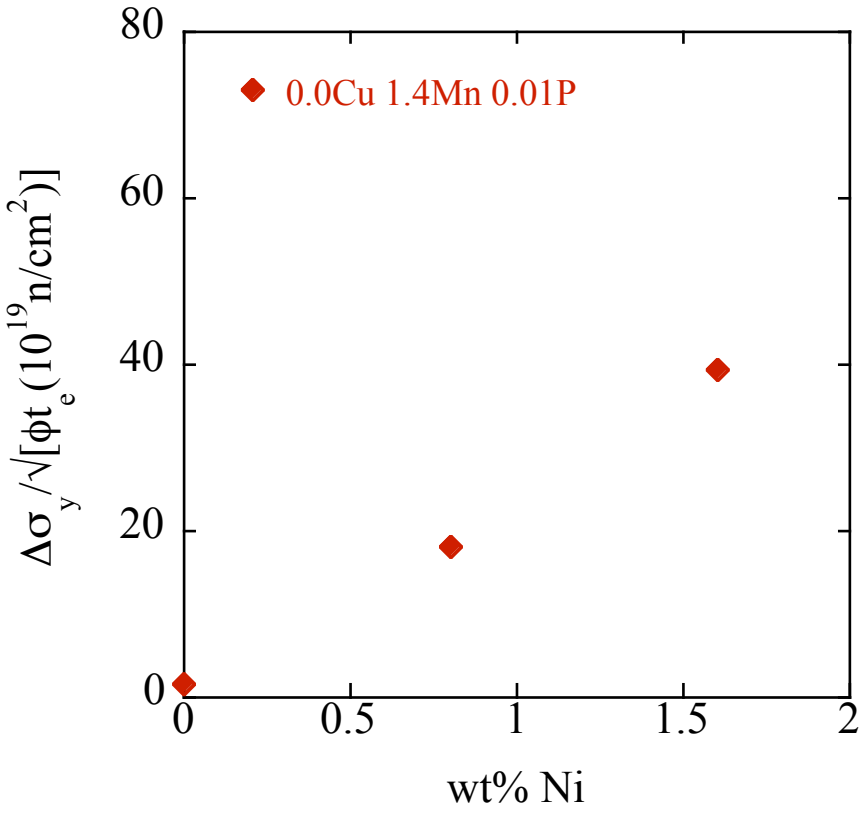


Low Sensitivity 1

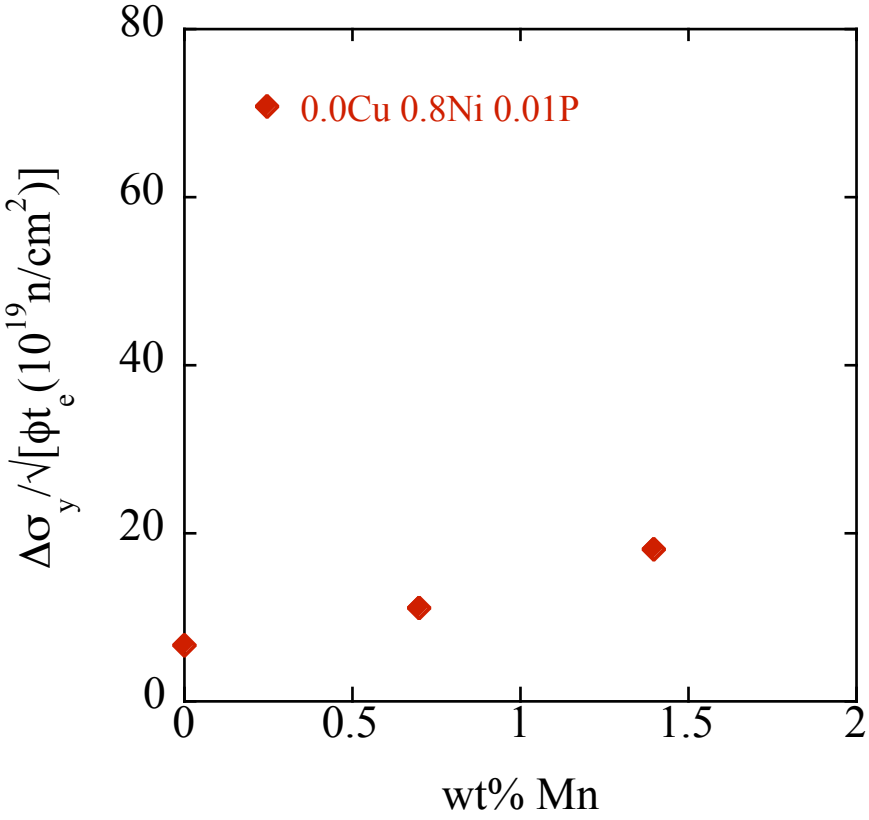


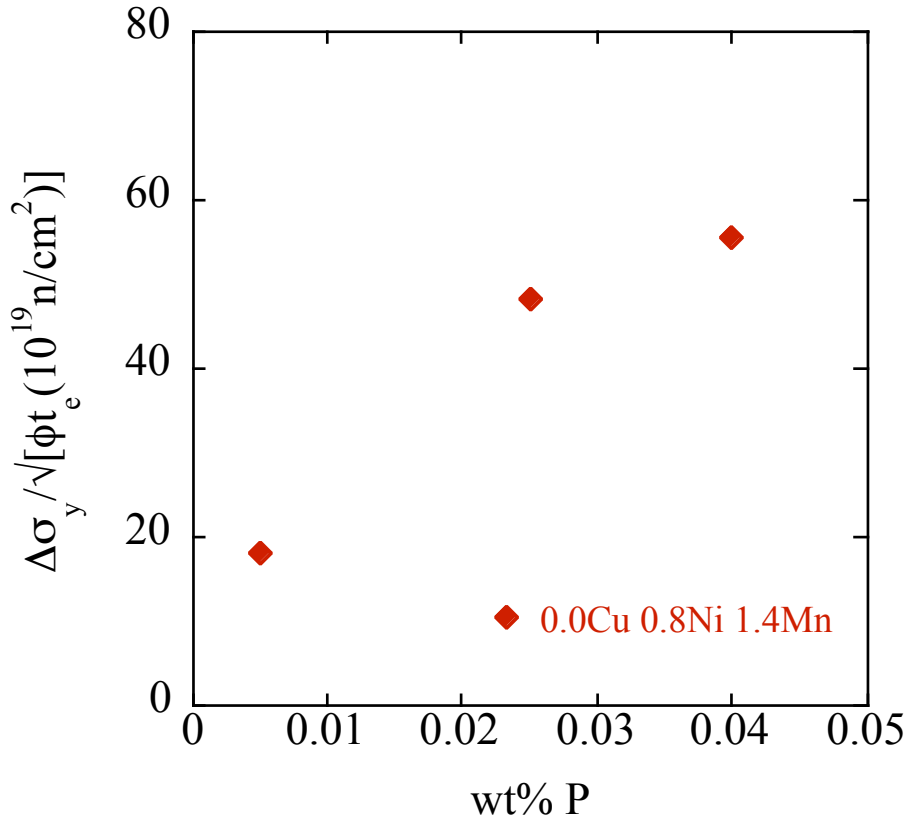
Cu and Ni Effects



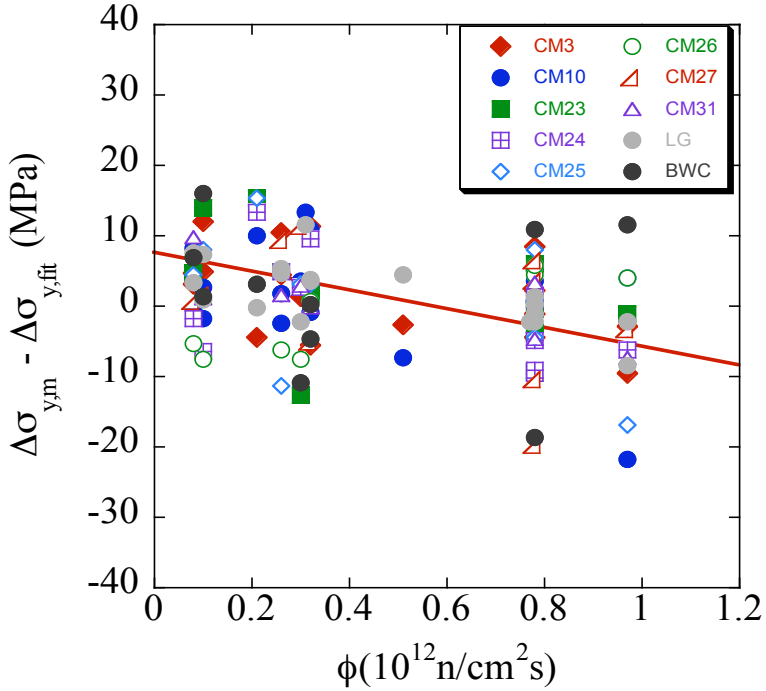


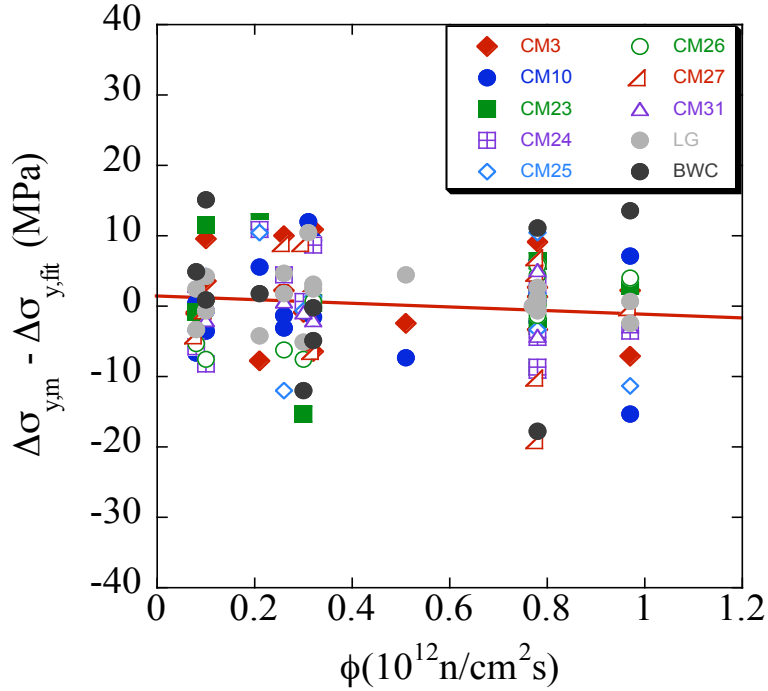
Slope Mn


80
60
40
20
0
0
0.5
1
1.5
2
0.0Cu 0.8Ni 0.01P
$\Delta\sigma_y/\sqrt{[\phi t_e(10^{19}n/cm^2)]}$
wt% Mn

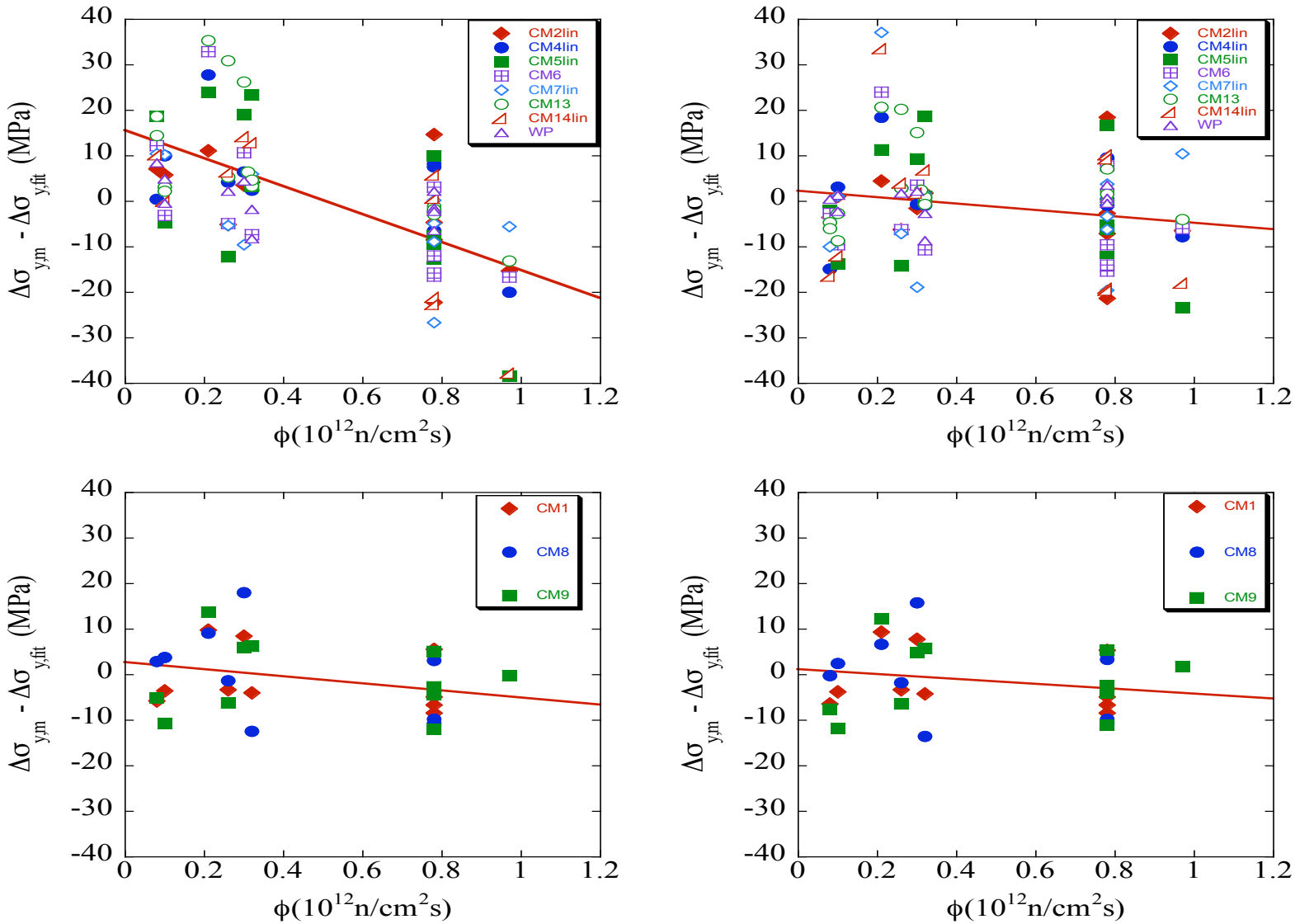

80
60
40
20
0
0
0.01
0.02
0.03
0.04
0.05
0.0Cu 0.8Ni 1.4Mn
$\Delta\sigma_y/\sqrt{[\phi t_e(10^{19}n/cm^2)]}$
wt% P

Baseline Residuals

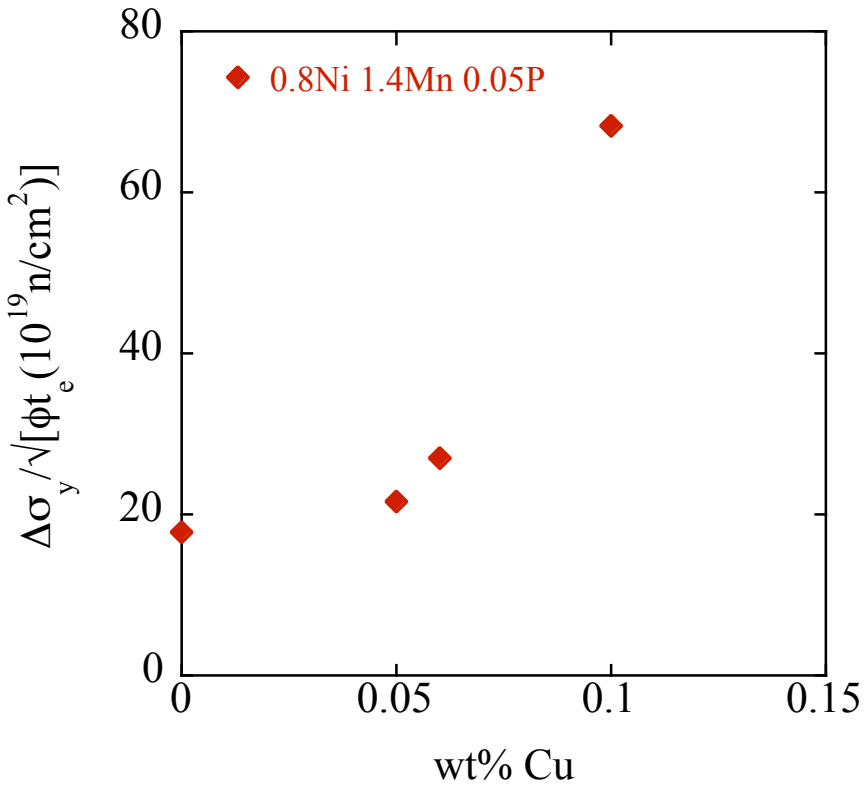


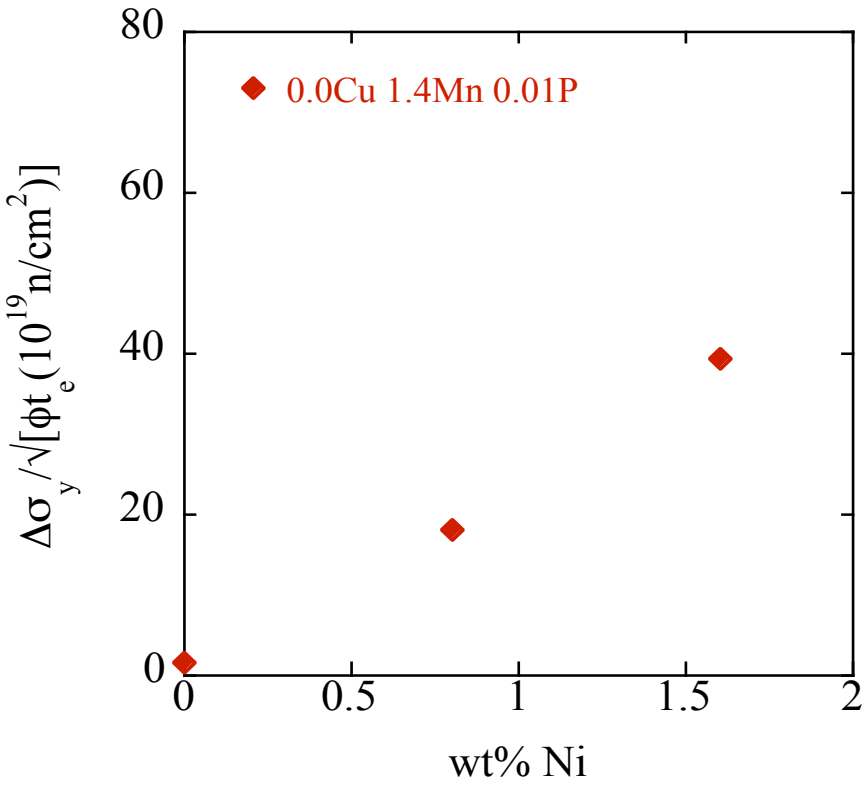


High and Low Sensitivity Residuals

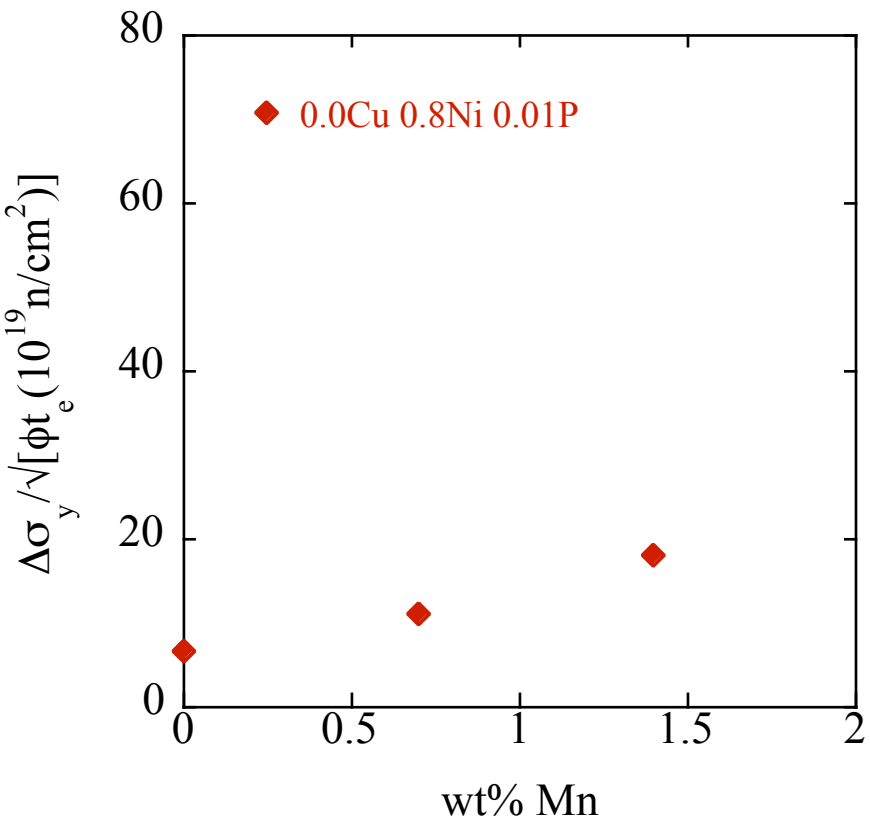


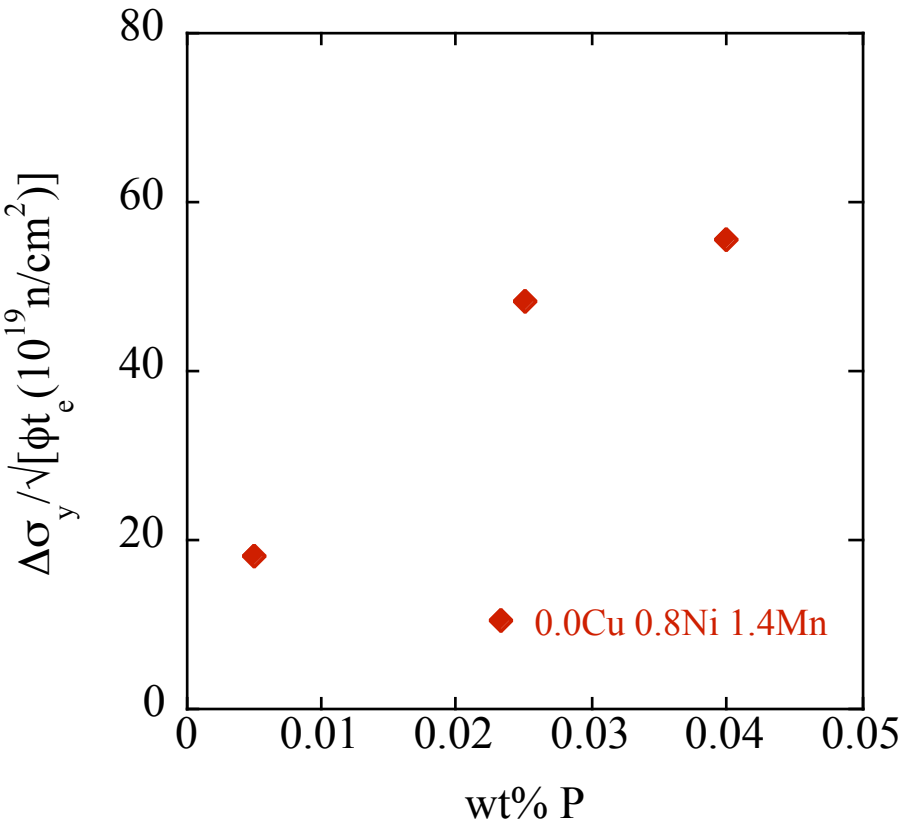
Cu and Ni Effects





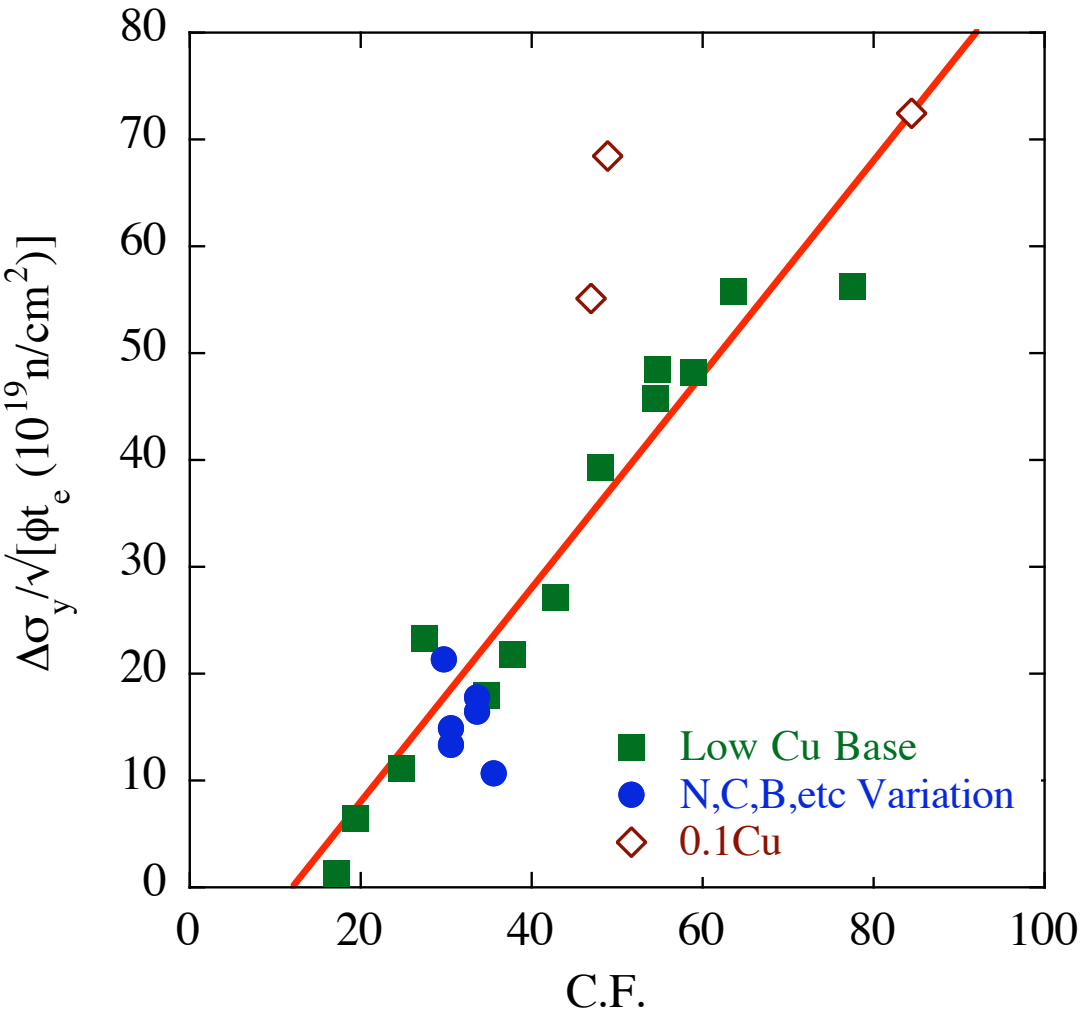
Mn and P Effects





MD CF (Preliminary)

$$B = 15.8\ Ni + 7.9\ Mn + 172Cu + 961P - 12.1 \pm 4.8$$



Other IVAR Related Experiments

- Major theory and modeling effort - predictions often led observations
- Size effects constraint loss experiment on Shoreham plate
- Large irradiated fracture matrix on small specimens, constraint loss and micromechanics
- Hardening mechanisms and constitutive law development for irradiated RPV steels
- Strain hardening effects on MC shape, ΔT_o & constraint loss
- Advanced small specimen techniques for irradiated alloys including advanced hardness methods
- Irradiation temperature change effects
- PIA and IE-PIA-RIE-PIA-RIE study
- Aging studies on the BWR very low flux effect

Implications to PREDB Analysis

- Most trends found in the Eason analysis of the PREDB are qualitatively consistent and ultimate quantitative agreement is expected
- CRP + MF model - $\Delta\sigma_y = \Delta\sigma_{yp} + \Delta\sigma_{ym}$ - fits the data well
- A plateau CRP hardening $\Delta\sigma_{ypm}$ is observed
- Pre-plateau $\Delta\sigma_{yp}(\phi t)$ shifted to higher ϕt with increasing ϕ
- Strong Cu-Ni interaction effect on $\Delta\sigma_{ypm}$ and the ϕt_e dependence
- Matrix feature $\Delta\sigma_{ym} = B\sqrt{\phi t_e}$ depends on ϕ
- Account ϕ-effect in both cases by - $\phi t_e \approx \phi t(\phi_r/\phi)^p$
- P-Mn effect in the MD term (IVAR sees both P and Mn effects as well as a direct P-Mn interaction)
- T-dependent MD term (weaker in IVAR)

Implications to PREDB Analysis

- Some differences - IVAR shows
- Strong Ni effect in the MD contribution
- Temperature dependence in CRP term (opposite small Eason fixup term)
- Mn effect in CRP term
- Decrease in the effect of P at higher Cu (limited information)
- A strong ϕ effect at higher $\phi > 5x10^{10}$ n/cm^2-s
- Some difference in product form coefficients
- Most of these differences can be rationalized by what IVAR can "see" and what the PREDB cannot in terms of both the S/N ratios and structures of the databases
- Work will continue to attmept to fully integrate the IVAR and PREDB evaluations

Some Open Questions

- Full analysis of the IVAR database and physical model linked to PREDB
- Flux effects in BWR regime
- Late blooming phases in low Cu steels
- Extrapolation of matrix feature damage beyond low flux database for life extension in PWRs
- Vessel attenuation
- MC and small specimen testing issues