# *Metallurgical Considerations for PVD Materials*

Williams ADVANCED MATERIALS

# WAM Headquarters


**Buffalo, NY USA** - Manufacturing Facility

- 100,000 $ft^2$ overall, 6,500 $ft^2$ of cleanroom, state-of-the-art machining/ milling/rolling/stamping/ cladding centers, target bonding, hydrostatic wire extrusion, high purity refining/recycling, metals casting, automated plating, full analytical capabilities, product Research & Development.

➢ Subsidiary of Brush Engineered Materials, Inc.

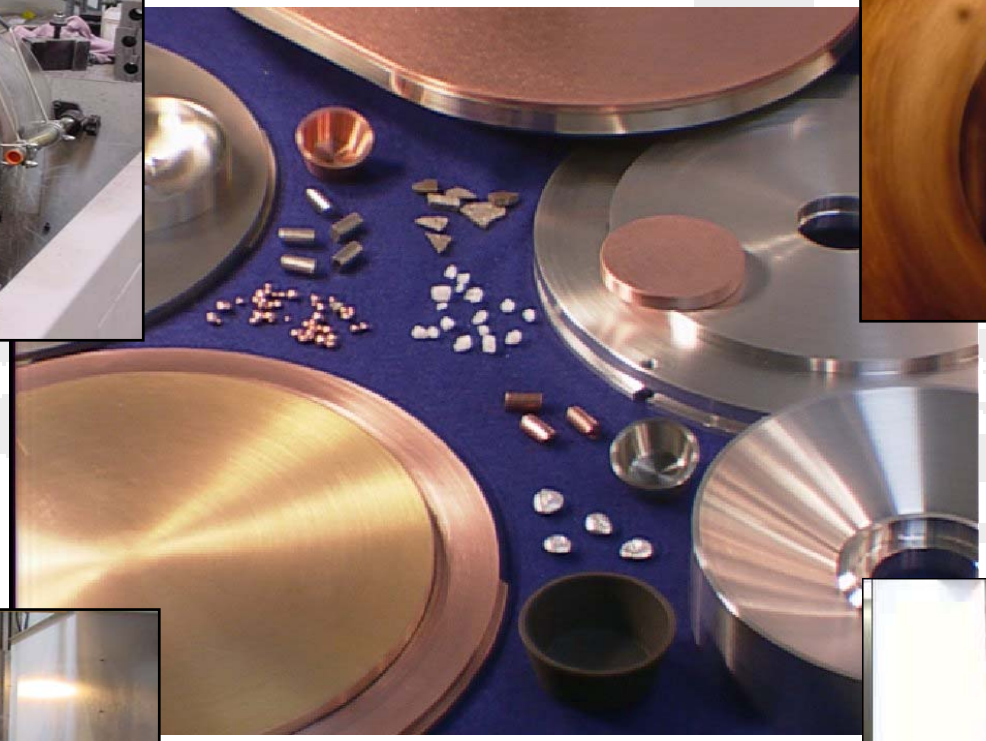
- (NYSE - BW) of Cleveland, OH USA
- Global Business w/ $500KK +/- Combined Annual Sales

Williams ADVANCED MATERIALS

# WAM - Thin Film Products

- **Brewster, NY USA –** *WAM-TFP*
  - 35,000 $ft^2$ with vacuum melting, hot-pressing, milling, hot & cold rolling, automated CNC machining and target bonding capabilities.
  - Class 100 clean room with CMM & Automated Cleaning
  - Acreage to more than double our facility as required.

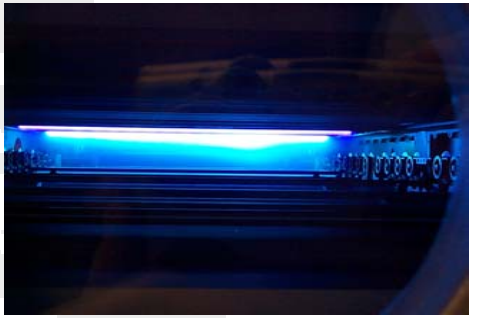
# Target Manufacturing Capabilities



# *Service and Support*

- Regional Offices **(Sales and Applications Engineering support)**

| | | |
|---|---|---|
| Buffalo, NY | (manufacturing) | Manila, Philippines |
| Brewster, NY | (manufacturing) | Buellton, CA /JV |
| Philippines | (manufacturing) | London, England |
| Singapore | (manufacturing) | Guadalajara, Mexico* |
| Ireland/JV | (bonding center) | Dallas, Texas |
| Taiwan | (bonding center) | Philadelphia, PA |
| Korea | (bonding center) | Tucson,AZ |
| Santa Clara, CA | (bonding center) | Shanghai, China |



- **Worldwide Representatives**

| | | |
|---|---|---|
| Florida | Italy | France |
| India | China | Germany |
| Japan | Netherlands | New England |

# Definitions

**PVD -** *Physical Vapor Deposition*

- a coating process whereby the deposition species are transferred and deposited in the form of individual atoms or molecules

**Sputtering –**

- the bombardment of a solid surface with a flux of energetic particles (ions) that results in the ejection of atomic species

**Evaporation –**

- the transfer of material to form a coating by physical means alone, essentially vaporization

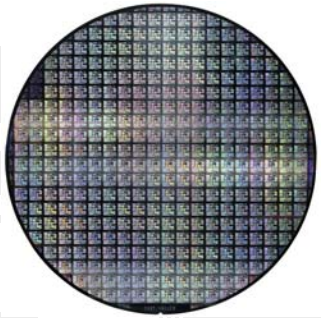
Metal Handbook, Desk Edition, 1998, ASM Intl.

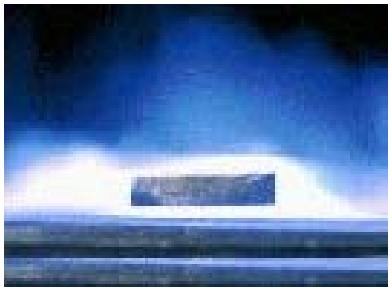
# ***Why Sputtering?***

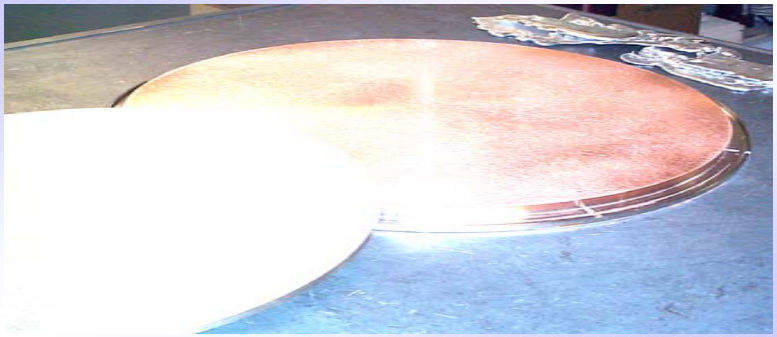
1. Improved Adhesion
   - The kinetic energy of sputtered atoms or molecules are orders of magnitude greater than of other deposition processes
2. Improved Stoichiometry
   - Can maintain composition when sputtering compounds
3. Improved Coverage of Steps
   - Higher kinetic energy = better step coverage
4. Flexibility
   - Research or volume applications

# ***Applications of PVD***

- CD and DVD reflective layers
- AR coatings
- Semiconductor fabrication
- Decorative coatings
- Hard drives
- LED's

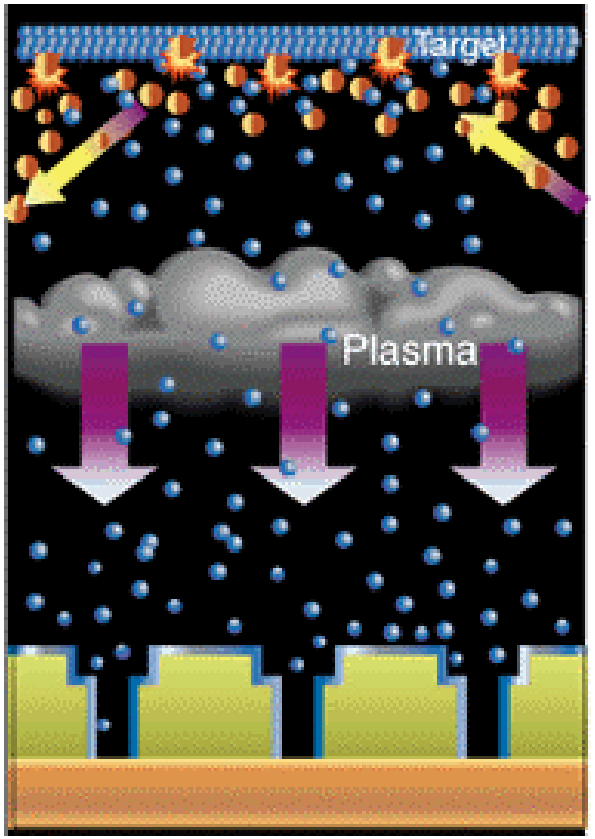






# How Does Sputtering Work?

- The ejection of the source material is accomplished by the bombardment of the surface of the target with gas ions accelerated by a high voltage
- Particles of atomic dimension from the target are ejected as a result of momentum transfer between incident ions and the target



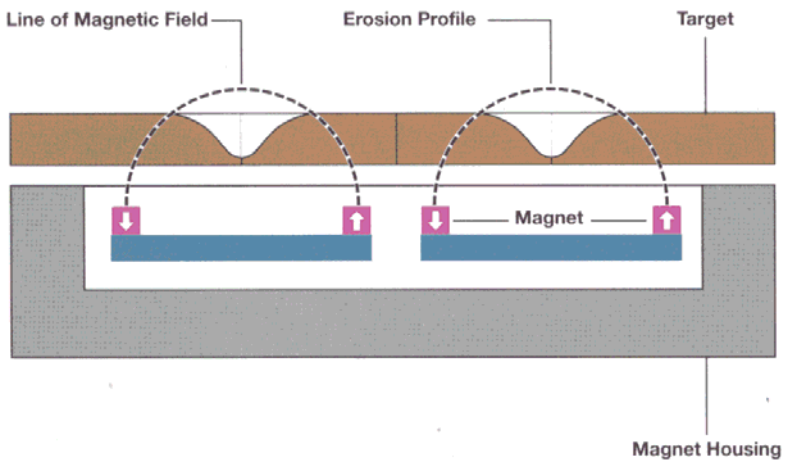

*"Billiard Ball Analogy"*

# ***Other Sputtering Notes***

- Vacuum
  - Sputtering takes place in a low pressure atmosphere of gas (Ar). Argon is used because it is inert, inexpensive and produces reasonable yields of sputtered atoms
  - Chamber is first pumped to a high vacuum to rid the chamber of any background or contaminant gases which would degrade the film
- Power Supply
  - May be DC, RF or a mixture of the two
  - Same purpose – generate plasma

# Magnetron Sputtering

- The rate limiting step in many sputtering systems is the generation of sufficient plasma
- The plasma can be effectively increased by the use of magnets
  - This however affects the erosion of the target and can affect the film uniformity



# ***Metallurgical Considerations***

- The sputtering target specifications are interrelated to the thin film desired
- Specifications for a sputtering target often include the following:
  - Dimensional
  - Composition
  - Impurities
  - Grain size
  - Orientation
  - Pass Through Flux (PTF)

# Dimensional

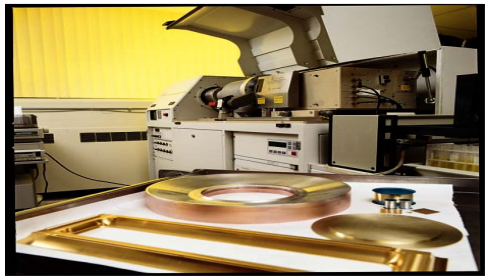
**Proper dimensional tolerances must be held to ensure target fit, proper cooling and general target performance**

- CNC machining used whenever possible
- Intensive quality control measurement

# Compositional & Impurity

**Off composition or high levels of impurities can effect both the sputtering process itself and the material characteristics of the deposited film**

- Must begin with the correct starting material purity
  - Additional impurities may be added during processing
  - Involves QC analysis of all starting materials
- Must use the correct manufacturing technology
  - Vacuum induction melting
  - Inert gas hot pressing
  - Compatible crucible materials
  - Material segregation
- QC analysis of all outgoing products

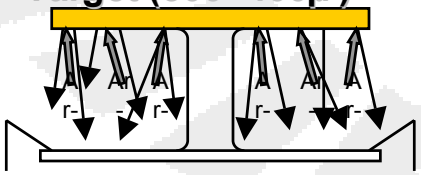


# Grain Size

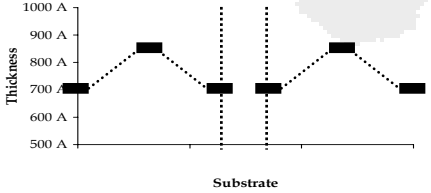
**The grain size of a sputtering target can have a profound impact on the film uniformity of a sputtered film**

## FILM UNIFORMITY

**Standard Grain Size Target (300 -400µ )**

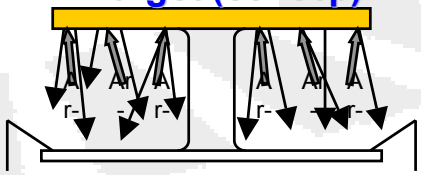


**Thickness Profile Across Substrate**

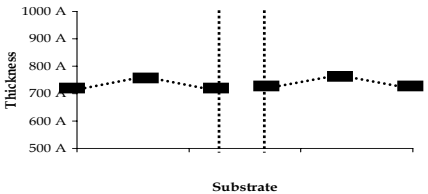



**Uniformity of +/- 8 to 10%**
**Δ Thickness of 100 to 150 A**

**Super Fine Grain Size Target (50 -80µ)**



**Thickness Profile Across Substrate**



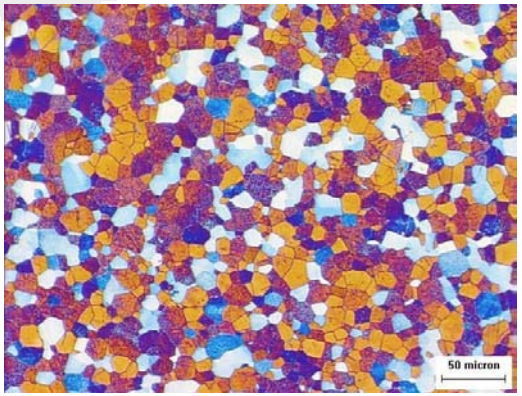

**Uniformity of +/- 3 to 4%**
**Δ Thickness of 40 to 50 A**

# Grain Size Con't

Grain size is controlled in 2 separate ways (melted targets)

1. Casting Technology
   - Effects starting grain size and uniformity
   - Especially important for brittle materials - VCT™
2. Thermo mechanical processing (TMP)
   - Grain size is controlled by the correct sequence of rolling and annealing schedules
     - ❖ Percent reduction, number of reductions
     - ❖ Annealing time, temperature, atmosphere

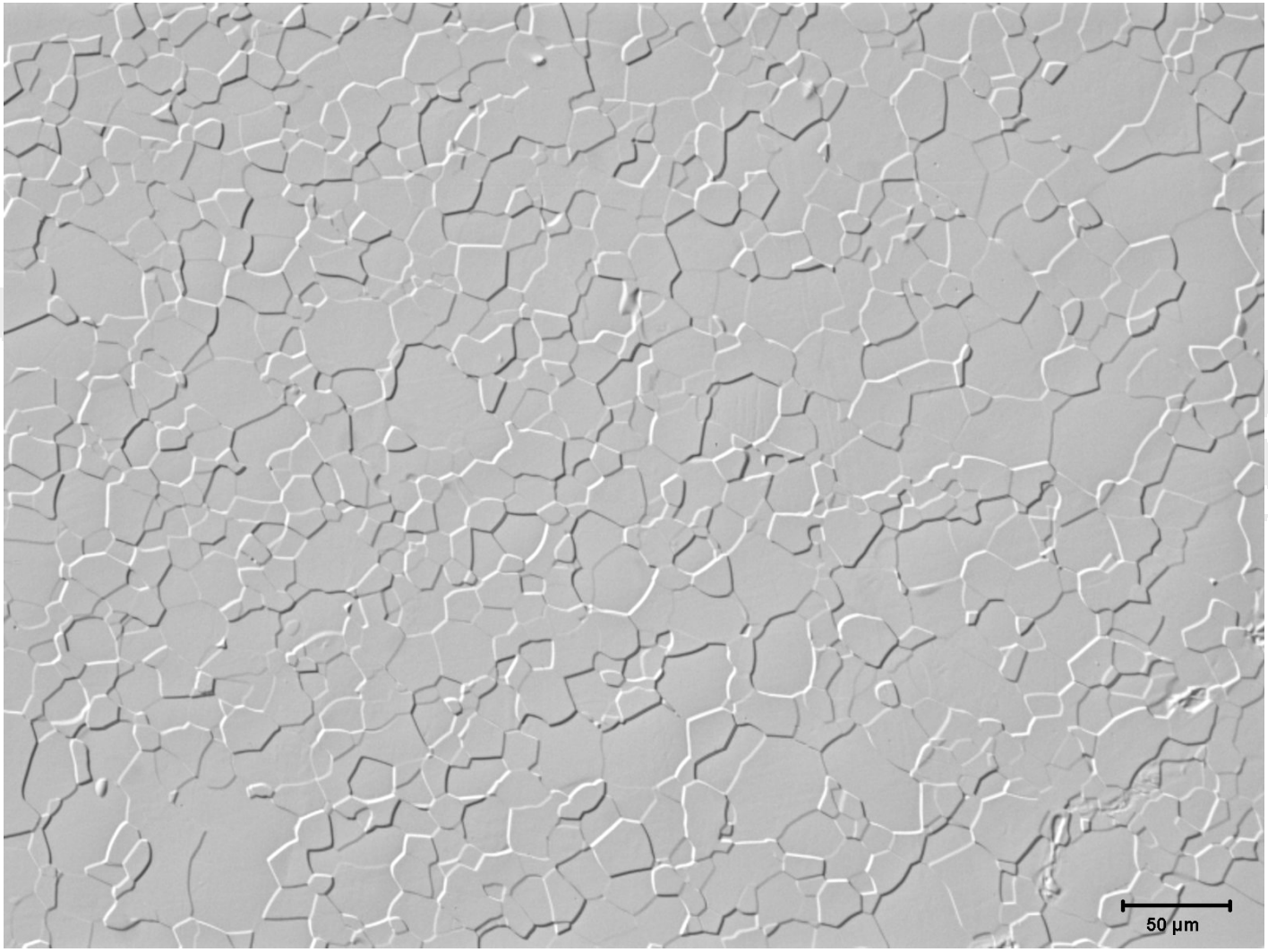
- Grain size is checked using metallurgical preparation techniques combined with image analysis



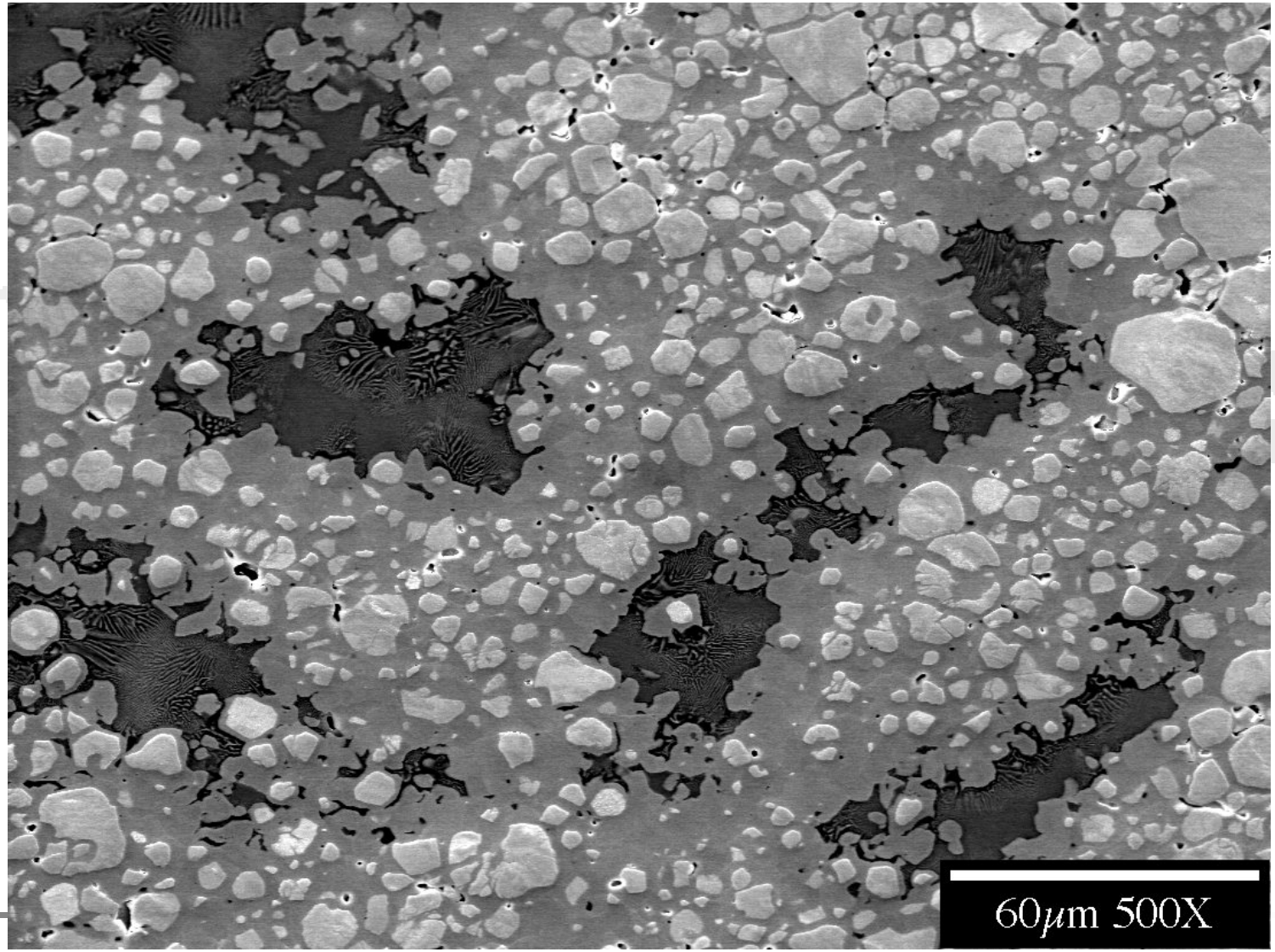
# Grain Size (Powder Methods)

- Inert Gas Hot Press
  - Powder Processing
    - High Melting Temperature
      - W/Ti, Ruthenium
    - Unworkable Alloys
      - Fe/Si/B/Nb/Cu
    - Ceramics
- Hot Isostatic Pressing (HIP)
- Controlled by the Mesh Size of the Powder
- Lower Purity
  - Non-metallics

# Random/Equiaxed Grains



# Grain Size (Powder Process)

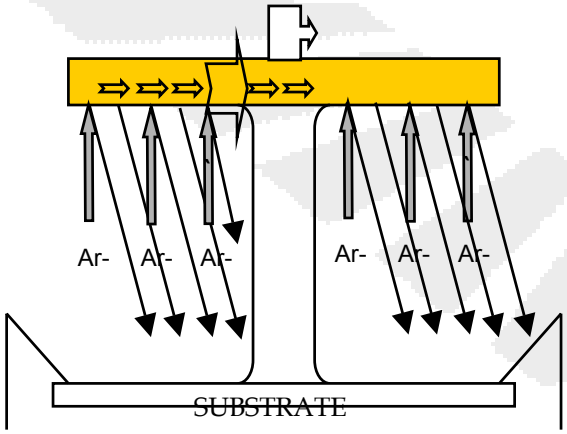


# Orientation

**The crystallographic orientation of the target can also effect the deposited film properties. The general goal is to have a target that has a completely random grain orientation.**

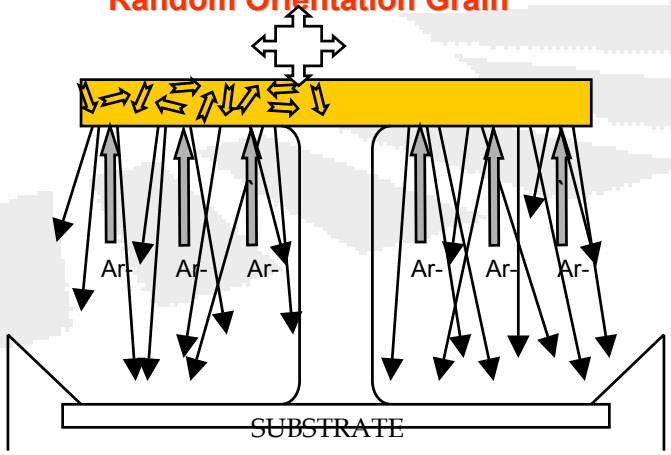
**Standard Target**

**Unidirectional Orientation Grain**




**SFG ™ Target**

**Random Orientation Grain**




**One Direction Sputtered Atoms Ejection**

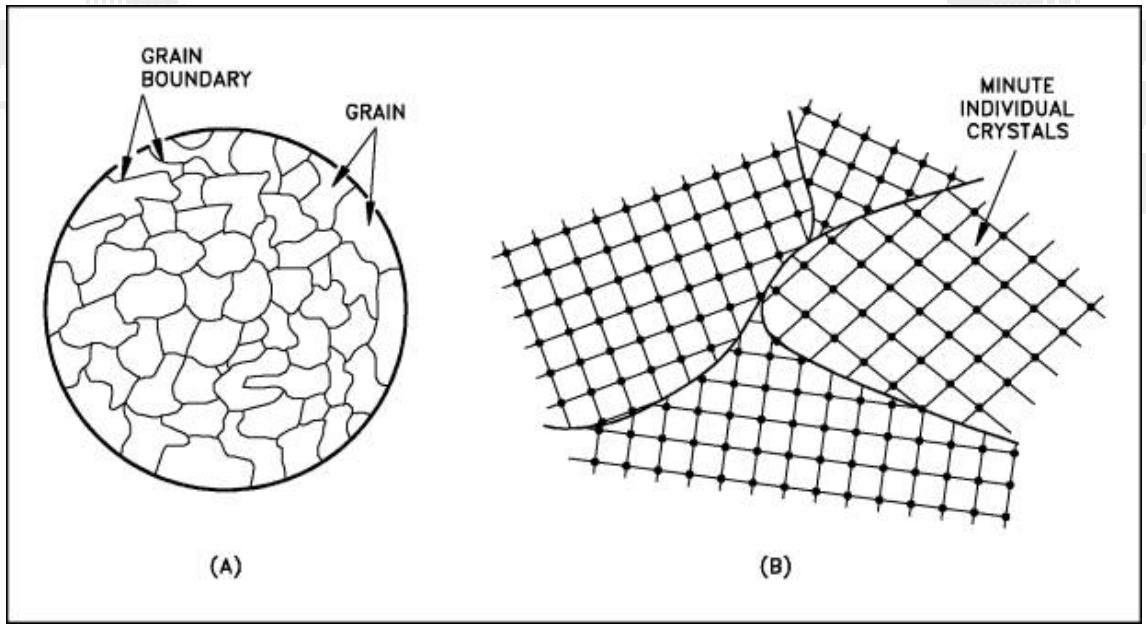
**Unidirectional Grain results:**
**Unidirectional Deposition**

**Random Sputtered Atoms Ejection**

**Random Grain results:**
**Random Deposition**

# CRYSTALLOGRAPHY
## Grain Structure

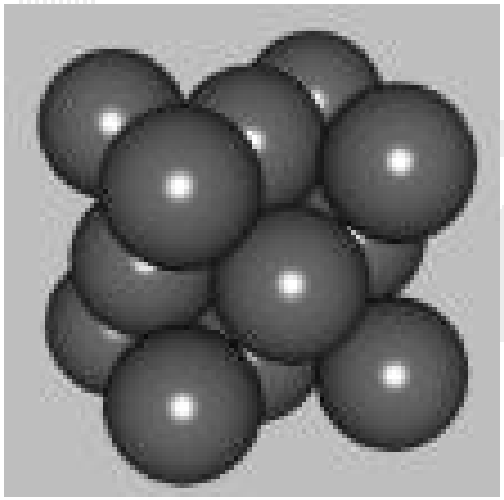
- Wrought Target Materials are Polycrystalline
- Each Grain is an Individual Crystal with a Specific Orientation in Space
- Grain Boundaries are Misfit Regions where Crystals Impinge



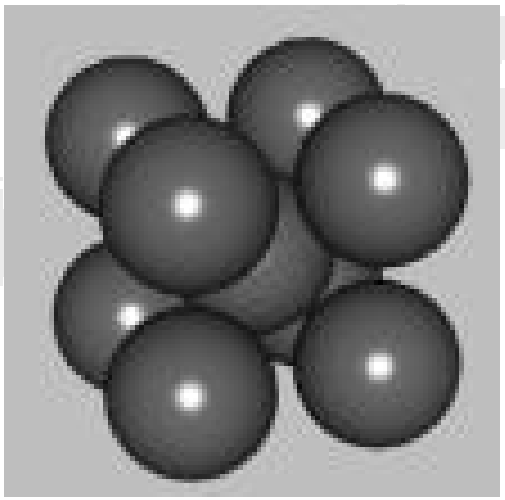
# CRYSTALLOGRAPHY

## Unit Cells (Cubic Metals)

- The Most Basic Arrangement of Atoms in a Crystal Structure is Called A *Unit Cell*
- Expanding the Unit Cell in 3 Dimensions Creates a Crystal Lattice



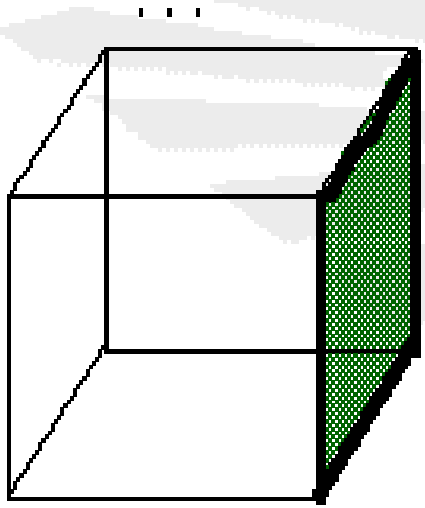
***Face Centered Cubic (FCC)***



***Body Centered Cubic (BCC)***

# CRYSTALLOGRAPHY

## Identifying Crystal Planes: Miller Indices



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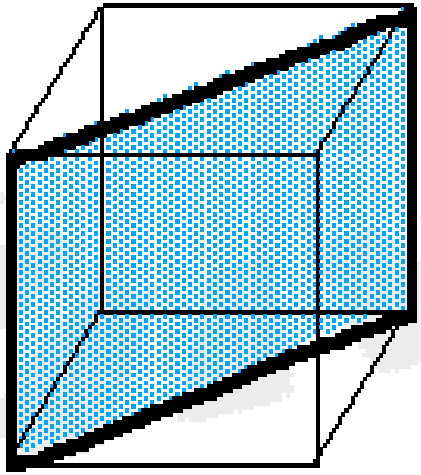

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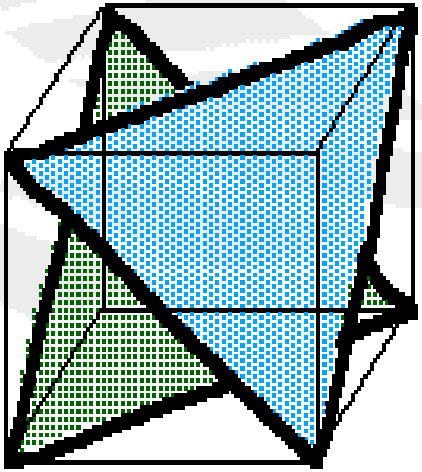


111

# SPUTTERING THEORY

## Orientation Effects On Sputtering

- Planes having High Packing Density of Atoms have High Sputter Yield
- Planes having an Open Atomic Packing Density are Most Transparent to Incident Ions and have Low Sputter Yield

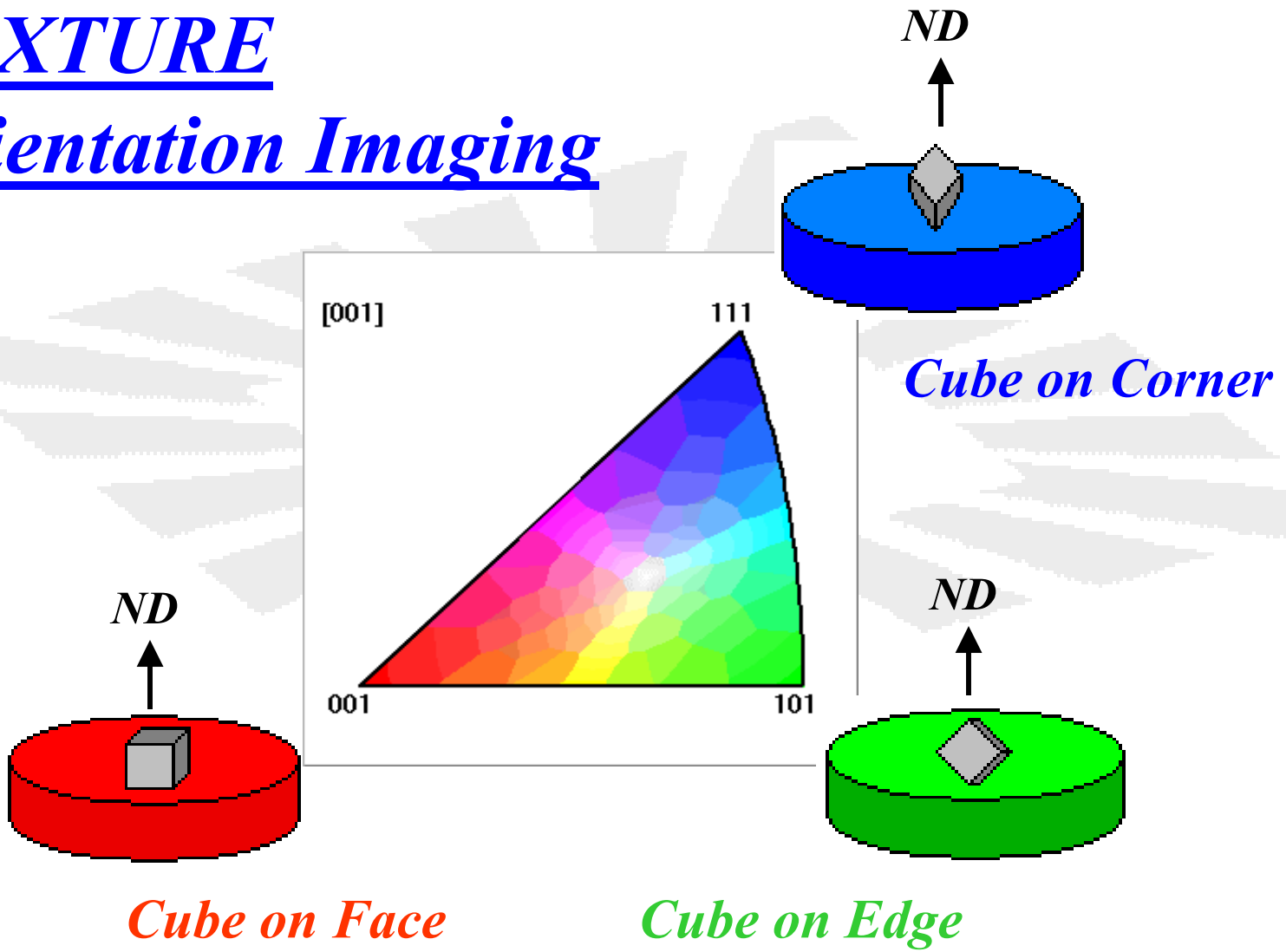
***For FCC:*** $S_{(111)} > S_{(100)} > S_{(110)}$

***For BCC:*** $S_{(110)} > S_{(100)} > S_{(111)}$

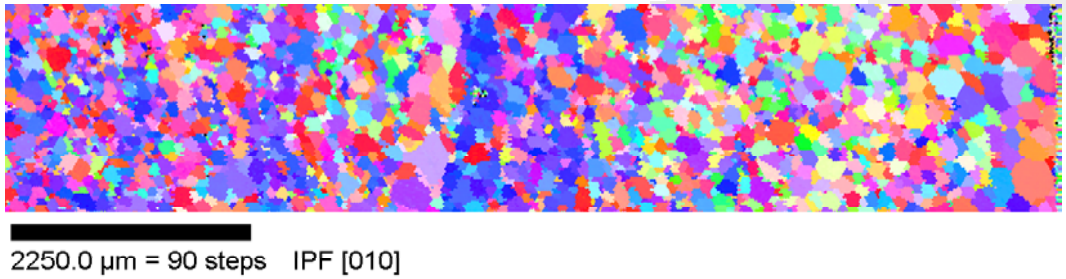
# ***TEXTURE***
# ***Orientation Imaging***

***Desirable Ta Target*** ***Undesirable Ta Target***

# TEXTURE
# Orientation Imaging

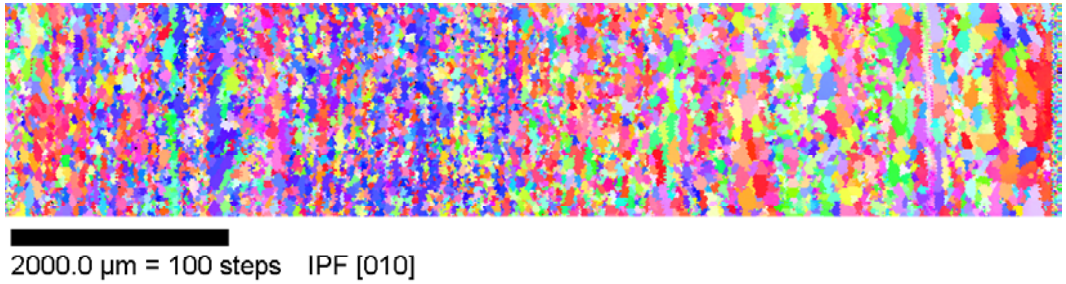


# TEXTURE
# Orientation Imaging




*Desirable Target*

*$\Omega$=88.8/mm $\Lambda$=5.16/mm*




*Undesirable Target*

*$\Omega$=265/mm $\Lambda$=20.8/mm*

***NOTE: Undesirable Target had FINER Grain Size but a Less Homogeneous Texture than the Desirable Target***

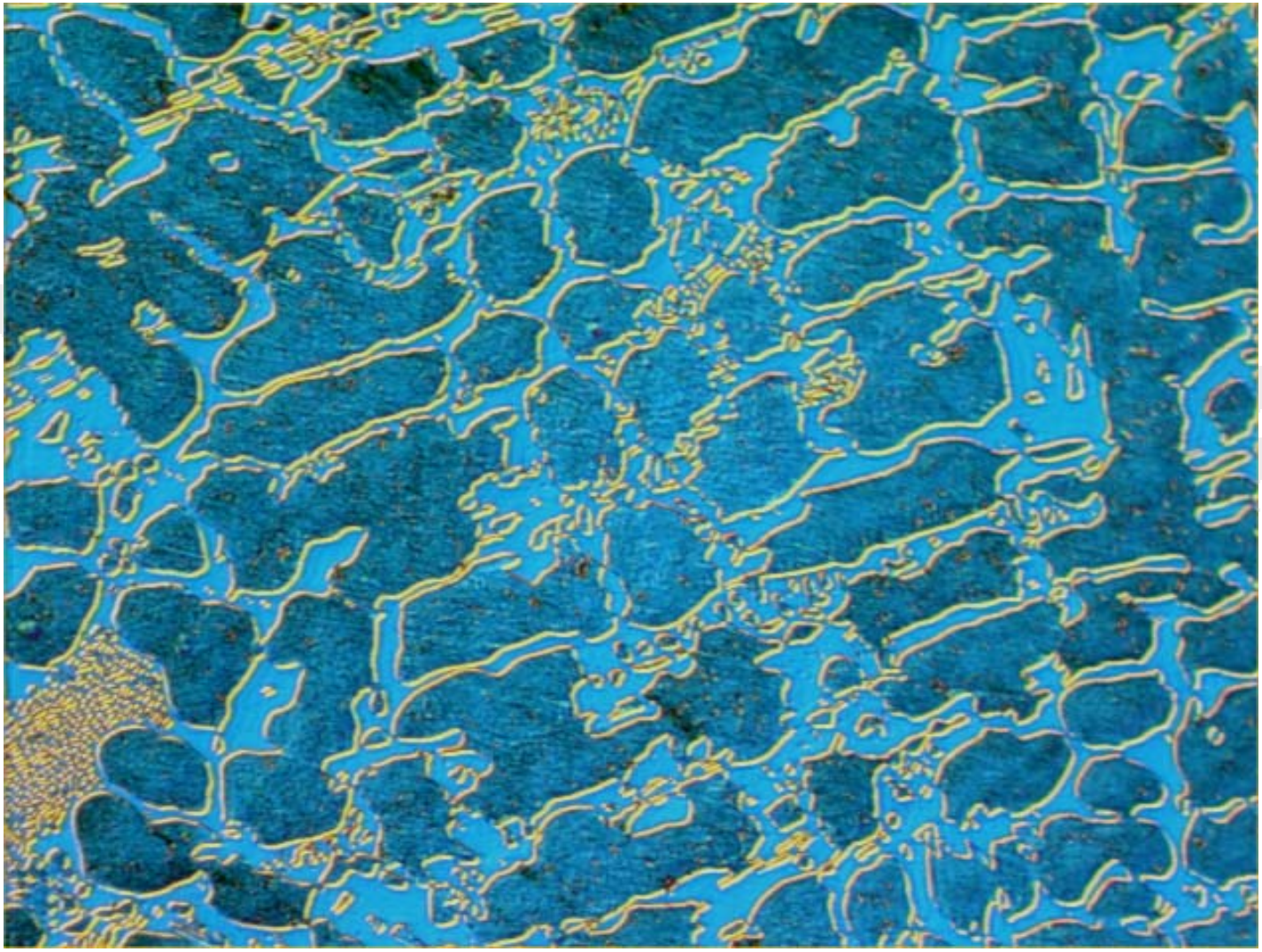
# ***Orientation*** *Con't*

Orientation is controlled by:

1. Hot rolling of targets
   - Dynamic Recrystallization
   - Random Orientation
2. Rotational rolling of targets
   - Target is rolled and rotated at specific intervals to promote randomness

➢ X-Ray Diffraction (XRD)/EBSD
   - Can be used to confirm a random orientation

# CoZrTa – Magnetic Alloy



# ***Pass Thru Flux***

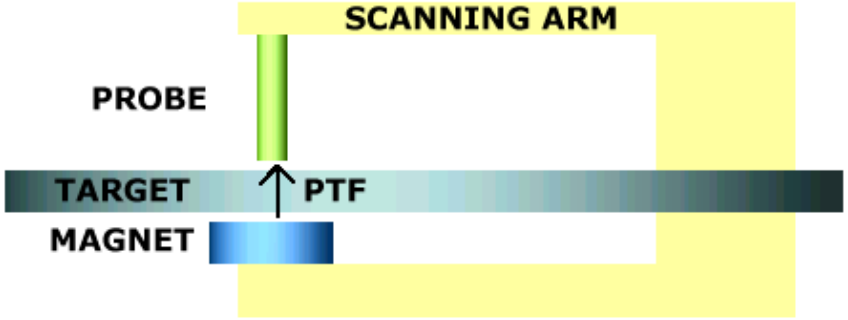
- When magnetron sputtering is used with a ferromagnetic sputtering target the pass through flux (magnetic lines) becomes critical to the performance of the system.
  - NiFe
  - CoFe
  - CoTaZr

- Also effects sputter target material utilization

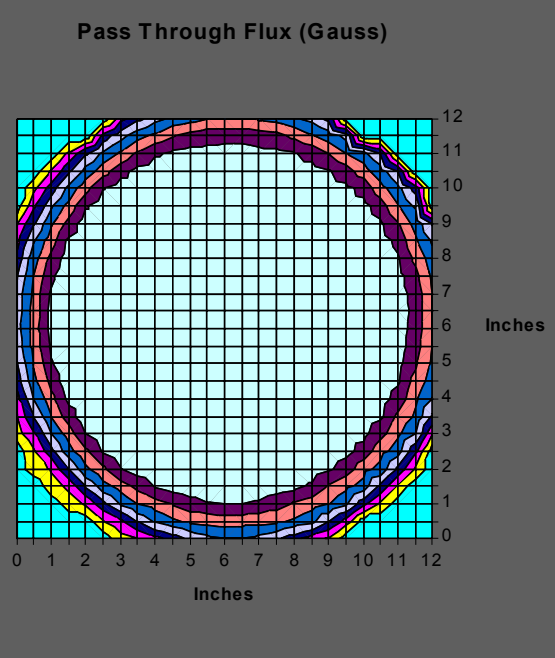
# ***Pass Through Flux (PTF)***

- Variations in the PTF can arise from
- Target thickness
  - Must keep target as perpendicular to cathode as possible
- Alloy uniformity
  - Alloy uniformity can affect PTF
- Processing history
  - Different processing techniques (ex. Rolling) can affect the PTF of the sputtering target

# Pass Through Flux (PTF)




## PTF Map

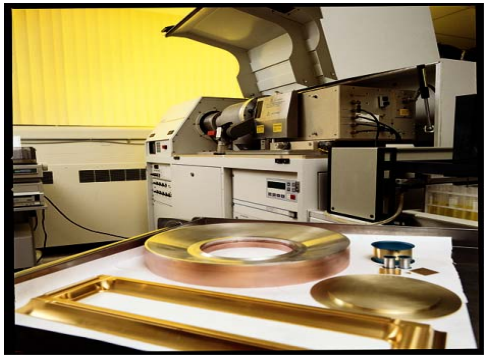


# *Summary - Product Attributes*

- **High Quality**
  - Uniform small grain size (SFG™)
  - High PTF
  - Low Alkali Content
  - Low Gas Content
  - Highest Purity
  - ISO 9001
- **Full Trace Ability**
  - Bar Coding
  - Lot Numbers, Work Orders
- **SPC Data Collection**
  - Grain Size
  - Processing Parameters
  - C of A

- **Quality Documentation**
  - Compositional and Trace Analysis
  - MSDS
  - Grain Size
  - Comprehensive Packing Slip
  - Ultrasonic C-Scan for Bonded Targets
  - PTF Mapping

# Integrated Analytical Capabilities



- Fully Integrated Analytical Lab
  - ICP- Mass Spectrometer (2)
  - ICP-CID Spectrometer (2)
  - DC Arc Spectrometer
  - LECO - Carbon, Oxygen, Nitrogen
  - Full Metallurgical Lab including Image Analysis
  - DSC / DTA – Melting Range
  - Surface Profilometer
  - Hardness Testing (Rockwell and Micro-hardness)
  - Fire Assay - Refines
  - GDMS (2005)
  - CMM
  - Pass Through Flux (full face PTF)
  - University of Buffalo for additional services

# Nanotechnology will extend CMOS scaling

90nm Node
2003

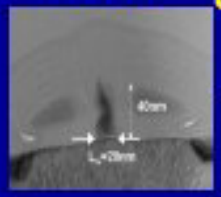
50nm Length
(IEDM2002)

65nm Node
2005

30nm
Prototype
(IEDM2000)

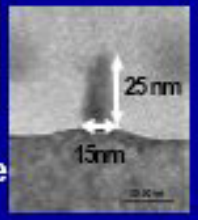
---2003

45nm Node
2007



20nm Prototype
(VLSI2001)

32nm Node
2009




15nm Prototype
(IEDM2001)

---2012

22nm Node
2011

10nm Prototype
(DRC 2003)

Nanotubes, nanowires

1-3nm

16 nm node
2013

7nm

---2013 –2017

11nm node
2015

5nm

8 nm node
2017

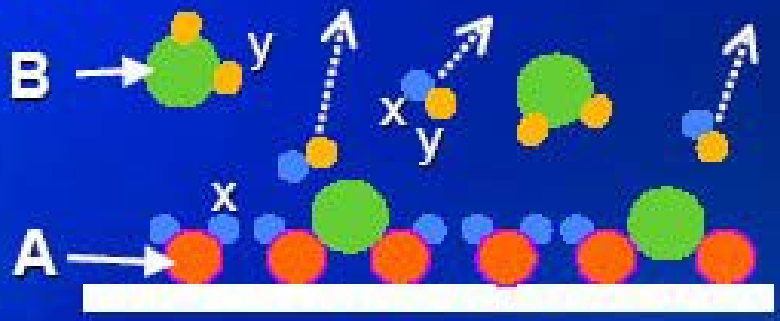
3nm

Innovations like quantum dots,
Nanowires, Naotubes, etc.

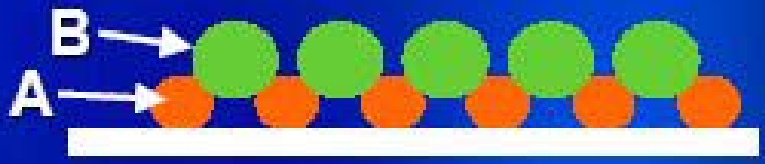
# Crafting Films with Atomic Layer Deposition


Step 1


Step 2

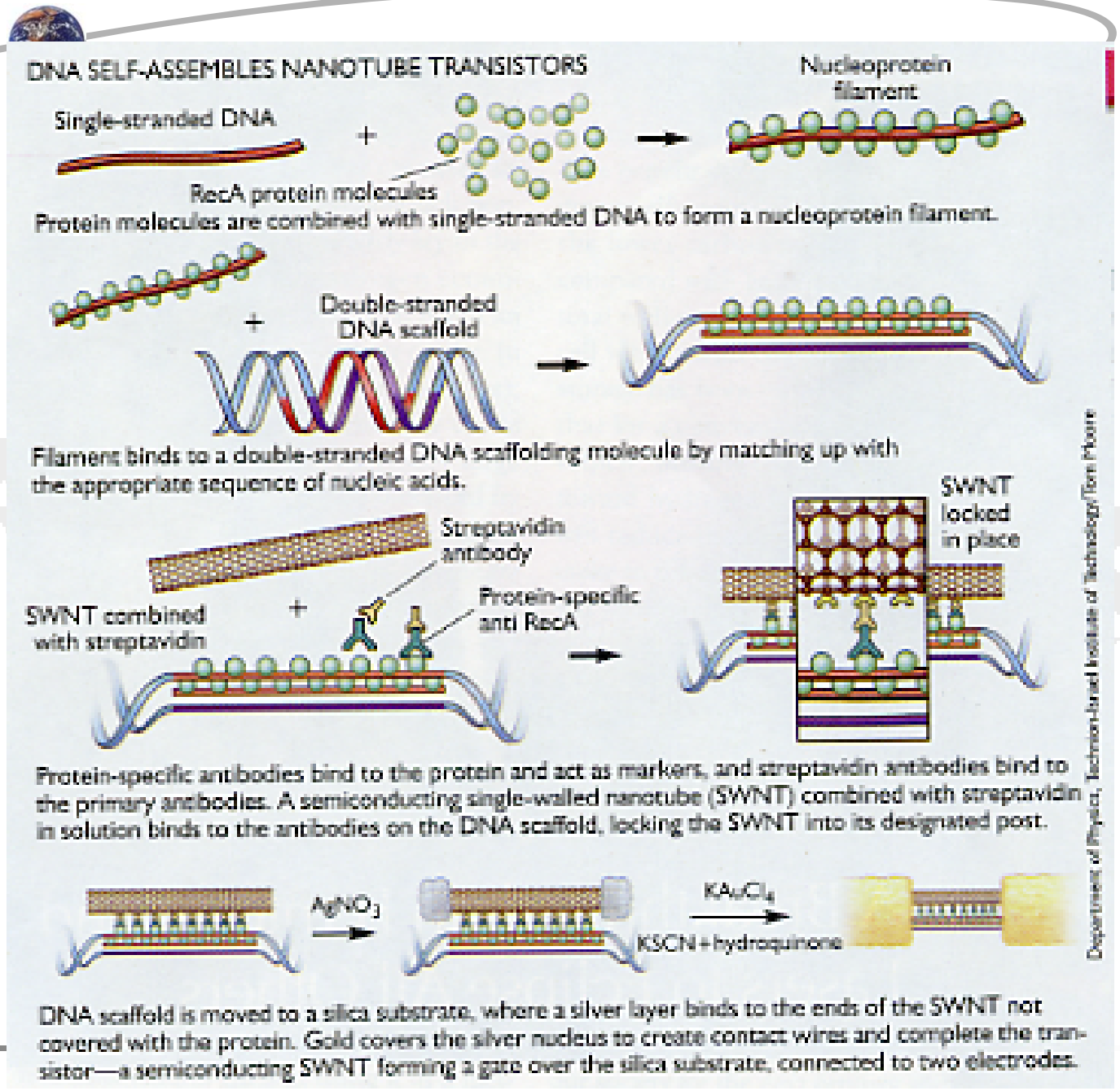



Step 3




Step 4

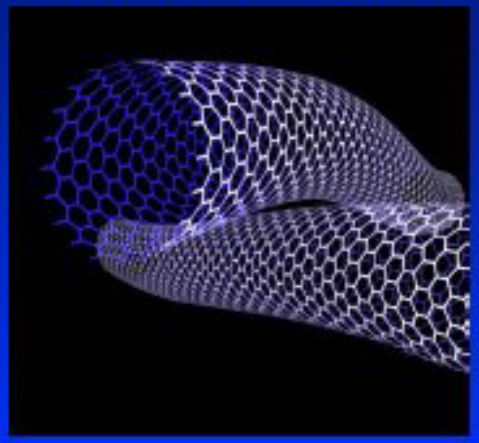
**ALD: Today's nanotechnology for self-assembly by atomic layer**


DNA SELF-ASSEMBLES NANOTUBE TRANSISTORS
Single-stranded DNA
RecA protein molecules
Nucleoprotein filament
Protein molecules are combined with single-stranded DNA to form a nucleoprotein filament.
Double-stranded DNA scaffold
Filament binds to a double-stranded DNA scaffolding molecule by matching up with the appropriate sequence of nucleic acids.
Streptavidin antibody
SWNT combined with streptavidin
Protein-specific anti RecA
SWNT locked in place
Protein-specific antibodies bind to the protein and act as markers, and streptavidin antibodies bind to the primary antibodies. A semiconducting single-walled nanotube (SWNT) combined with streptavidin in solution binds to the antibodies on the DNA scaffold, locking the SWNT into its designated post.
$AgNO_3$
$KAuCl_4$
KSCN + hydroquinone
DNA scaffold is moved to a silica substrate, where a silver layer binds to the ends of the SWNT not covered with the protein. Gold covers the silver nucleus to create contact wires and complete the transistor—a semiconducting SWNT forming a gate over the silica substrate, connected to two electrodes.
Department of Physics, Technion-Israel Institute of Technology/Tom Moore

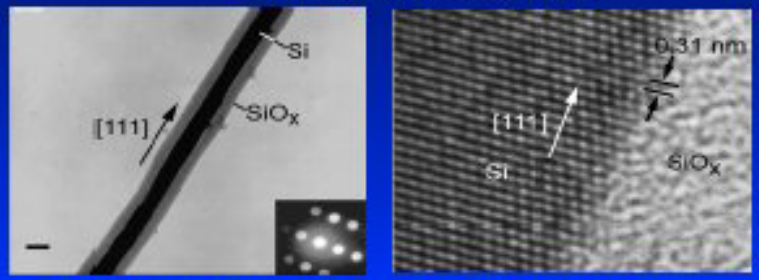
# Nanotubes/Nanowires ( >> 2010?)

- Collaborations with universities in progress
- Good individual device data, many integration and materials issues to be resolved

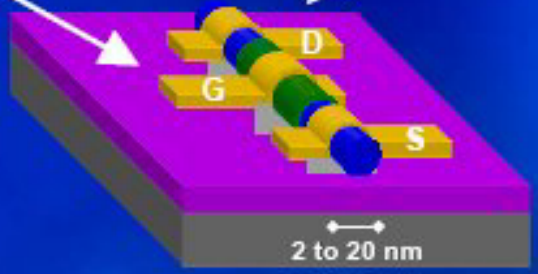
## Carbon Nanotube



## Silicon Nanowire

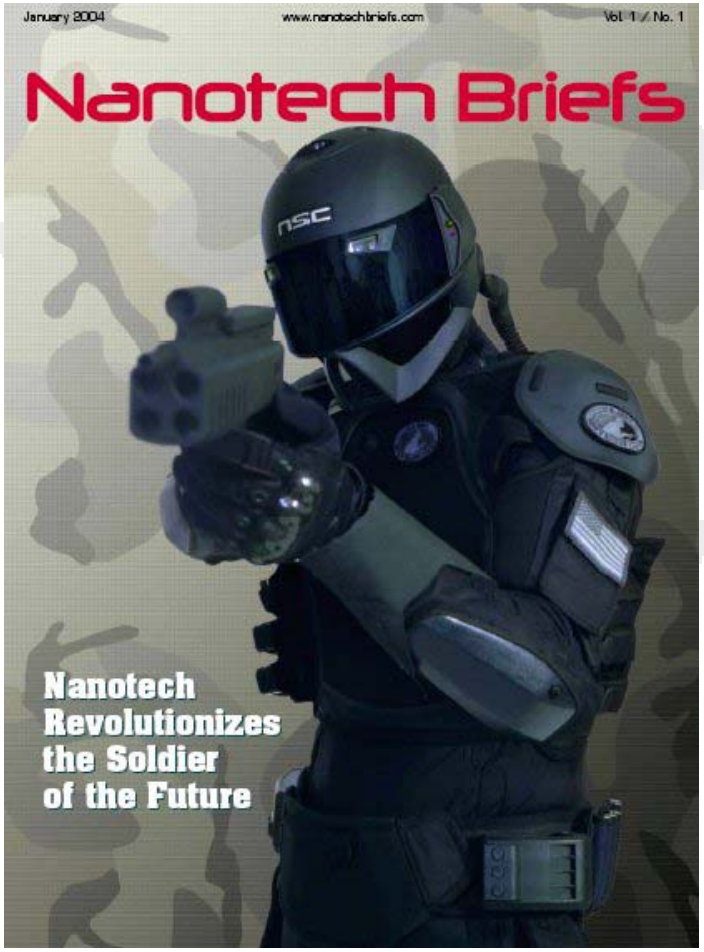



Source: Morales & Lieber, Science **279**, 208 (1998)




intel.

# Soldier with Nano Gear




# *Open Discussion*

# *Q & A*