Application of Nanostructured powders synthesized by new chemical processes

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Characteristics of Nanopowder Materials

Surface Effect

- **Enhanced catalytic properties**
- **Enhanced absorption ability**
- **Capilary Condensation**

Bulk Effect

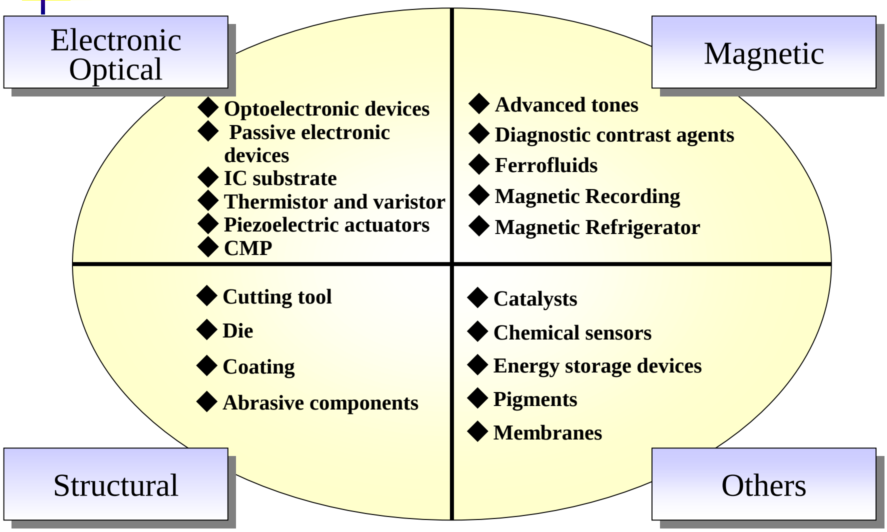
- **Appearance of New Phases**
- **Decrease of Melting Point**
- **Polycrystallization of Single Crystal**
- **Enhanced Scattering Effect of Waves**

Interaction between Nanopowders

- **Electric and heat transfer**
- **Compressability**
- **Solid state reactivity**

Property	Materials	Size(Å)	Nano-materials	Micro-materials
Magnetic Property	Fe	50	1030Oe	~470Oe
Melting Point	Au In	30 40	933K 370K	1300K 430K
Light Absorption (6~10μm)	Au	100	95%	2~5%
Transition Temperature of Superconductivity	Al	90	5.3K	3.4K
Heat Transfer	Ag	100	2.0mK	20mK
Sintering Temperature	Ni W	200 220	~200℃ ~1000℃	700℃
Catalytic Property (As : standard Activity)	Ni	10	6As	~3As

Application Field of Nanopowder Materials



Research of Nanopowders in KIMM

Area	Materials
Tool Materials	Abrasives, Cutting tools, CMP, Wear-resistant components **WC-Co hard materials**
Magnetic Materials	Ferrofluid, Magnetic refrigerator, Recording media, Hard/Soft Magnets, **Fe/Co magnetic materials, Nd-Fe-B hard magnet**
Electric/Electrode Materials	Thermistor, Varistor, Piezoelectric actuator **Cu-Al_2O_3 electrode, W-Cu heat sink**
Chemical/Catalytic Materials	Chemical Sensor, Membranes, Filter, **TiO_2 photocatalysts**

Mechanical Properties of WC/Co

- Chemical compositions(contents of Co)
- Particle size of hard phase(WC)
- Homogeneity of WC/Co (mean free path)

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- Reduction of WC size
- Decrease of mean free path

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- Hardness
- Compressive Strength
- TRS Strength
- Wear Resistance

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Key for high mechanical properties
- Fabrication of very fine WC
- Higher homogeneity of WC and Co phases

Manufacturing of WC/Co Alloys

Solid Phase Process

APT powder → Calcinations → Milling(+C) → Reduction → Milling(+Co) → Carburization

G.S. > 300nm

Liquid Phase Process

W,Co chlorides → Spray drying → Removal of Chlorides → Reduction/ Carburization; or Milling(+C) → Reduction/ Carburization

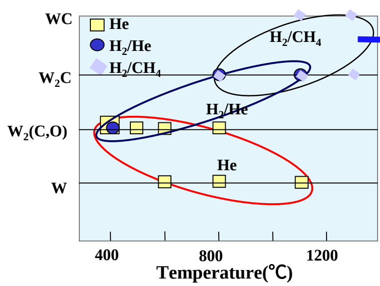
G.S. > 100nm

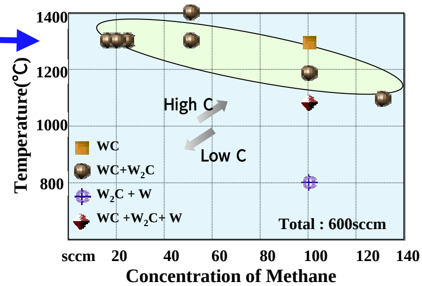
Gas Phase Process

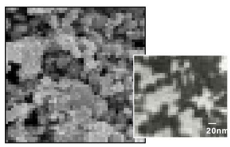
W, Co metalorganics → Evaporation/ Carburization → WC, WC - Co

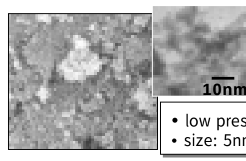
G.S. < 30nm

n-WC powder





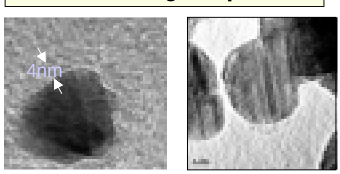




- low pressure(10^{-3}atm)
- size: 5nm

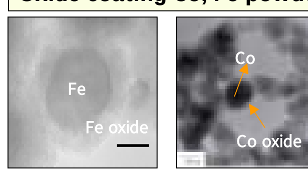
Coating of n- WC,Co powder

Carbon coating WC powder

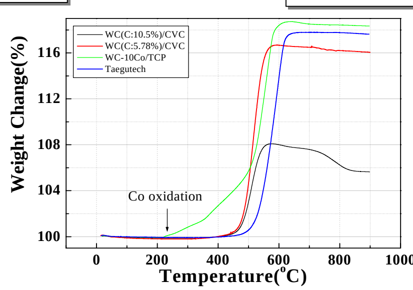


- WC powder: 4nm

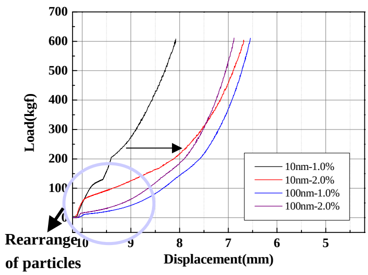
Oxide coating Co, Fe powder

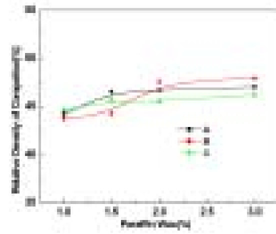


- Core(metal)/Shell(Oxide)
- Oxide shell : 3nm



Compactabilityof n-WC powder

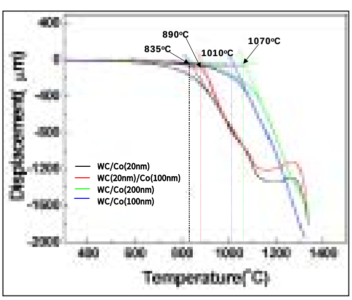




type	Power size	BET (m^2/g)	A.D. (g/cm^3)
A (NRL)	20nm	18.4	0.77
B	100nm (granule)	2.02	1.85
C	100nm	3.09	2.2

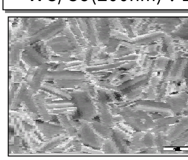
Compact desity 47%

Sinterbility of n-WC/Co powder

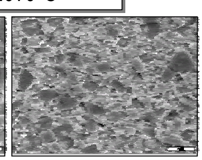


Solid sintering temp.

- WC(20nm)+Co(20nm) : 835°C
- WC(20nm)+Co(100nm) : 890°C
- WC/Co(100nm) : 1010°C
- WC/Co(200nm) : 1070°C



20nm→2㎛
(x 100)



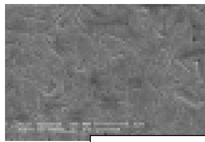
100nm→1㎛
(x 10)

- sintering temp: WC size(1070→835°C)
  Co size (890→835°C)
- abnormal grain growth (100 times,)

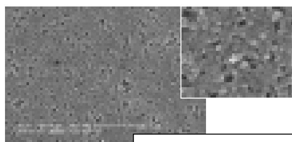
Grain growth inhibitors

Plasma sintering/grain growth inhibitor

Plasma sintering

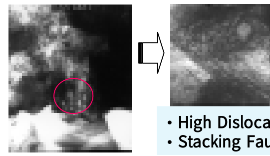


WC-10Co
facetted shape 1㎛ (x 50)



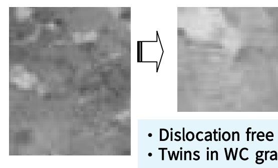
WC-10Co-0.6VC
round, 70nm(x 4)

Conventional WC-10Co



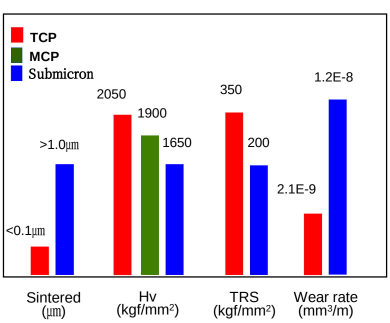
- High Dislocation Density
- Stacking Faults

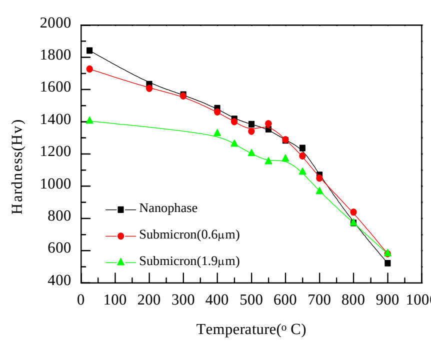
Nano. WC-10Co



- Dislocation free WC grains
- Twins in WC grains

Properties of Nanostructured WC-Co Alloy



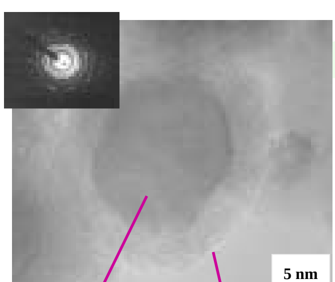


Nano sized Fe and Co Magnetic Materials

- **Magnetic Fluid : Magnetite(Fe_3O_4)**
- **Saturation Magnetization of Fe = 2 times of that of Fe_3O_4**
  Coercivity ; proportional to inverse of particle size (Hc = a+b/D)
- **Synthesis of nano-sized Fe ⇒ High performance magnetic fluid materials(Saturation Magnetization : 1500emu/cm^3, Coercivity : 3000Oe)**

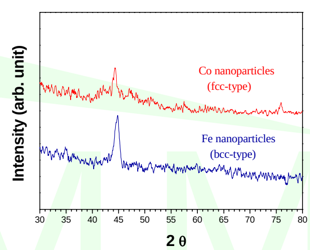
Development of Fe based nanopowder for magnetic fluids by Chemical Vapor Condensation

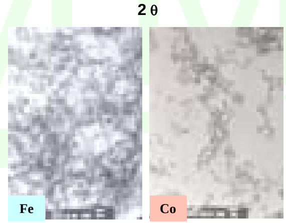
Microstructures of Fe Nanoparticles



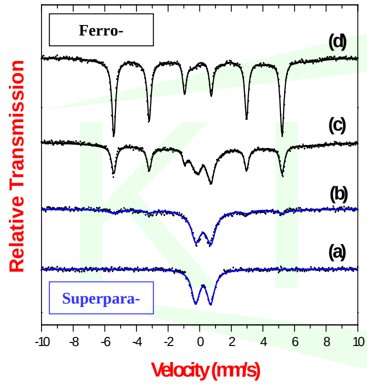
Core
Crystalline Fe

Shell
Crystalline Fe oxides

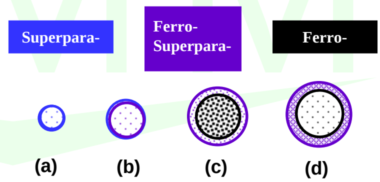




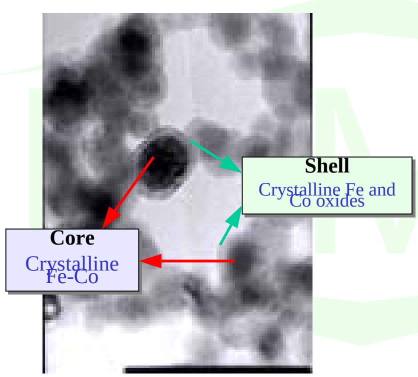
Mössbauer spectroscopy of Fe nanoparticles

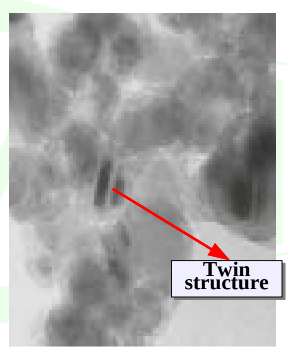


Samples	α-Fe (%) Bulk	α-Fe (%) Middle	Super-paramagnetism (%)	Fe_3O_4 (%)	Mean size (nm)
(a)	2	0	98	0	5.39
(b)	20	5	75	0	~7.0
(c)	34	24	21	21	10.32
(d)	70	0	10	20	~12.0

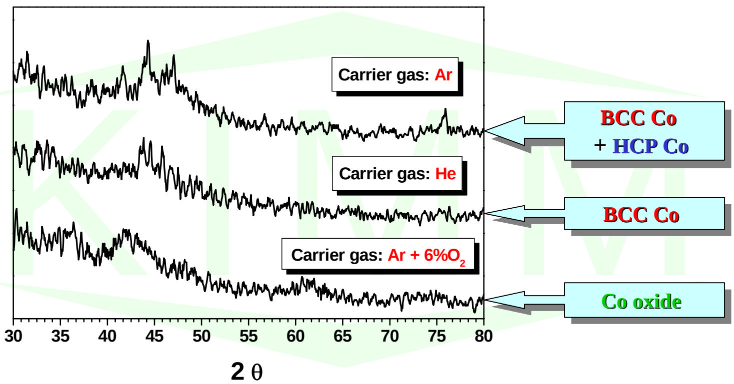


Microstructures of Fe-Co nanoparticles

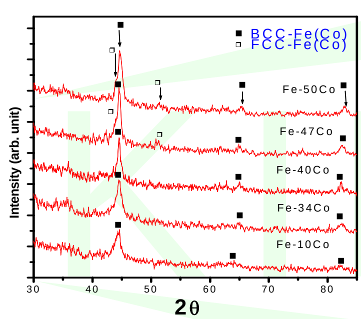


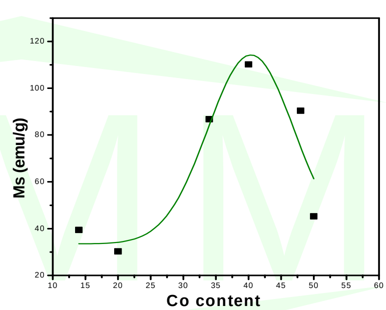


Phases of Co nanoparticles with carrier gases



Phases and saturation magnetization





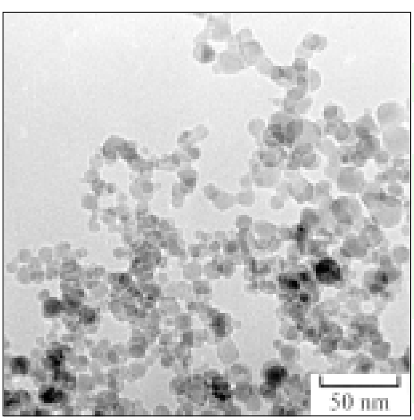
Different phases with different Co content

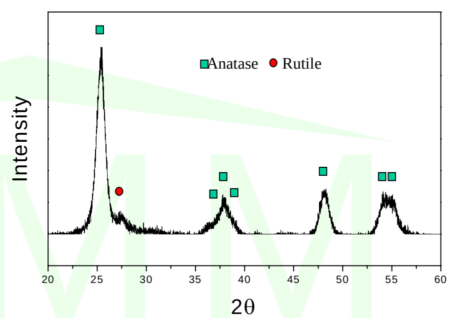
Saturation magnetization reaches highest value near 40 wt% Co

Nanostructured TiO_2 photocatalytic materials

- Decrease of particle size
  - Improved UV scattering
  - Enhanced photocatalytic activity
  - Improved gas sensing property
  - Enhanced opto-electronic property
- Synthesis of nano sized TiO_2 powders by Chemical Vapor Condensation process (Non-Agglomerated 10nm powder)

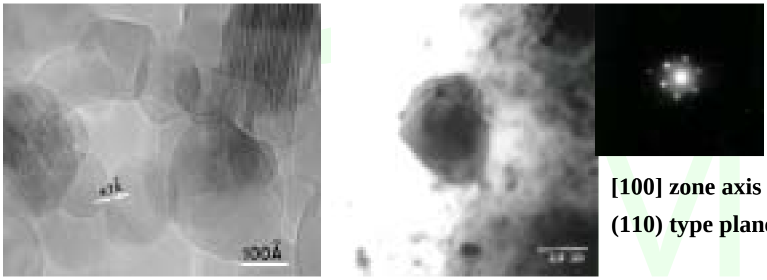
Microstructure of Nano-sized TiO_2 Powder





size : 10nm
loose agglomerates
phases : Anatase + Rutile (<2%)

Phase Change Depending on Powder Size



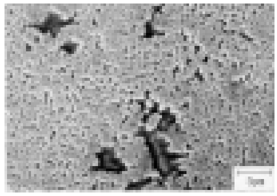
Anatase
a=3.75 Å, c=9.51 Å

Rutile
~60nm

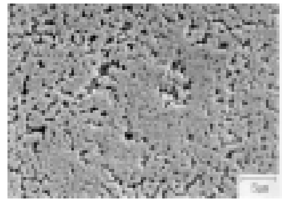
Nanostructured W-Cu heat sink materials

- Poor sinterability due to the negligible solid solubility between W and Cu
- Conventional Process
  - Infilteration : Low thermal & electric conductivity due to the addition of sintering activator
  - Liquid phase sintering : Low properties due to larger W size
- Development of new process to achieve high density nanostructured W/Cu materials, using nanostructured powder (W : 60nm)

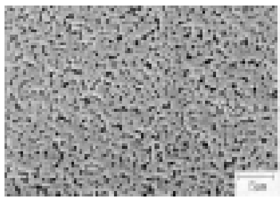
Microstructures of W/Cu Alloys



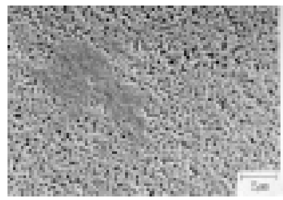
Mixing method
W:0.51%, Cu:20%



Thermochemical method
(W-20wt%-0.5wt%Co)

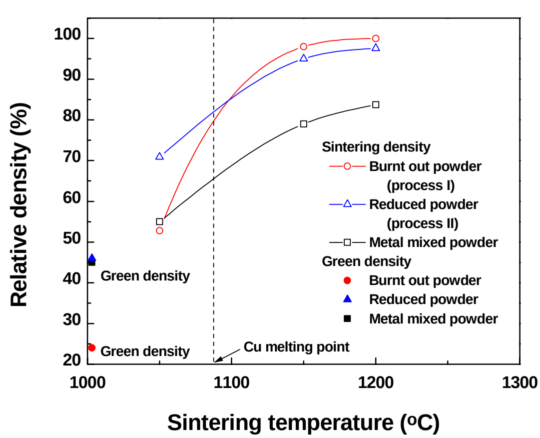


Mechano-thermochemical method(I)
Burnt out powder, Two-reduction step



Mechano-thermochemical method(II)
Reduced powder (200C/1h+700C/1h)

Sinterabilty of Nanostructured W/Cu Powders



Comparison of Thermal Properties

	W-20Cu Thermkon -76	W-20Cu Thermkon -83	Mo-15Cu Thermkon -65M	Mo-20Cu Thermkon -70M	W-20Cu KIMM 700-8	W-20Cu KIMM 700-8	W-20Cu KIMM 750-4
Density (g/㎤)	15.94	14.63	9.89	9.81	15.48-L 15.17-T	15.43-L 15.17-T	15.16-L 14.85-T
Specific Heat (J/Kg·K)	223.2	240.1	294.5	300.0	223.2	223.2	223.2
Thermal Conductivity (W/mK)	207 (180-210)	242 (180-210	141 (180-210	170 (180-210	233.0	245.8	221.3
Thermal Expansion Coefficient (ppm/K)	6.5 (7.2-8.0)	8.0(8.1-8.9)	5.4 (6.0-7.0)	6.5 (6.8-7.6)	7.80	7.88	7.25

Prospect of Nanopowder Materials