

# Nanoscale Stable Precipitation-Strengthened Steels for Nuclear Applications

## Nuclear Energy Enabling Technologies (NEET) Reactor Materials

LANL - *K.D. Clarke (PI)*, A.J. Clarke, R.D. Field, S.A. Maloy, Y. Wang, R.E. Hackenberg

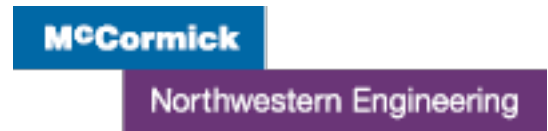
NETL - O.N. Dogan, P.D. Jablonski

CSM - K.O. Findley, J.G. Speer, K. Tippey

NU - S. Vaynman, M.E. Fine



DOE-NE Materials Crosscut Coordination Meeting, August 20, 2013

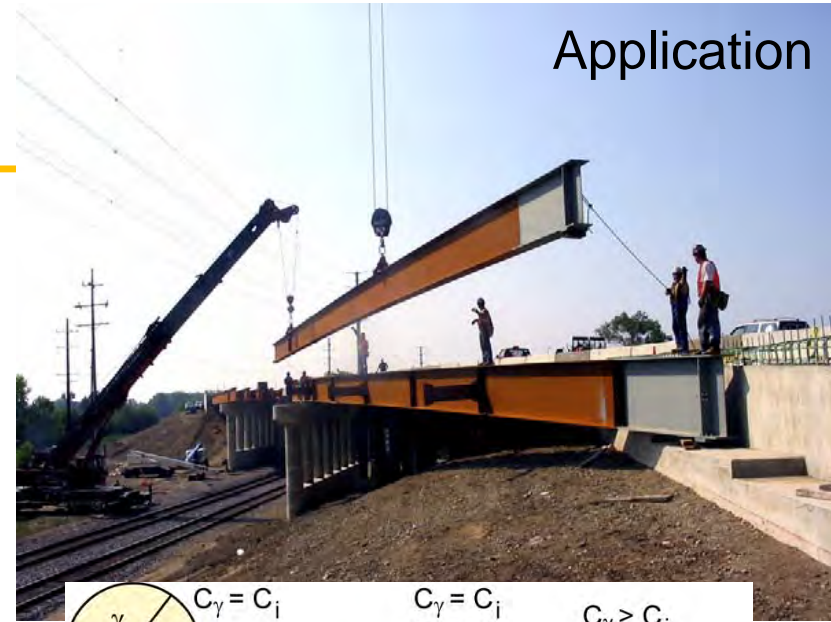


UNCLASSIFIED

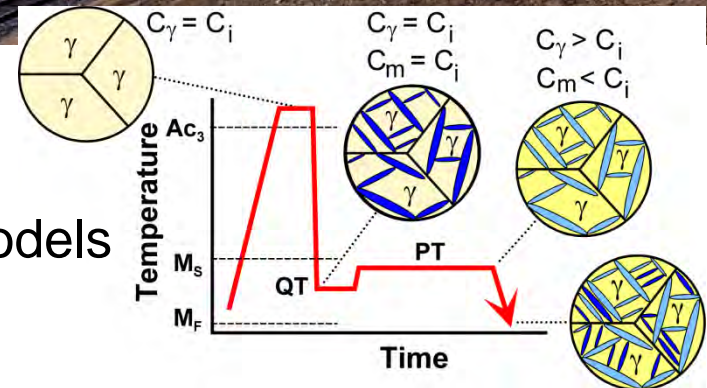
# Overview

- Background
- Investigative Team
  - Colorado School of Mines (CSM)
  - Northwestern University (NU)
- Materials Processing Strategies
- Material production
- Experimental Plan
  - Thermal studies
  - Mechanical testing
  - Irradiation studies
  - Characterization

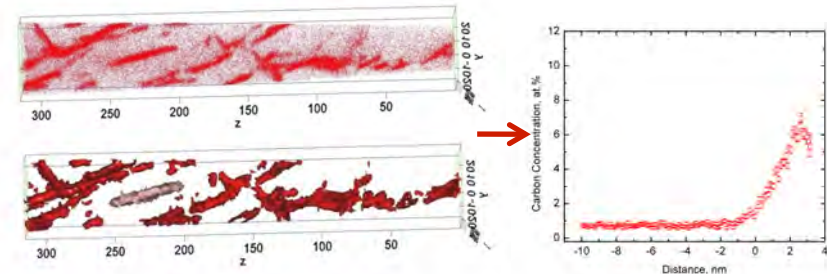
Application



Theory/Models



Characterization/Testing



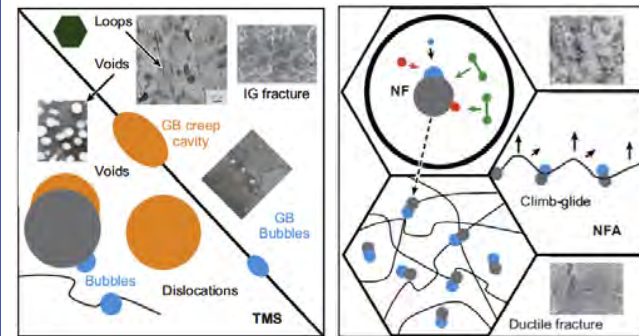
# Project Background and Objectives

- Thermal conductivity
- Compatibility
- High-temperature strength
- Ductility
- Creep resistance
- Irradiation resistance
- Affordability

Nuclear Enabling Technologies (NEET) Workshop Report, July 29, 2010, Rockville, Maryland, accessed March 9, 2012 at [http://www.ne.doe.gov/pdfFiles/Neet\\_Workshop\\_07292010.pdf](http://www.ne.doe.gov/pdfFiles/Neet_Workshop_07292010.pdf)



Stable, nanoscale precipitates and stable dislocation sinks improve irradiation resistance in steels.



Odette, G.R., Alinger, M.J., and Wirth, B.D., *Annu. Rev. Mater. Res.*, vol. 38, 2008, pp. 471-503.

- **Create precipitation-hardened steels with improved thermal stability.**
  - Develop alloying strategies to improve irradiation performance of steels.
  - Manufacture by conventional methods, with focus on manufacturability and affordability.
  - Produce desired microstructures: stable precipitates and dislocation structures.
  - Perform ion-beam experiments to determine irradiation resistance.
  - Examine microstructural effects on mechanical properties.

# Partnership - CSM, NU, NETL and LANL

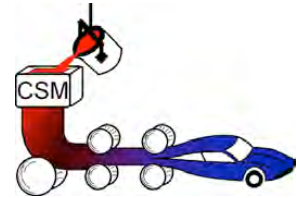
## ■ NETL

- Produce and process steels with desired compositions and microstructures
- Thermodynamic/kinetic expertise for alloy design



## ■ CSM - Advanced Steel Processing and Products Research Center

- Global leader in automotive and structural steel development
- History of developing novel steels using thermal/mechanical processing



## ■ NU - Infrastructure Technology Institute

- Pioneers of tailored-precipitation hardened steels for structural uses
- Understanding of precipitation strengthening and kinetic control

McCormick

Northwestern Engineering

## ■ LANL

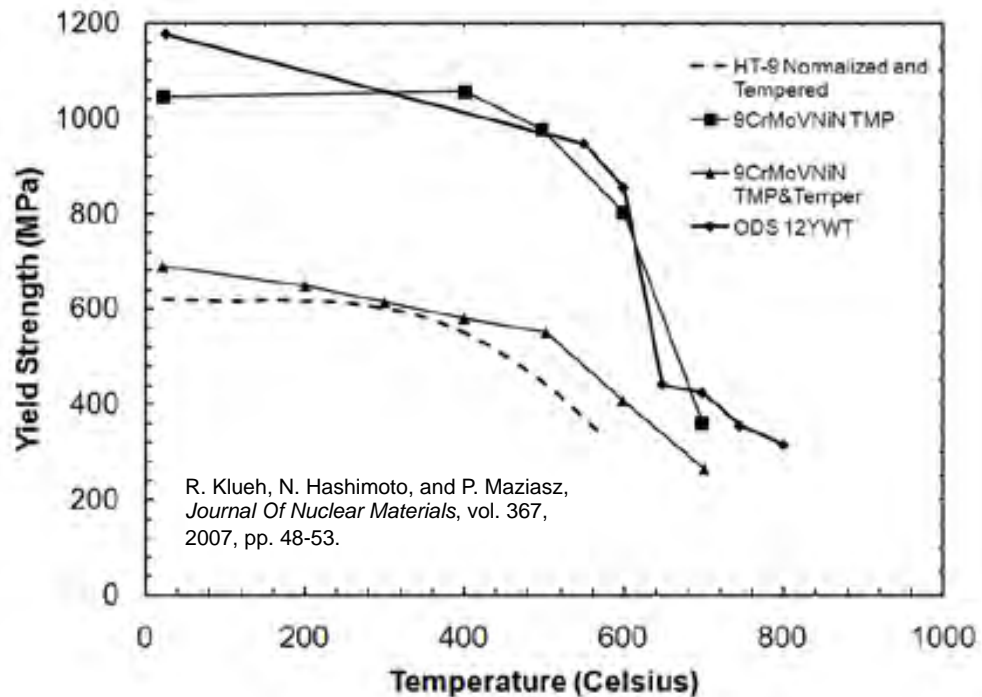
- Materials Science & Technology: Metallurgy (MST-6) steel physical metallurgy expertise
- Facilities for materials characterization
- Irradiation testing (IBML)



UNCLASSIFIED

# Material Design Strategies - CSM and LANL

- **Advanced High-Cr martensitic or ferritic-martensitic steels**
  - Traditional material in reactors
  - Optimize alloying and deformation processing for strength and creep resistance





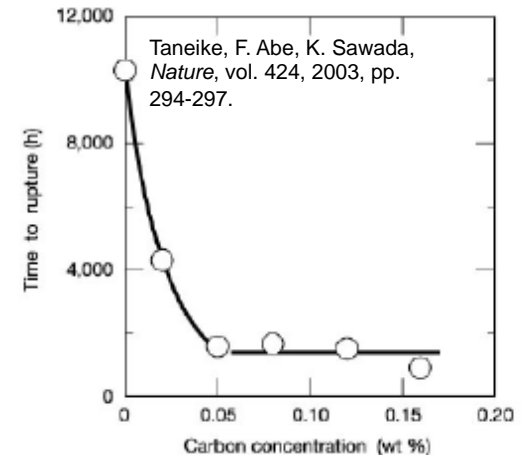
# Material Design Strategies - CSM and LANL

Table 1 - Composition of NF616, wt. pct.

C	Si	Mn	Cr	Mo	W	V	Nb	B	N
0.07	0.06	0.45	9.0	0.5	1.8	0.2	0.05	0.004	0.06

## ■ Base alloy, NF616 - Normalized and Tempered

- Lower C content to promote MX precipitation over  $M_{23}C_6$ 
  - More resistant to coarsening
  - Excellent creep strength
- Increased Mn and N
  - Stabilize austenite for thermomechanical processing
  - Coarsening resistance (VN)
- Mo, W alloying optimized for solid solution strengthening



*What is effect of C content on precipitation, aging stability, and resulting mechanical behavior?*

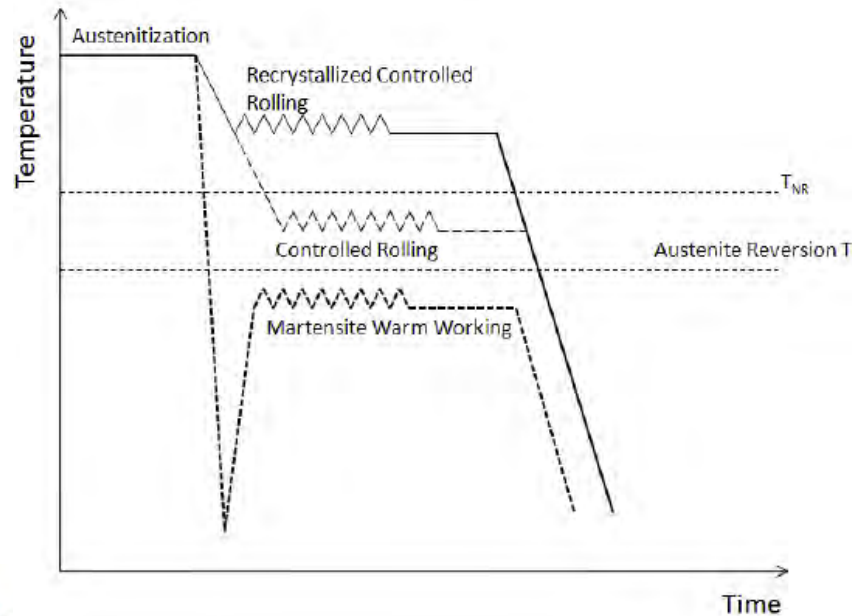
*To what extent can excess N improve austenite stability and coarsening resistance?*

# Material Design Strategies - CSM and LANL

## ■ Thermomechanical Processing (TMP)

- Thermal solutionizing treatments
- Cooling rate
- Hot- or warm-working

## ■ Initial Material supplied by NETL

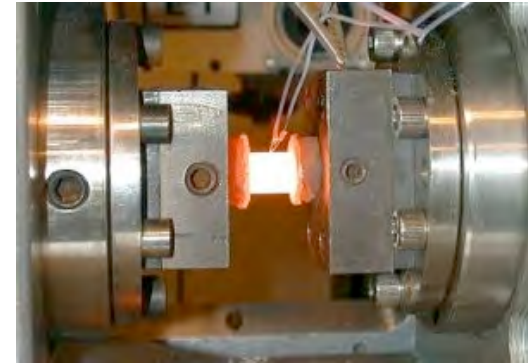


# Material Design Strategies - CSM and LANL

## ■ Thermomechanical Processing (TMP)

- Recrystallized controlled rolling
  - Decrease austenite grain size for nucleation sites
  - Decrease martensite packet/block size for strength
  - Increased interfacial area
- Controlled rolling
  - Retain dislocation substructure for nucleation sites
  - Increased strength
- Martensite warm working
  - Precipitate orientation relationship to matrix altered
  - Control precipitate growth kinetics

## ■ Laboratory-scale processing for promising processing routes

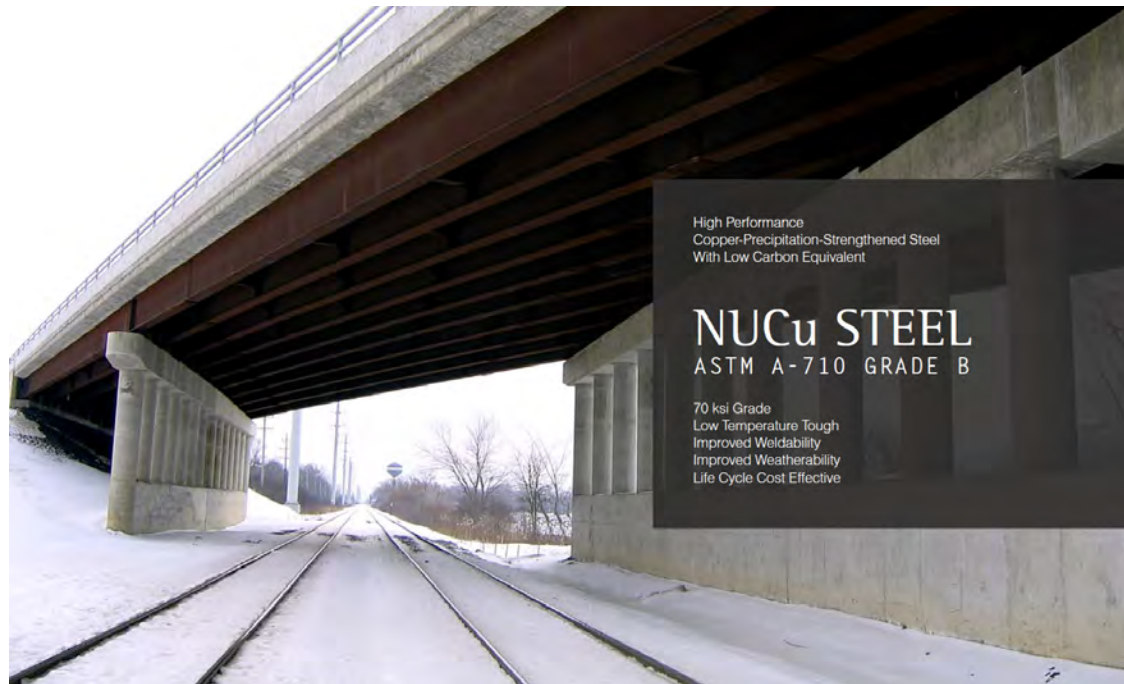




# Material Design Strategies - NU and LANL

## ■ Novel Tailored-Precipitate Ferritic (TPF) steels

- Designed nanoscale fine Al, Cu, Nb, or Mo-based precipitates plus deformation processing.
- Optimize for strength, corrosion resistance, irradiation resistance, weldability, and thermal stability.



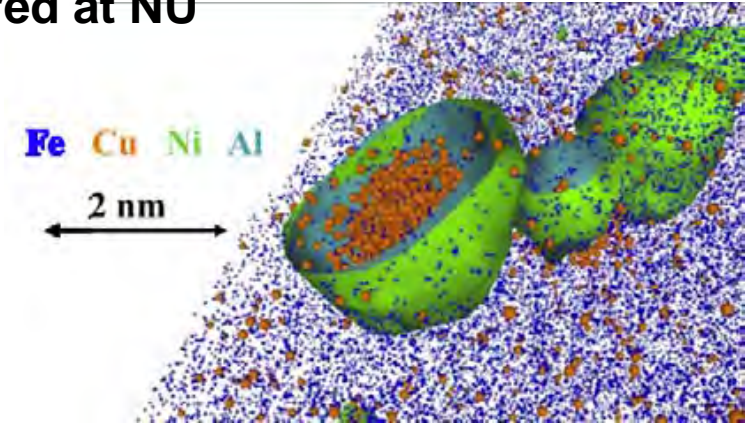
**Front Cover:**  
Bridge in Lake Villa, Illinois, build with  
NUCu 70W (ASTM A710 Grade B) Steel

UNCLASSIFIED

# Material Design Strategies - NU and LANL

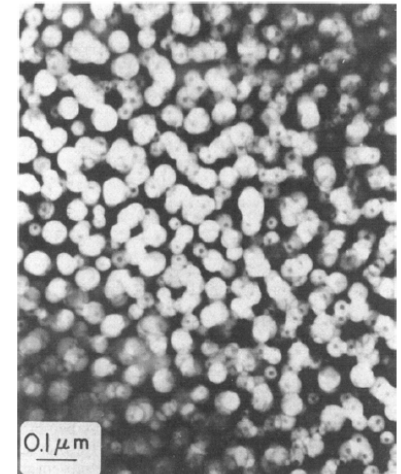
## ■ Corrosion resistant structural steels tailored at NU

- 1.3-2.5 Cu, 2.7Ni, 0.6Al
- YS = 1000 Mpa
- EI% = > 20%
- Impact energy > 70 J at -40 °C
- Unique structure - Ni-Al “shell”
- Initial irradiation testing being initiated



## ■ NU results suggest NiAl precipitates can be optimized for irradiation resistance (i.e., 17-7 PH stainless steels)

- Meets strength and ductility requirements
- Precipitate morphology shown to be adjustable with processing
- High-temperature stability (700 °C)
- Mo impedes morphology change/coalescence - Mo migrates to interface



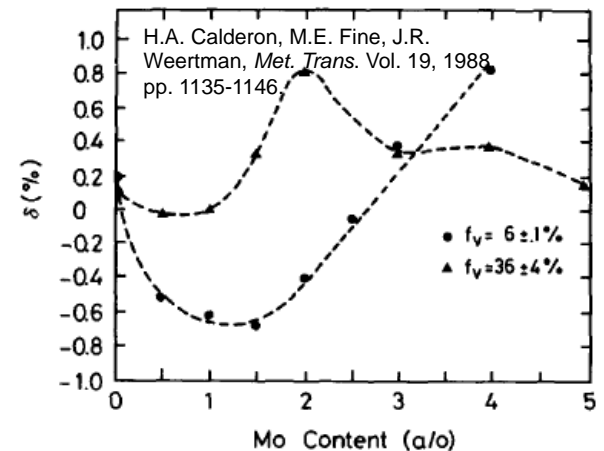
H.A. Calderon, M.E. Fine, J.R. Weertman, *Met. Trans.* Vol. 19, 1988, pp. 1135-1146.

## ■ Nb or V carbide optimization

# Material Design Strategies - NU and LANL

## ■ Design precipitates for application to nuclear reactors

- Alloys have been designed to take advantage of NiAl precipitate stability
  - NiAl - stable ordered phase (B2 structure)
  - Wide composition band possible - computational and experimental verification
- Quaternary alloying strategies, Mo
  - Control lattice parameter mismatch
  - Slow coarsening rate
  - Shape control
  - Size control
- Examination of Nb and V carbide behavior
- Thermomechanical processing



**Can FeNiAl alloys be further optimized for use in irradiation-resistant materials?**

# Material Design Strategies - NU and LANL

## ■ Weldability

- Previous steels have been extremely weldable, which is rare in advanced, high-strength materials. Important for manufacturability.
- Controlled carbon equivalent and ability to control temperature during welding - no brittle heat affected zone



UNCLASSIFIED



# Partnership - CSM, NU, NETL and LANL

## ■ Tailored-Precipitate Ferritic Steels (TPF)

- NiAl - alloys have been produced at NETL
  - Four 5kg heats
    - 2.8Ni, 2.3Al
    - 2.6Ni, 3.0Al
    - 0.08C, 0.1Nb
    - 0.08C, 0.1V
- Aging studies underway at NU - test thermal stability and mechanical properties as a function of time and temperature
- Irradiation studies being initiated at LANL

## ■ Advanced Hi-Cr ferritic or ferritic-martensitic steels

- NF616 produced at NETL
- Thermomechanical examinations underway at CSM
- Irradiation studies being initiated at LANL





# Experimental Plan - CSM, NU, NETL, and LANL

---

- **Material Production and Thermomechanical processing**
- **Aging studies (CSM, NU)**
  - 650 °C up to 5000 hours (>200 days)
  - Microstructural and mechanical evaluations to determine best-performing materials
- **Mechanical Testing (CSM, NU, LANL)**
  - Tensile testing to 700 °C
  - Creep-rupture testing
  - Nano-indentation
- **Irradiation, Ion Beam Materials Laboratory (IBML) at LANL**
  - Irradiation processes being determined
  - Mechanical and Microstructural response
- **Microstructural Characterization and Interpretation (CSM, NU, LANL, NETL)**
  - Precipitate analysis via TEM and EDS, along with 3DAP
  - EBSD for texture and grain-boundary type information
  - Understand relationships between processing, microstructure, and properties

# Summary

- Develop stable nano-precipitates for reactor applications
- Use flexible processes and tailored compositions to produce desired microstructures
- Characterize microstructures with advanced techniques
- Optimize microstructures for reactor applications
- Initial iterations of materials designed and produced through conventional steelmaking processes
- First stages of testing underway

