

# Irradiation for the Novel Radiolytic Formation of Superalloy Nanoparticles



Sandia National Laboratories

Tina M. Nenoff, Zhenyuan (Mark) Zhang, Jianyu Huang,  
Paula Provencio, Kevin Leung, Donald T. Berry

## Problem

### Utilization of Novel NP Formation without Sintering or Oxidation

Superalloys: Ni-based alloys that are hardened with refractory metals to higher temperatures while retaining superb mechanical strength e.g.,  $\gamma$ -phases Ni/(Co, Cr, Mo, or W) or  $\gamma'$ -phases Ni<sub>3</sub>(Al, Ti)

Synthesis of nanoparticles of alloy compositions allows for both nano- and bulk-scale applications of alloys.

Sintering of nanoparticles of an alloy allows for more defect-free bulk alloys, non-destruction of the refractory elements, and access to metastable phase spaces and morphologies.

Radiolysis is a room-temperature method to produce kinetically favorable, metastable alloy nanoparticles.

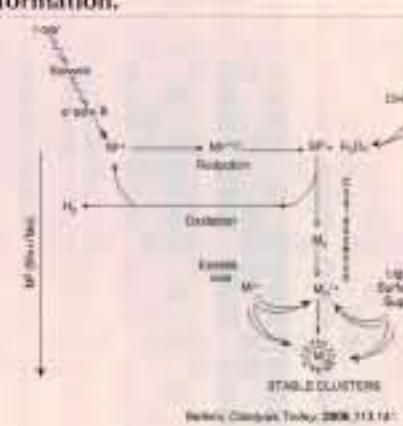
Ability to make homogenous NPs, independent of reaction size, means large quantities are possible.

## Approach

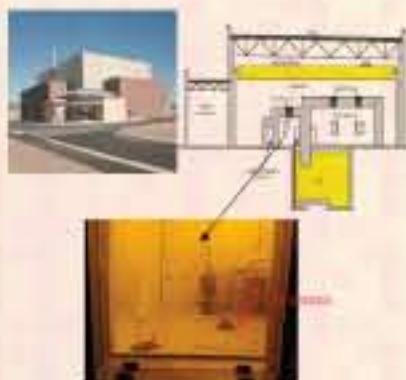
### Room Temp Radiolysis at SNL's GIF Facility

Radiolysis is a method by which metal ions are reduced in water by ionizing radiation:

The dose rate dictates the [e<sup>-</sup>] in the reaction solution thereby affecting the chemistry of the NP formation.



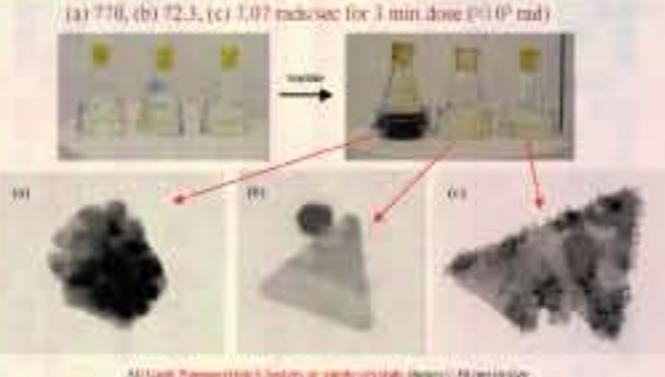
Sandia's Gamma Irradiation Facility (GIF) is a <sup>60</sup>Co source:  $1.345 \times 10^3$  Ci, = 300 K rad/hr.



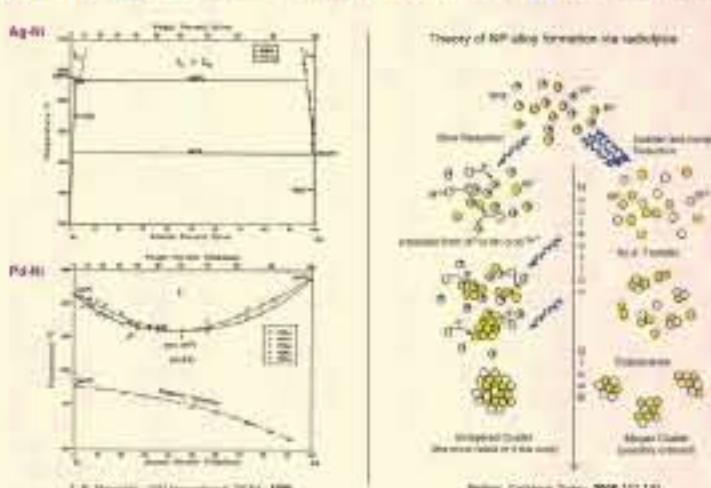
## Results

### Gold Nanoparticles, Morphology Determined by Dose Rate

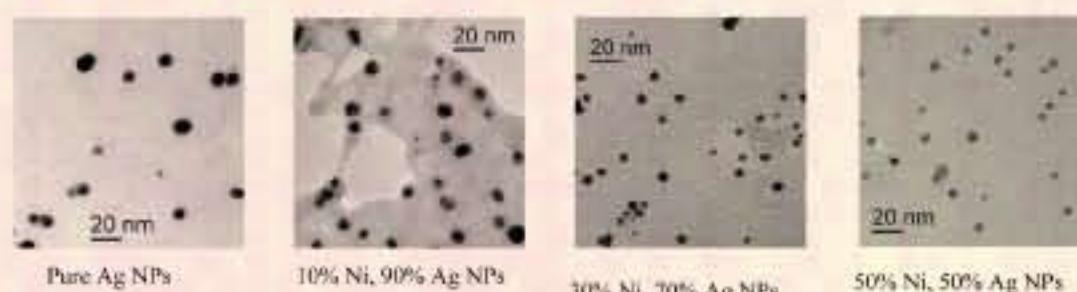
Reaction Conditions: 25-mL solvents in 100-mL vials  
HAuCl<sub>4</sub>·H<sub>2</sub>O (1000 ppm Au in dilute HNO<sub>3</sub>, polyvinyl alcohol) (PVA; MW of 48000), DI H<sub>2</sub>O  
Purple solution with N<sub>2</sub> sealed and stored in dark  
Exposed solutions to γ-irradiation, allowed to age and crystals to grow



### Kinetically Driven Access to New Phase Spaces: Thermodynamically inaccessible or limited Ni-based NPs

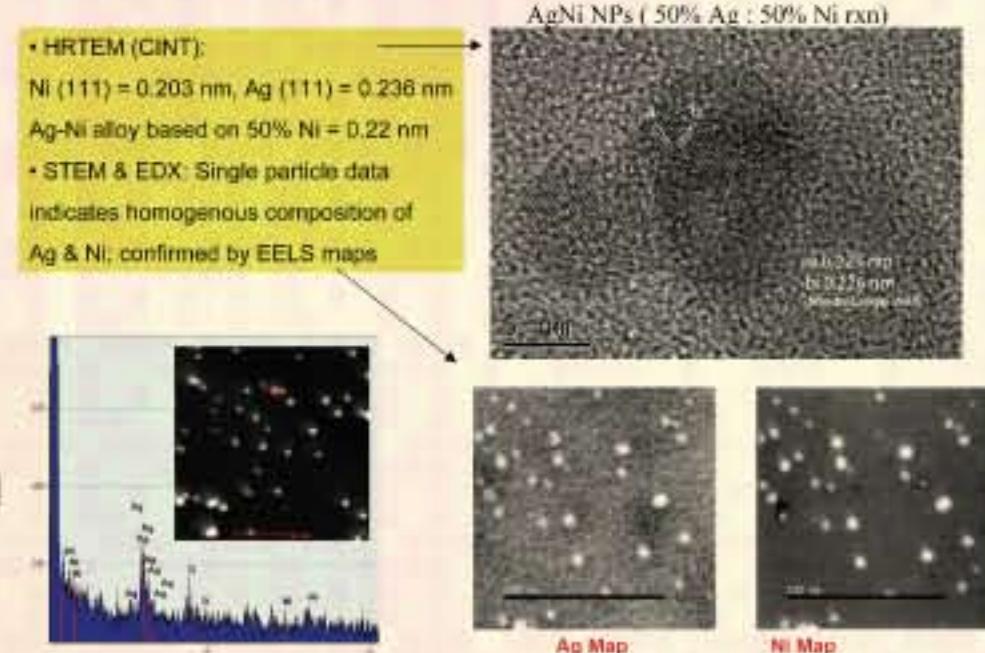


### Reaction Composition Effect on NP sizes: TEM



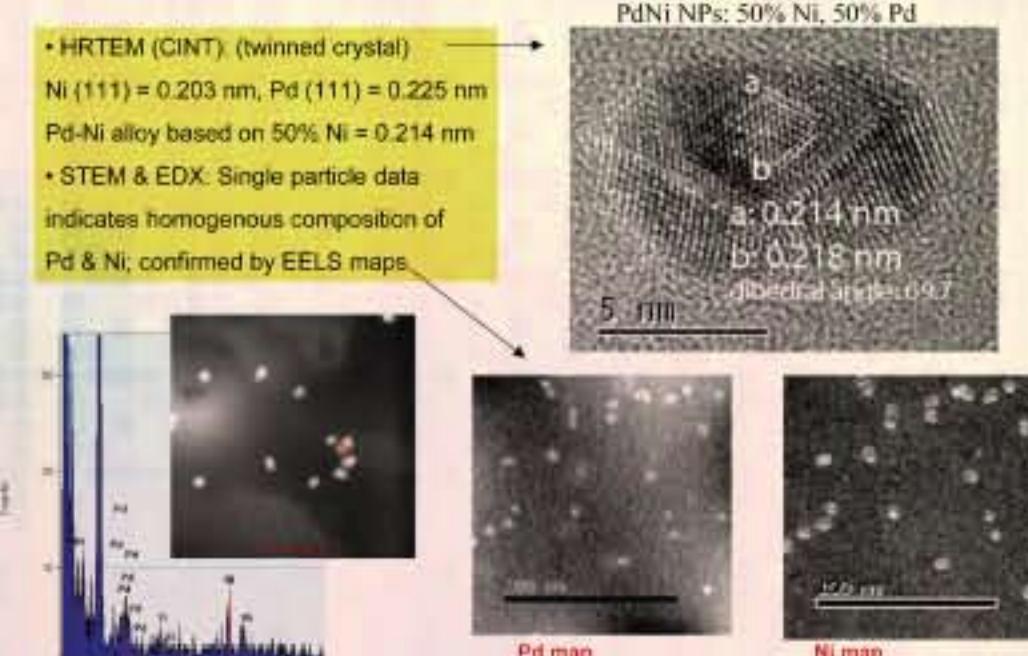
### AgNi Superalloy Nanoparticles

- HRTEM (CINT): Ni (111) = 0.203 nm, Ag (111) = 0.236 nm  
Ag-Ni alloy based on 50% Ni = 0.22 nm
- STEM & EDX: Single particle data indicates homogenous composition of Ag & Ni; confirmed by EELS maps



### PdNi Superalloy Nanoparticles

- HRTEM (CINT): (twinned crystal)  
Ni (111) = 0.203 nm, Pd (111) = 0.225 nm  
Pd-Ni alloy based on 50% Ni = 0.214 nm
- STEM & EDX: Single particle data indicates homogenous composition of Pd & Ni; confirmed by EELS maps



## Significance

- Long-term Science for SNL Business SMUs (NTM, ERN) plus DOE National Security Mission and Strategic Goals.
- Defense, Energy, Science applications require SuperAlloys: lightweight, corrosion-resistant, sintered refractory materials (weapons casings & connects, aircraft, satellites, power plants, gas turbine engines & burners)
- Leverage future funding by DOE/NE (nuclear fuel alloys), DOE/H<sub>2</sub> (H<sub>2</sub> dissociative membranes), DOD/DARPA/DTRA