

Irradiation for the Novel Radiolytic Formation of Superalloy Nanoparticles

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Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US DOE's
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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.: γ -phases Ni/(Co, Cr, Mo, or W) or γ' -phases Ni₃(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 meta-stable 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

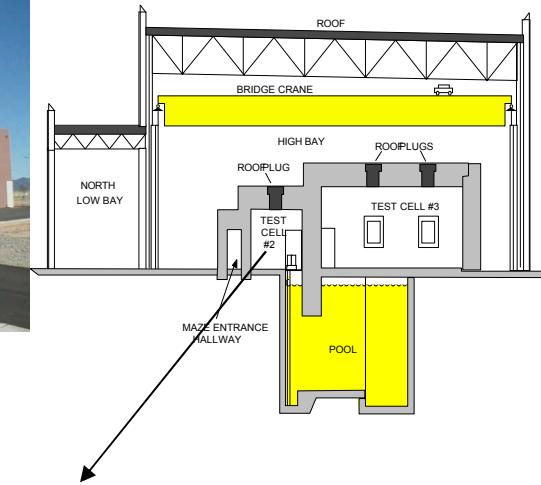
Approach:

Room Temp Radiolysis at SNL's GIF Facility

Radiolysis is a method in which metal Ions are reduced in water by Ionizing Radiation: The dose rate dictates the $[e^-]$ in the reaction solution thereby affecting the chemistry of the NP formation.

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

Sandia's Gamma Irradiation Facility (GIF) is a ^{60}Co source : $1.345 \times 10^5 \text{ Ci}$, $\approx 300\text{K rad/hr.}$



C

A
B
Co source

Results: Gold Nanoparticles, Morphology Determined by Dose Rate

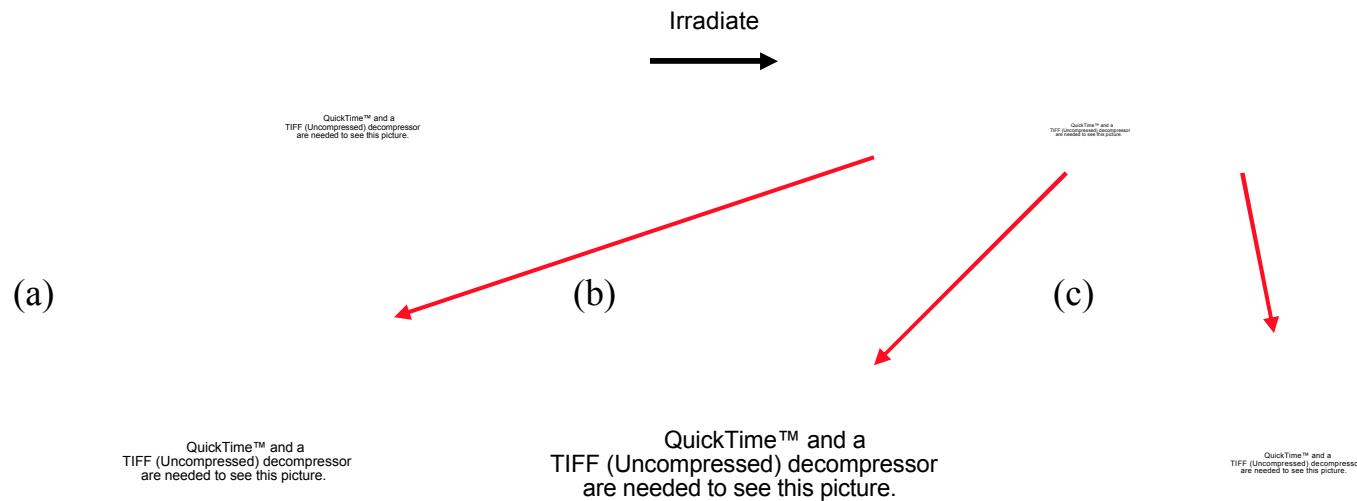
Reaction Conditions: 25ml solutions in 100ml vials

$\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ (1000ppm Au in dilute HNO_3), poly(vinyl alcohol) (PVA, MW of 88000), DI H_2O

Purged solution with N_2 , sealed and stored in dark

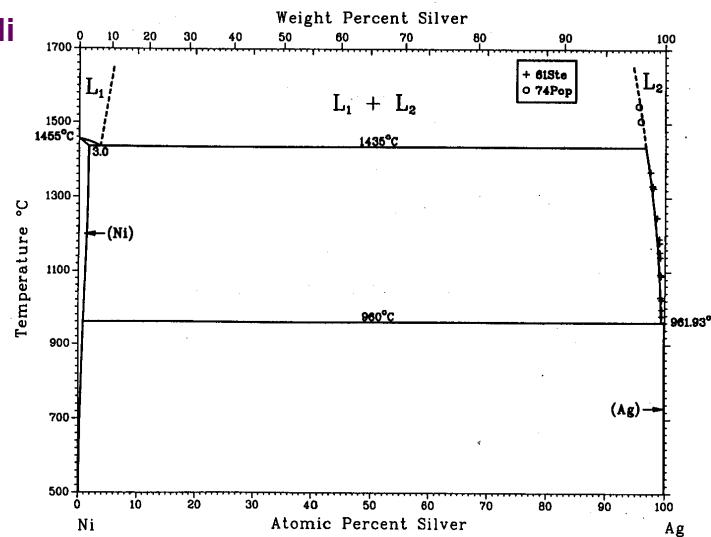
Exposed solutions to γ -irradiation; allowed to age and crystals to grow

(a) 770, (b) 72.3, (c) 7.07 rads/sec for 3 min dose ($\approx 10^3$ rad)

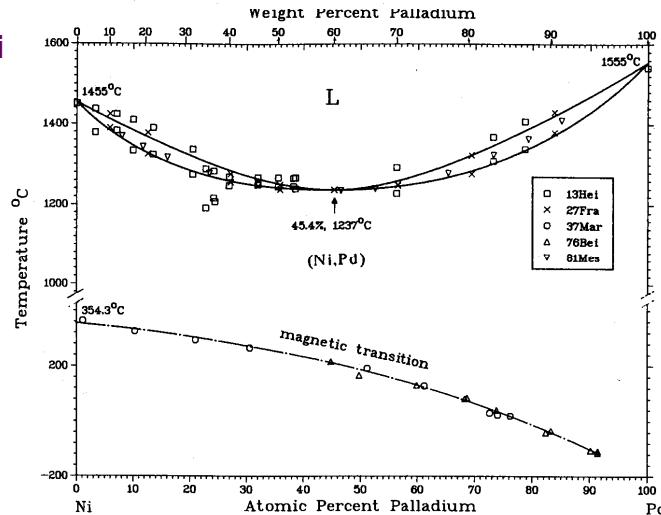


Kinetically-driven Access to New Phase Spaces: Thermodynamically inaccessible or limited Ni-based NPs

Ag-Ni



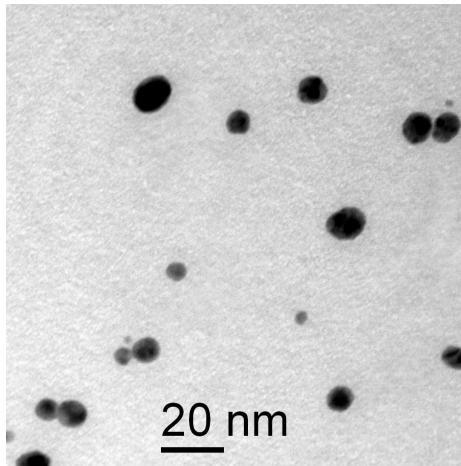
Pd-Ni



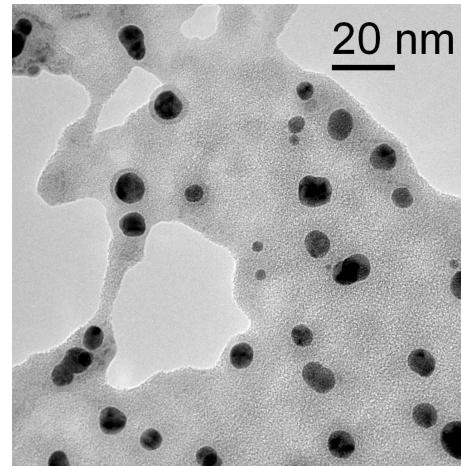
Theory of NP alloy formation via radiolysis

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

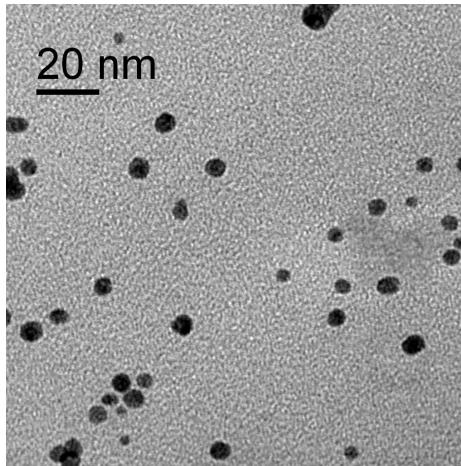
Reaction Composition Effect on NP sizes: TEM



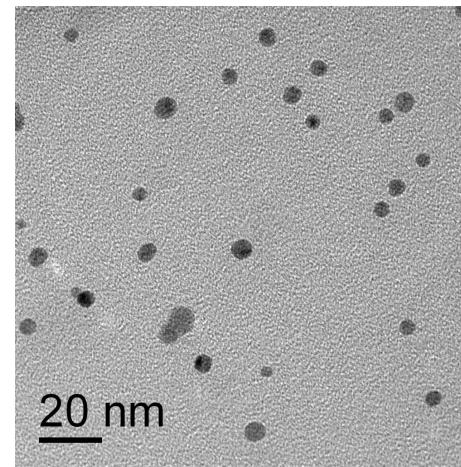
Pure Ag NPs



10% Ni, 90% Ag NPs



30% Ni, 70% Ag NPs



50% Ni, 50% Ag NPs

AgNi Superalloy Nanoparticles

- HRTEM (CINT):

$\text{Ni (111)} = 0.203 \text{ nm}$, $\text{Ag (111)} = 0.236 \text{ nm}$

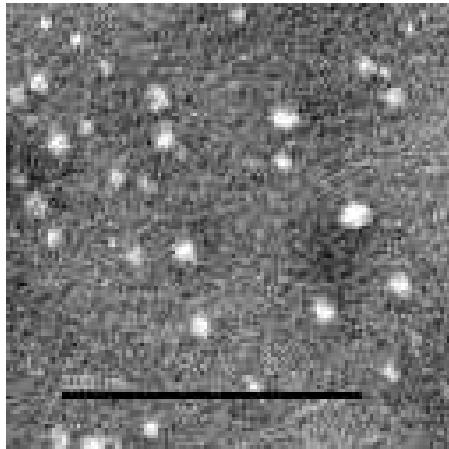
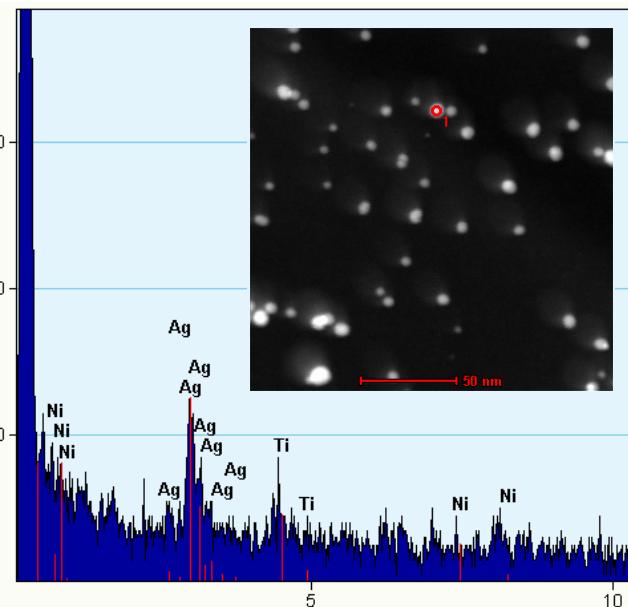
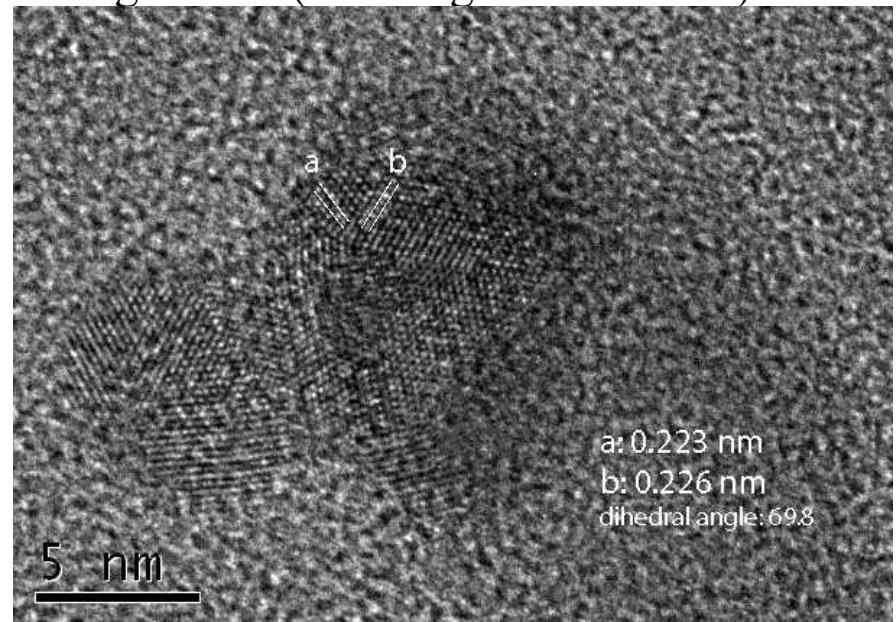
Ag-Ni alloy based on 50% Ni = 0.22 nm

- STEM & EDX: Single particle data

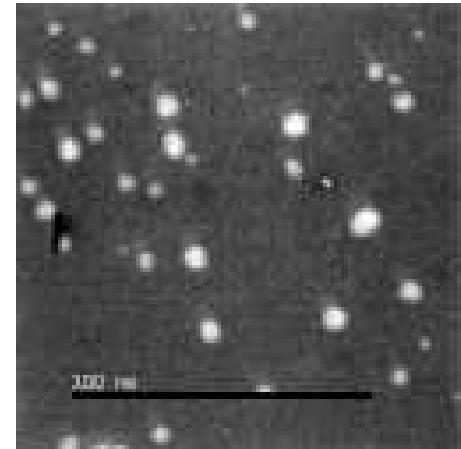
indicates homogenous composition of

Ag & Ni; confirmed by EELS maps

AgNi NPs (50% Ag : 50% Ni rxn)



Ag Map



Ni Map

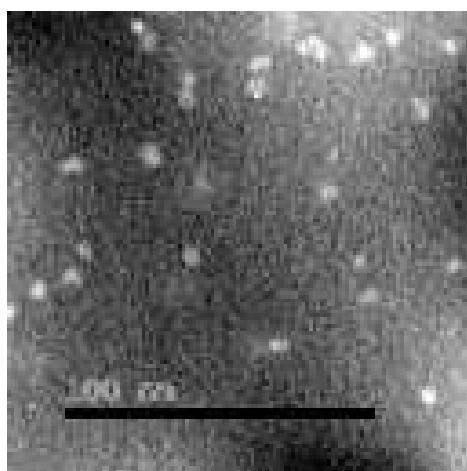
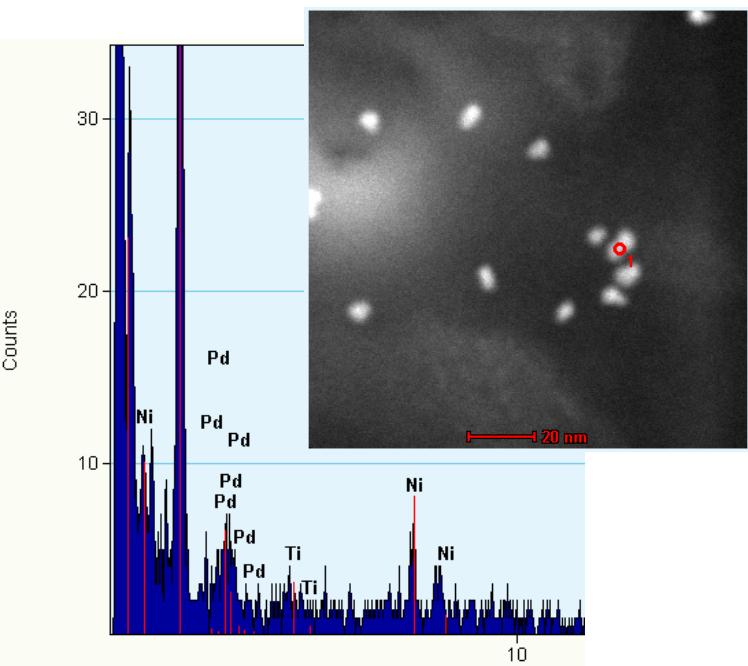
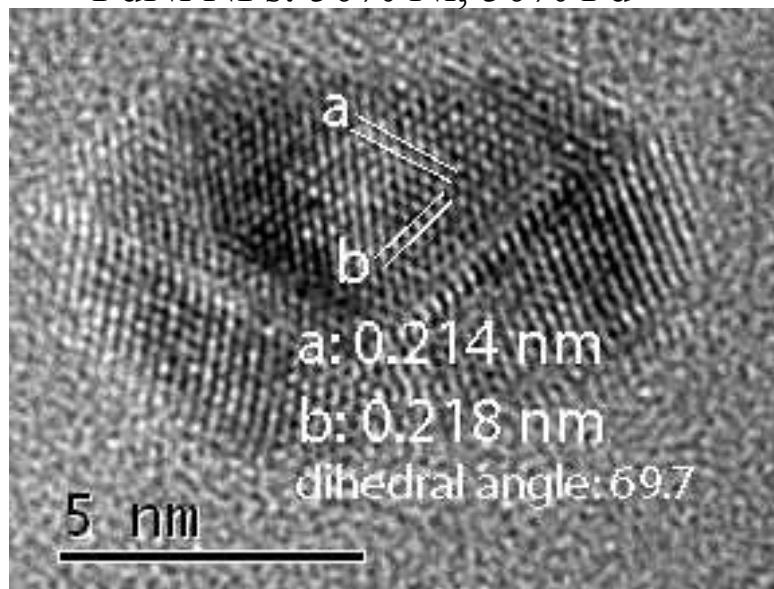


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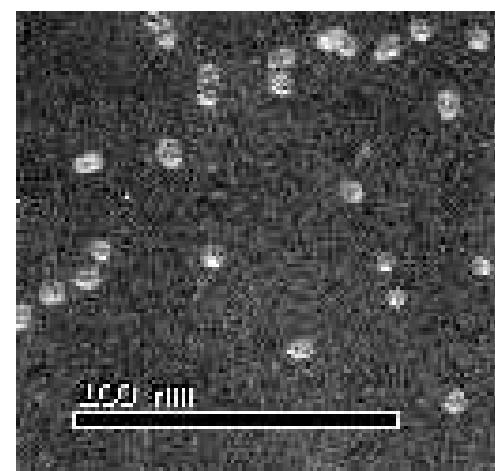
PdNi Superalloy Nanoparticles

- 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

PdNi NPs: 50% Ni, 50% Pd



Pd map



Ni map



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Significance:

Long Term Science for SNL Business SMU's (NTM, ERN) plus
DOE National Security Mission and Strategic Goals.

Defense, Energy, Science applications require SuperAlloys:
light weight, 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/H₂ (H₂ dissociative membranes), DOD/DARPA/DTRA