Sputtering Tutorial: Thin Film Metallization on AlN and Al_2O_3

CMC Laboratories, Inc.

Review of the Sputtering Process

Key Characteristics of Sputtering

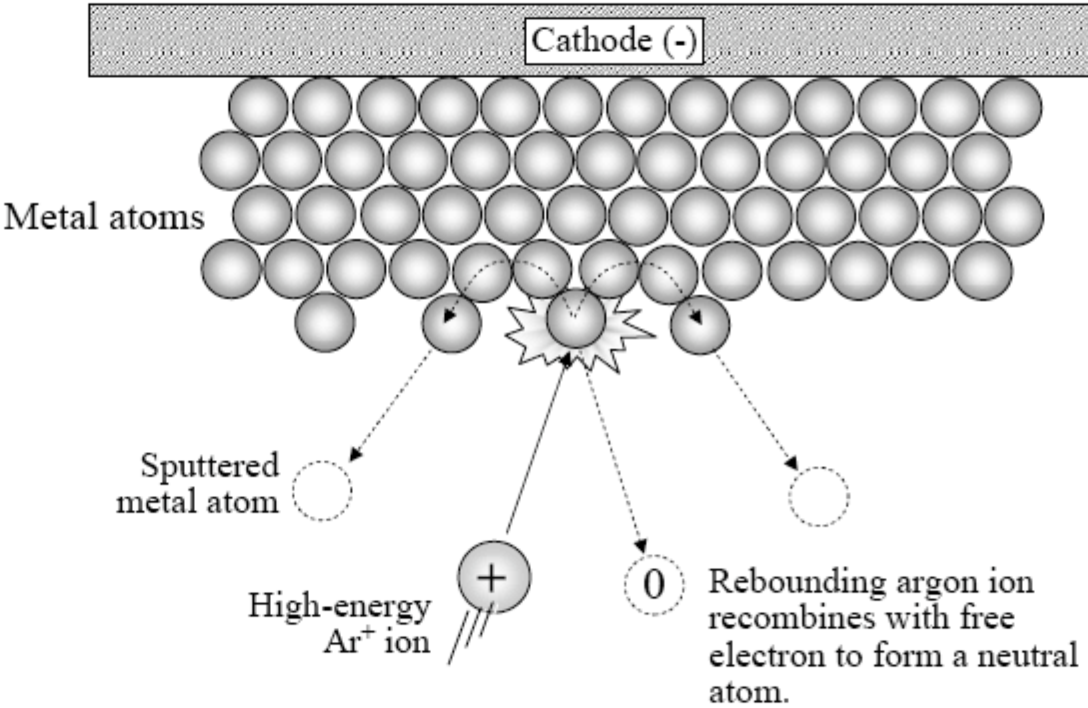
- Atom-by-atom deposition technique
- Atoms generally have little energy when deposited- easy to produce non-equilibrium phases (atoms trapped at their deposition site)
- Almost any solid material can be sputtered- very versatile
- Film morphology (grain size and shape, porosity, surface roughness) is very dependent on deposition conditions

Basic Sputter Deposition Steps

- A high vacuum is generated in the sputter tool to eliminate residual N_2 and O_2. Goal 10^{-7} Torr range.
- The chamber is backfilled with Ar gas in the 10^{-3} torr range
- An Ar plasma is ignited ($Ar^+ + e^-$) by applying an electric field
- A target composed of the intended metal for deposition is biased negatively
- Ar^+ ions attracted to the target collide and dislodge atoms of the target material
- Some of these atoms collide with the substrate material and bond forming a deposited film
- The film is also bombarded with Ar atoms

Target Sputtering Schematic

Dislodging Metal Atoms from Surface of Sputtering Target



Rebounding argon ion recombines with free electron to form a neutral atom.

Sputter Tool Process Schematic

Simple Parallel Plate DC Diode Sputtering System

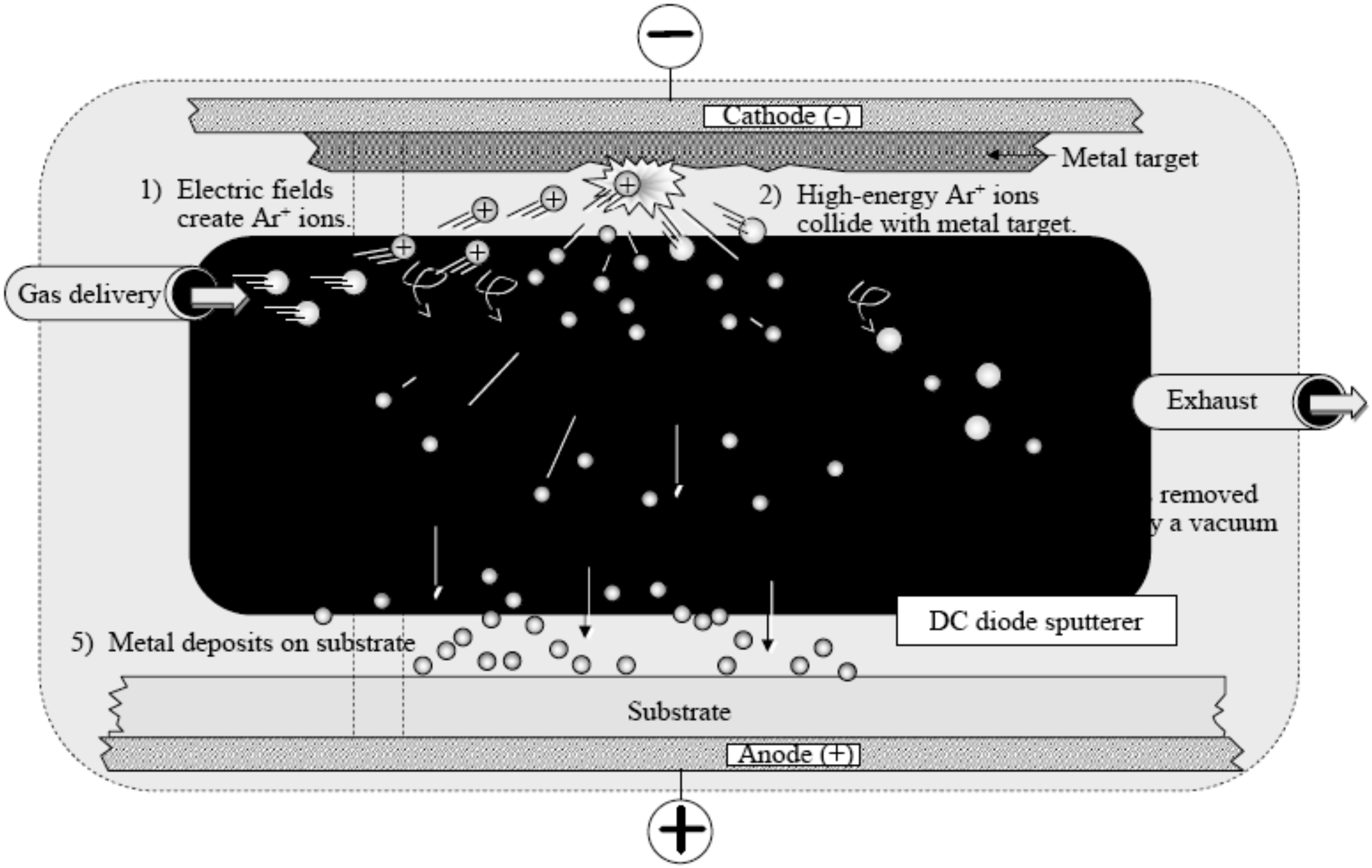
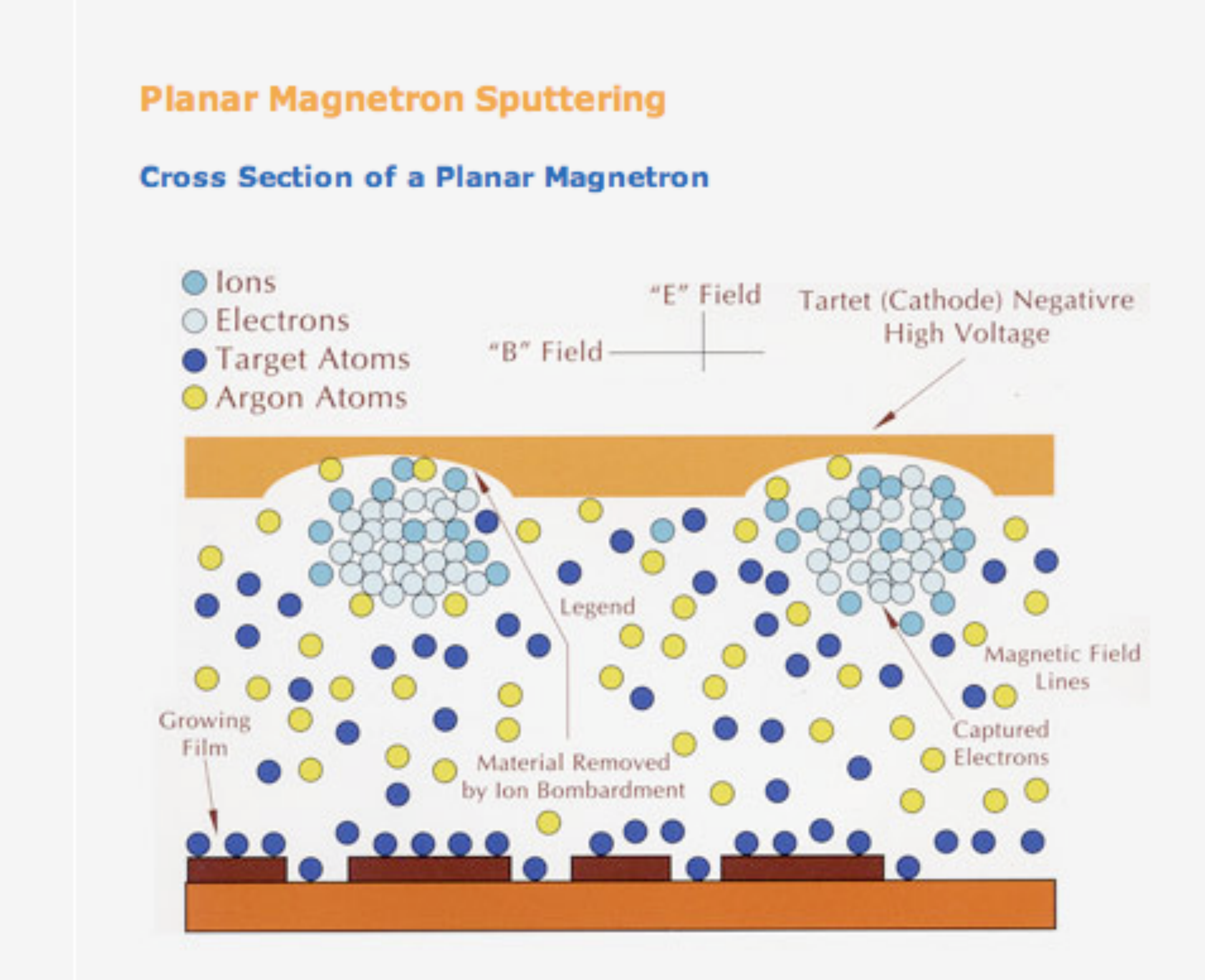


Figure 12.16

Sputter Tool Process Schematic- Details

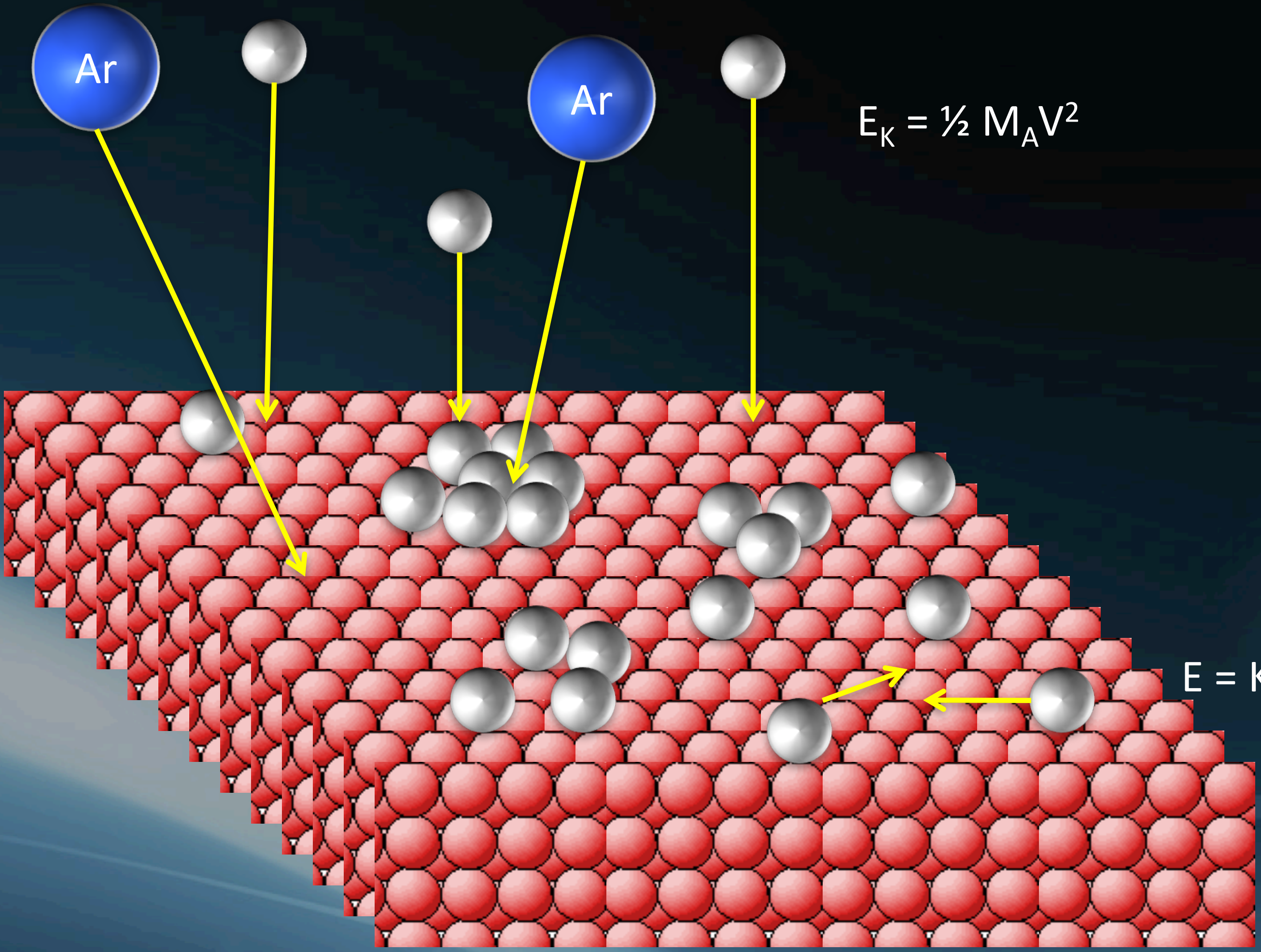


Planar Magnetron Sputtering

Cross Section of a Planar Magnetron

Thin Film Deposition Process Schematic

- Depositing atoms have kinetic energy from the sputter process
- They collide with the substrate
- Some localized heat is created
- Atoms diffuse on the surface to low energy sites
- Diffusion is driven by the substrate temperature and localized heat from collisions
- Small nuclei form that grow and coalesce into a film
- Ar atoms also bombard the surface

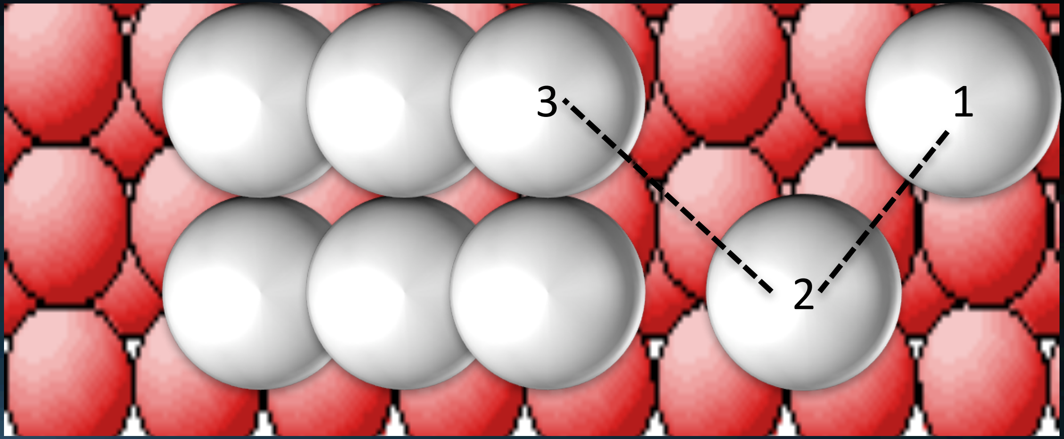


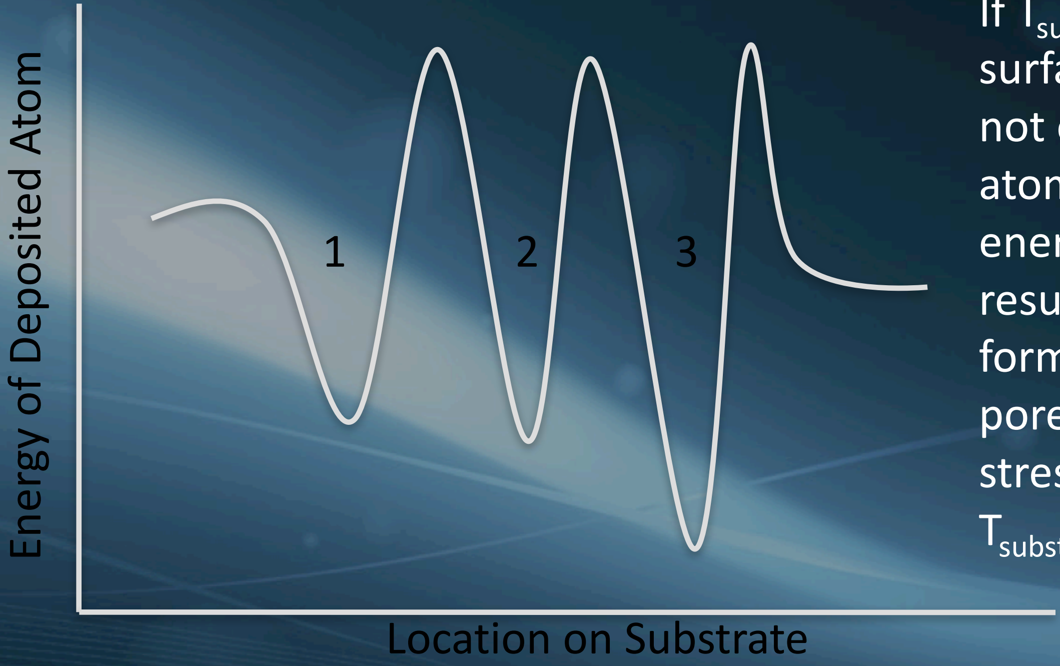
$E_K = ½ M_A V^2$

$E = KT$

$T = T_{Substrate} + T_A$

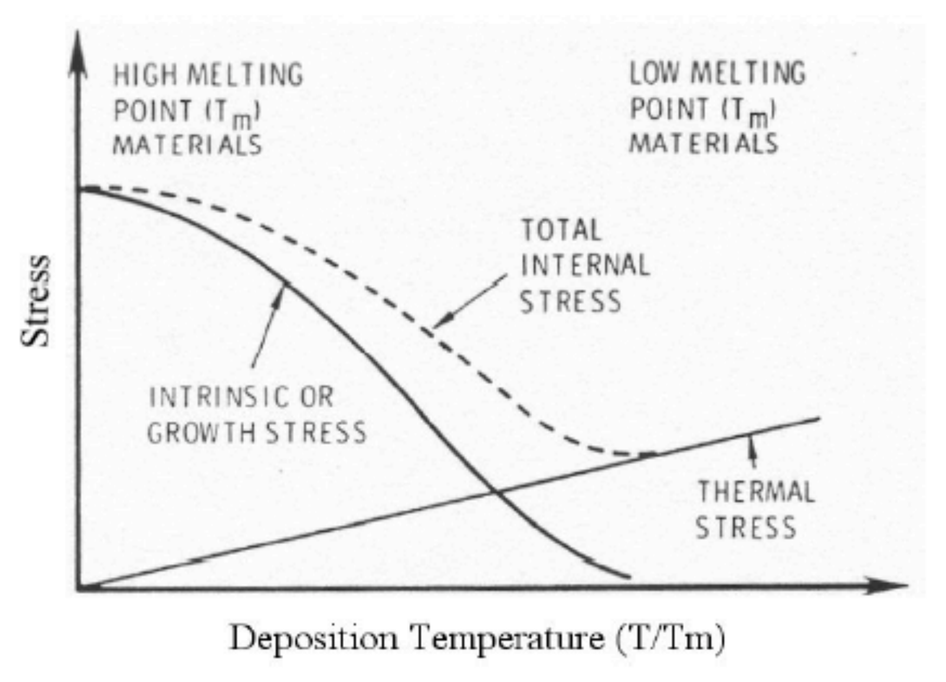
Surface Diffusion Process





If $T_{substrate}$ is too low, surface diffusion does not occur, and deposited atom trapped at higher energy sites 1 or 2. This results in defect formation (such as pores) or intrinsic film stress. Diffusion scales as $T_{substrate}/T_M$.

Film Stress with Substrate Temperature



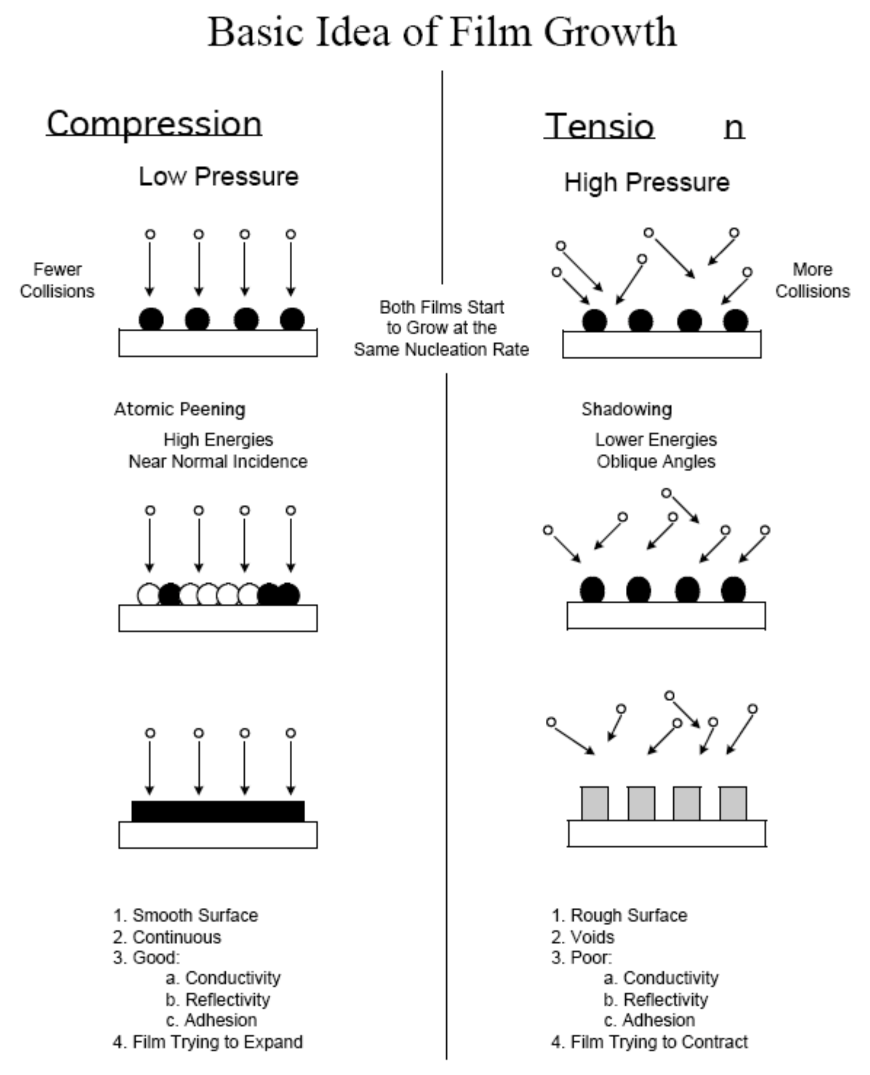
- Intrinsic film stress decreases with increasing $T_{substrate}$
- Relevant parameter to measure diffusion is T/T_M
- With higher $T_{substrate}$, TCE stress between the film and substrate increases
- Trade-off between these 2 factors must be determined experimentally

From Thornton, J.A. and Hoffman, D.W. Thin Film Solids, 171: 5-31 (1989)

Film Growth Mechanisms vs. Argon Pressure

Ar bombardment during film growth

- For low Ar pressure, time between Ar/Ar collisions is long
- Ar mean-free-path is long
- Ar energy bombarding the film surface is high
- "Atomic peening effect" increases film density
- Produces films with compressive stress and smooth surfaces
- For high Ar pressure, Ar mean-free-path is short
- Ar energy is low
- No atomic peening
- Films are low density, tensile stress, rough surfaces



Basic Idea of Film Growth

1. Smooth Surface
2. Continuous
3. Good:
    a. Conductivity
    b. Reflectivity
    c. Adhesion
4. Film Trying to Expand

1. Rough Surface
2. Voids
3. Poor:
    a. Conductivity
    b. Reflectivity
    c. Adhesion
4. Film Trying to Contract

Film Microstructure and Stress Relationship

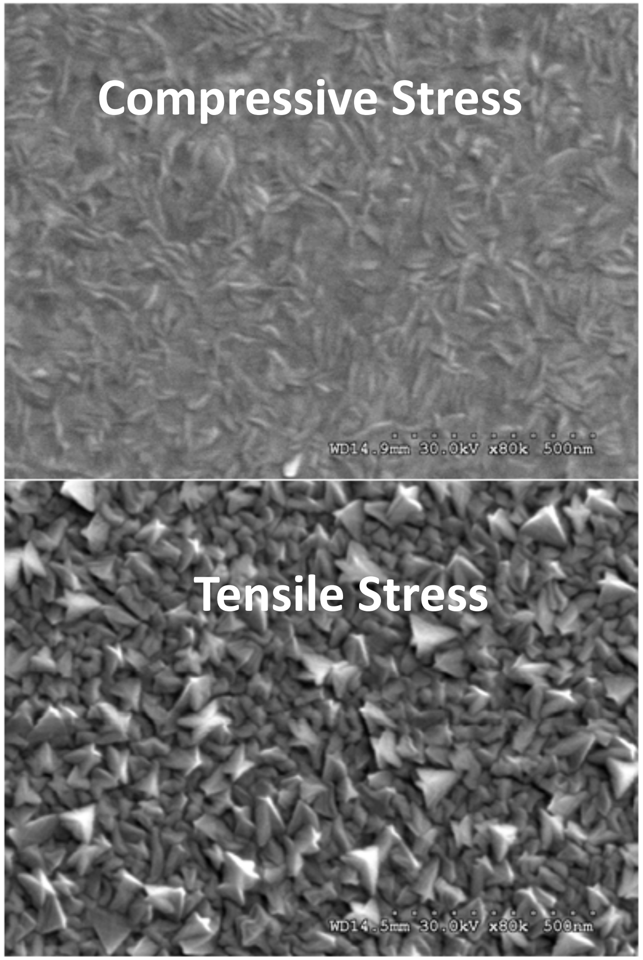


Figure 3: SEM images of approximately 2000 Å thick iron thin films deposited at sputtering chamber pressures of 0.8mTorr (*top*) and 8mTorr (*bottom*). Images were taken using a 30kV electron beam to obtain a magnification of 80,000. Length scales are displayed at the bottom right.

Film Stress vs. Argon Pressure

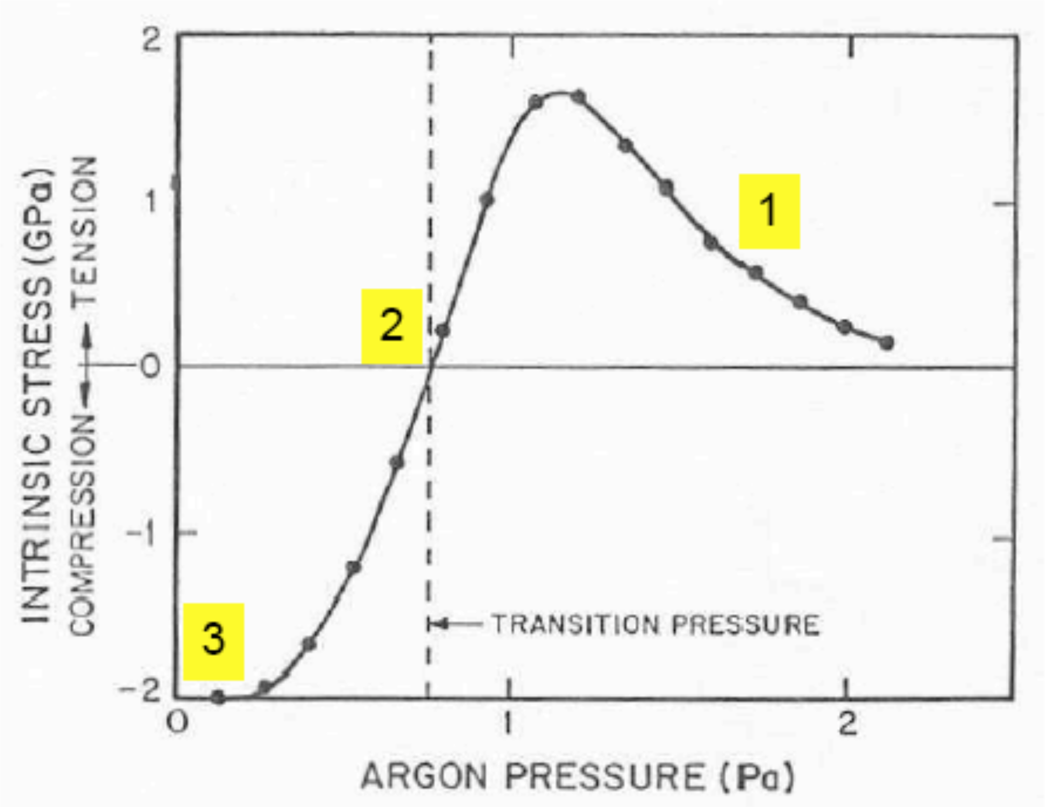
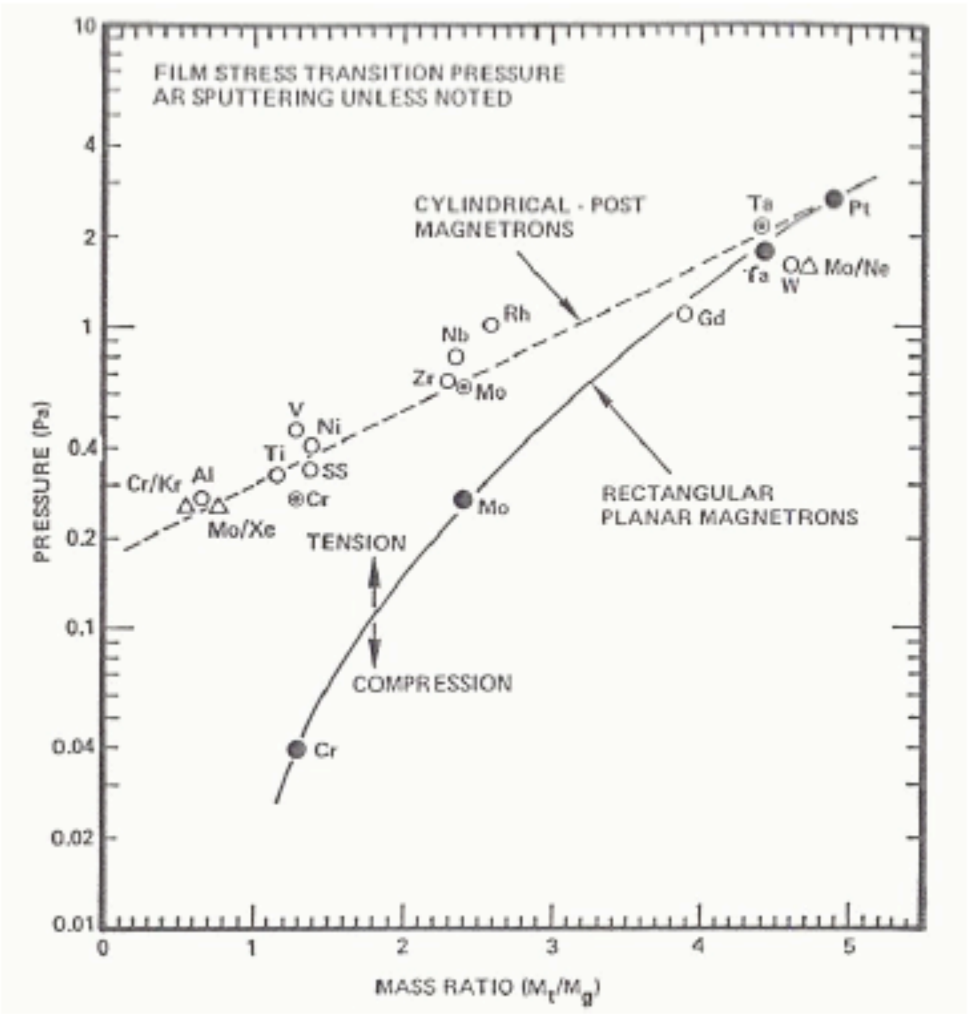
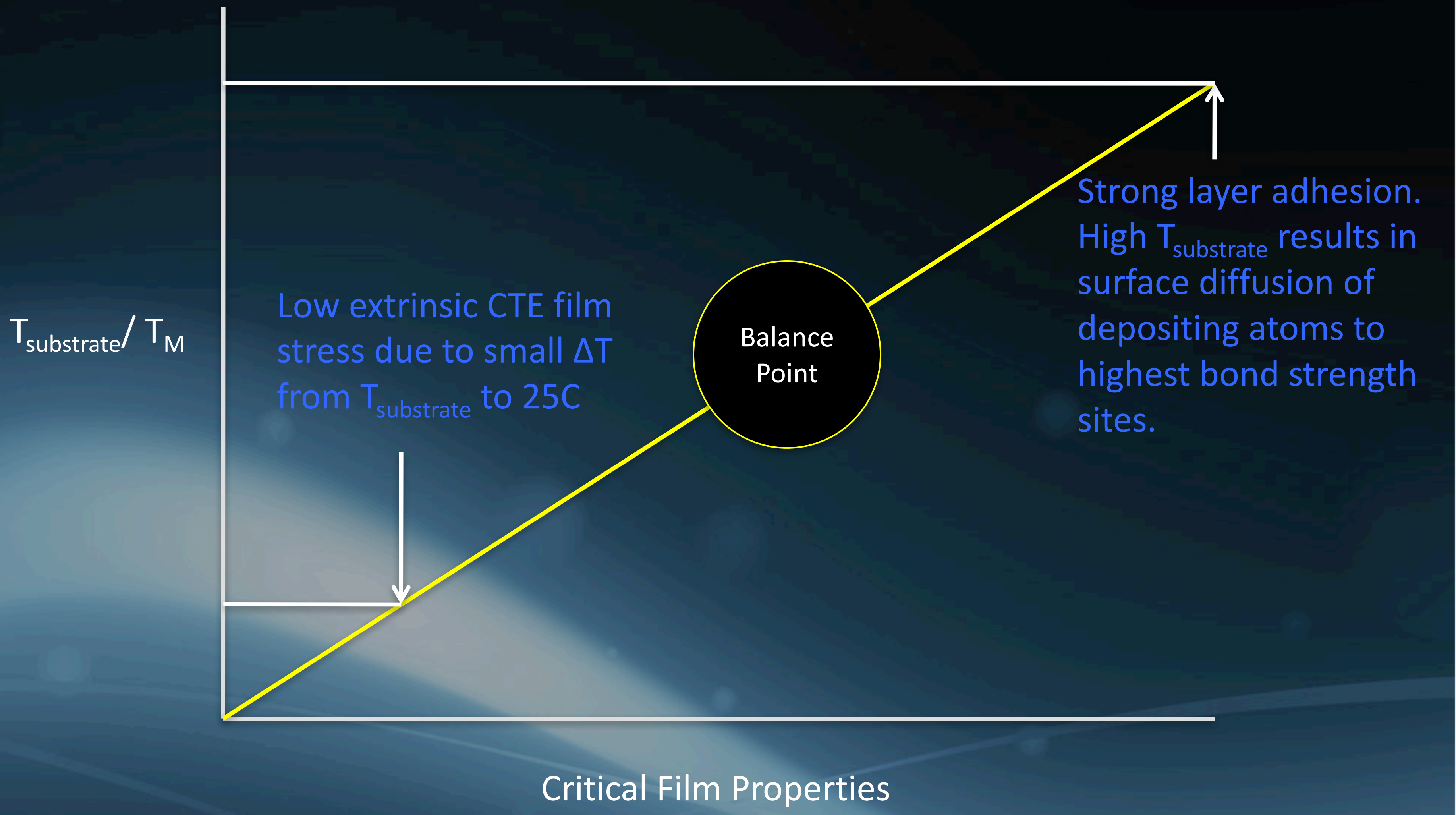


Figure 14: Typical stress-pressure curve for a metal film at low T/T_m deposition temperatures [17]

Stress Transition Pressure for Different Metals



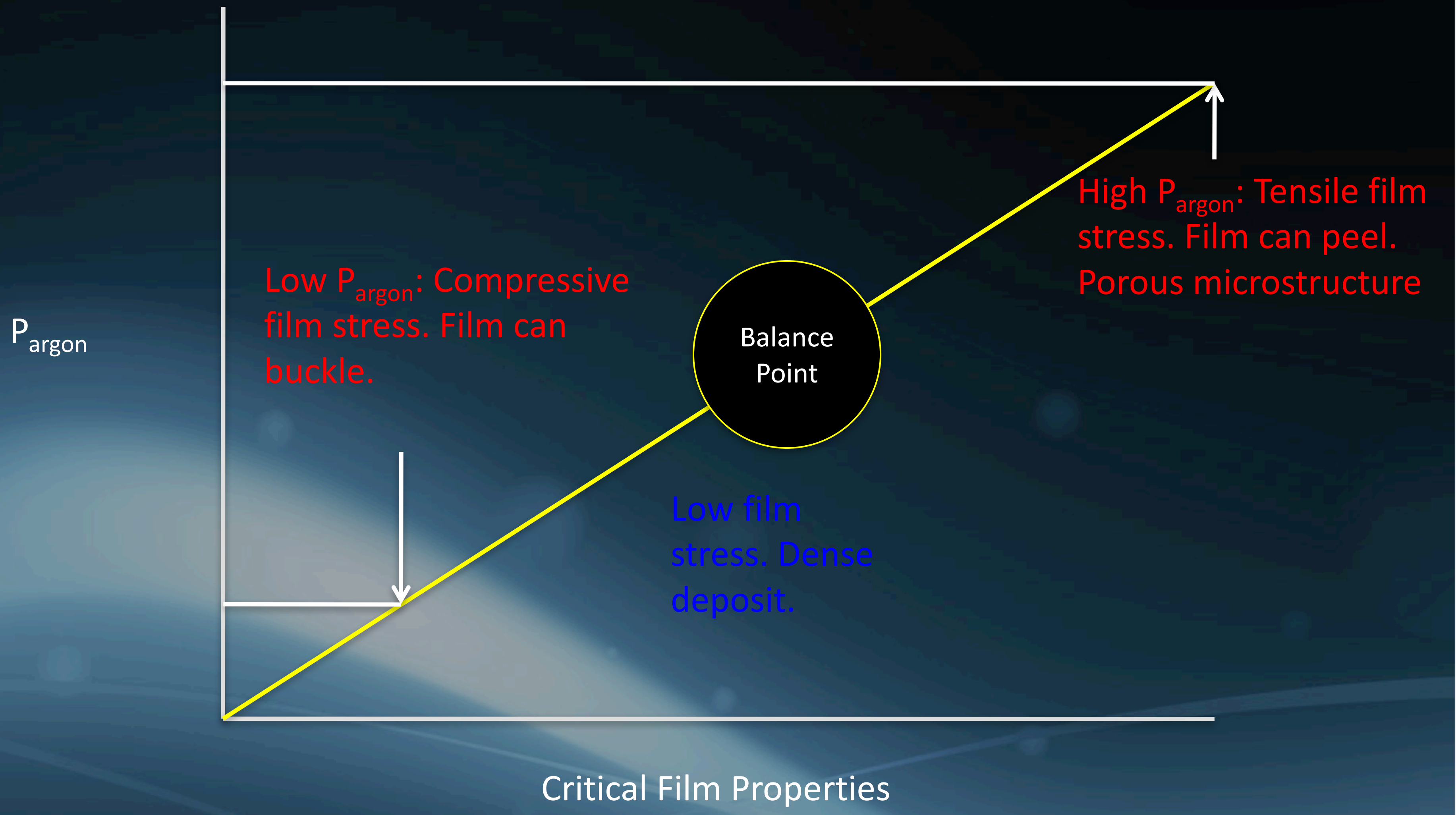
Key Parameters in the Sputter Process- $T_{substrate}$



Low extrinsic CTE film stress due to small ΔT from $T_{substrate}$ to 25C

Strong layer adhesion. High $T_{substrate}$ results in surface diffusion of depositing atoms to highest bond strength sites.

Key Parameters in the Sputter Process- Argon Pressure



P_{argon}

Low P_{argon}: Compressive film stress. Film can buckle.

Balance Point

High P_{argon}: Tensile film stress. Film can peel. Porous microstructure

Low film stress. Dense deposit.

Critical Film Properties

Sputtered Film Microstructure Mapping

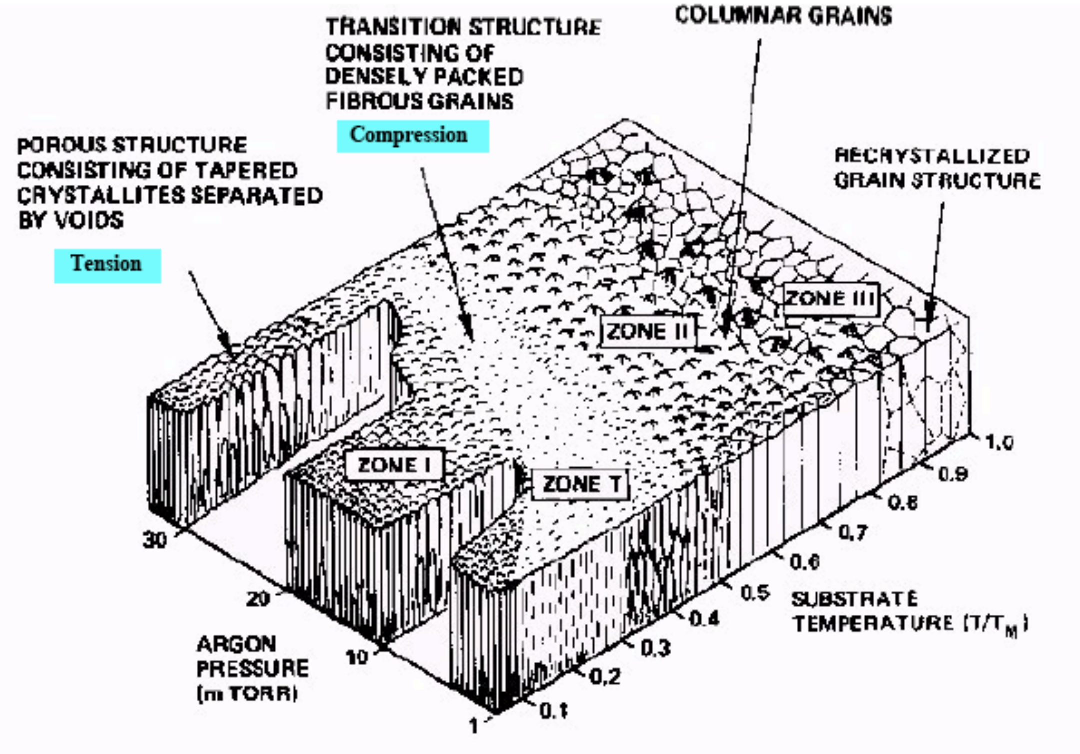


Figure 11: Schematic representation of the influence of substrate temperature and argon pressure on the microstructure of metal coatings deposited by cylindrical magnetron sputtering [12]

Surface Preparation Prior to Sputter Deposition

- Clean off all contamination
  - Organics
  - Polishing or lapping residue
- Etch or anneal away any damaged layers
  - Micro-cracks
  - Created during lapping
  - Created during laser processing

Surface Preparation Prior to Sputter Deposition

Preparation Stage	Processing Approach	Example
Clean Organics	Plasma Clean	O_2 plasma Si wafers
Clean lapping solutions or handling contamination	Alkaline soak	Anodex cleaner
Eliminate Micro-cracks	Etch away the damaged layer	Buffered oxide etch for Si
Eliminate Micro-cracks	Heat treatment near sintering temperature to anneal away cracks	Al_2O_3 T > 1600C, for AlN T> 1750C

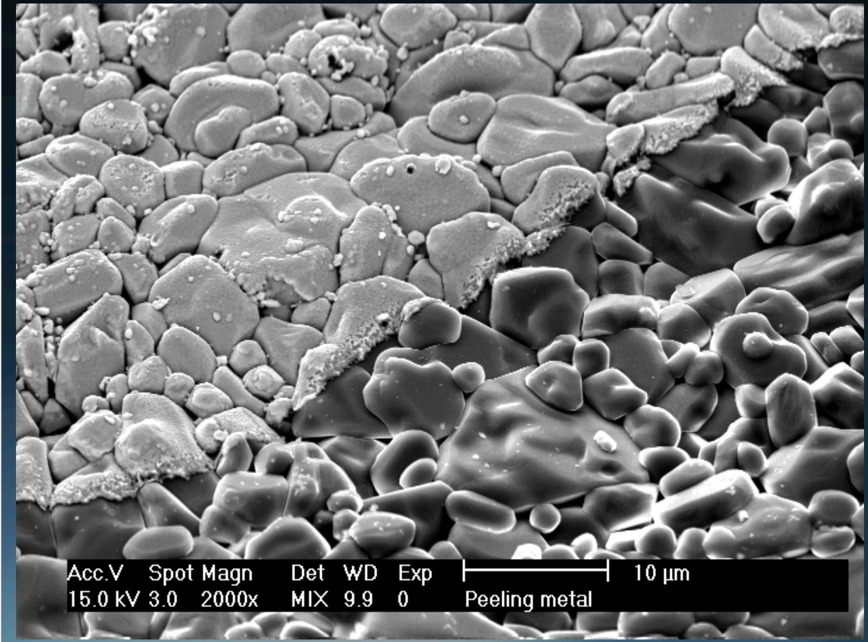
Thin Film Deposition on AlN and Al_2O_3

Goals for Thin Film Deposition on AlN and Al_2O_3

- Thin film adhesion to oxide or nitride ceramic surface
- Strong adhesion between multiple thin film layers
- Acceptable film stress (minimize impact on adhesion)
- Preferred failure mode: ceramic fracture
- Diffusion barrier to prevent Ti or W on solder surface
- Oxide free solder layer
- Compatibility with multiple solders: PbSn, SAC, AuSn

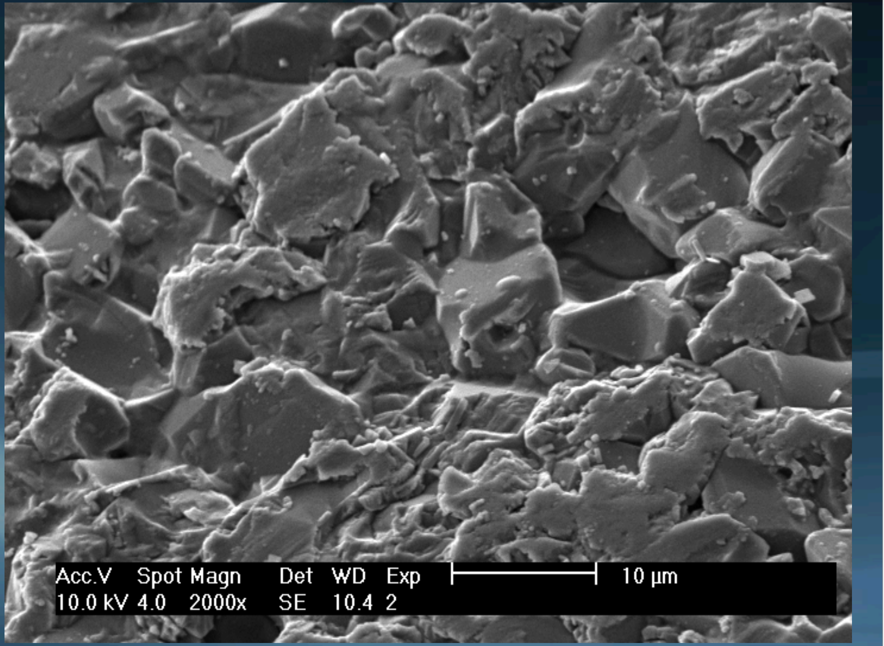
AlN and Al_2O_3 Ceramic Surface Requirements

Al_2O_3 Surface Finish



As-Fired (Ti coating on part of sample)

Lapped



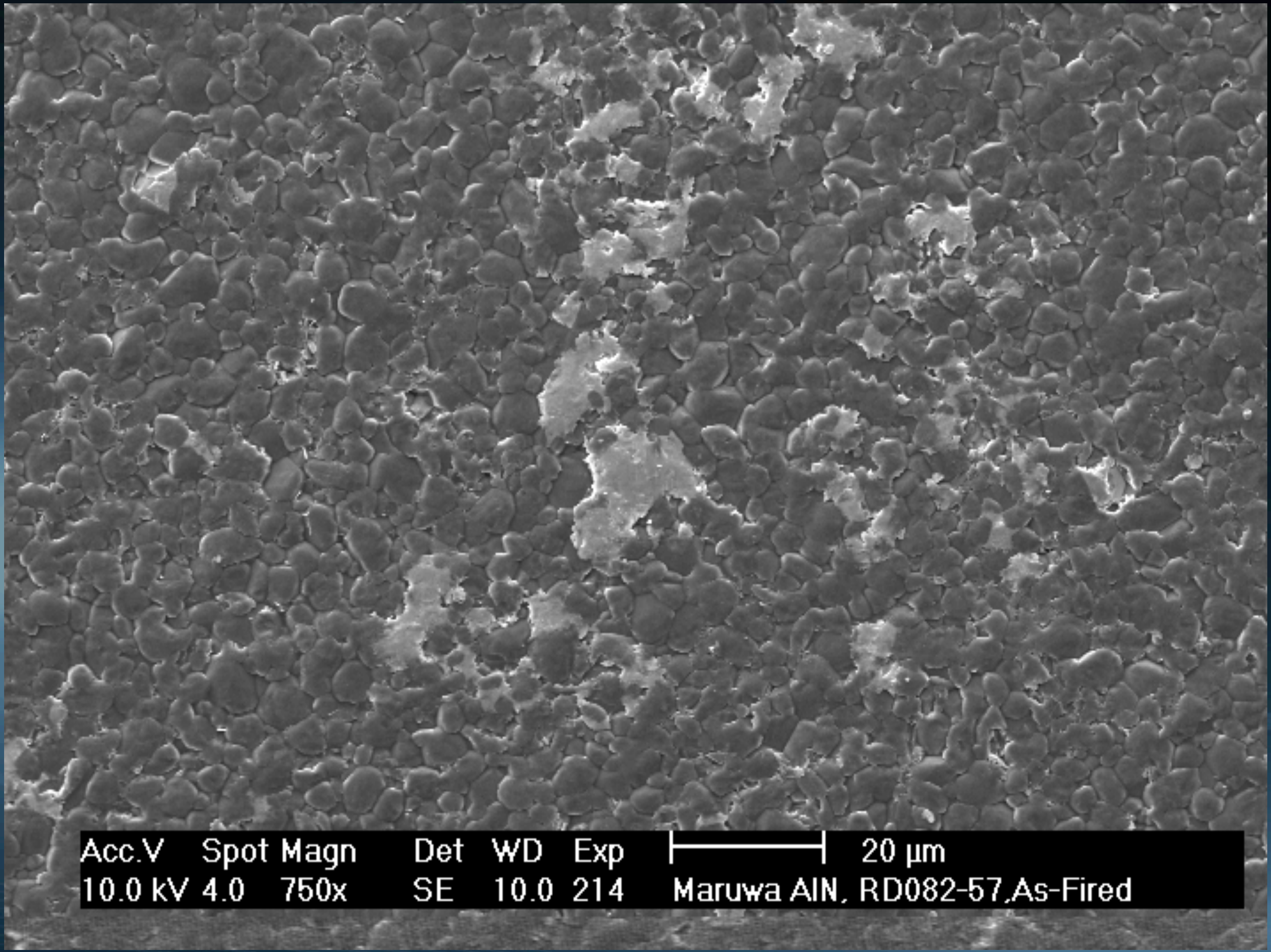
Al_2O_3 Surface Preparation

- As-fired or Polished
  - Alkaline clean
  - Acid dip
  - Thorough DI H_2O rinse
- Lapped
  - Adjust lapping to minimize damage layer extent
  - Anneal if feasible
  - Alkaline clean, acid dip, DI H_2O rinse

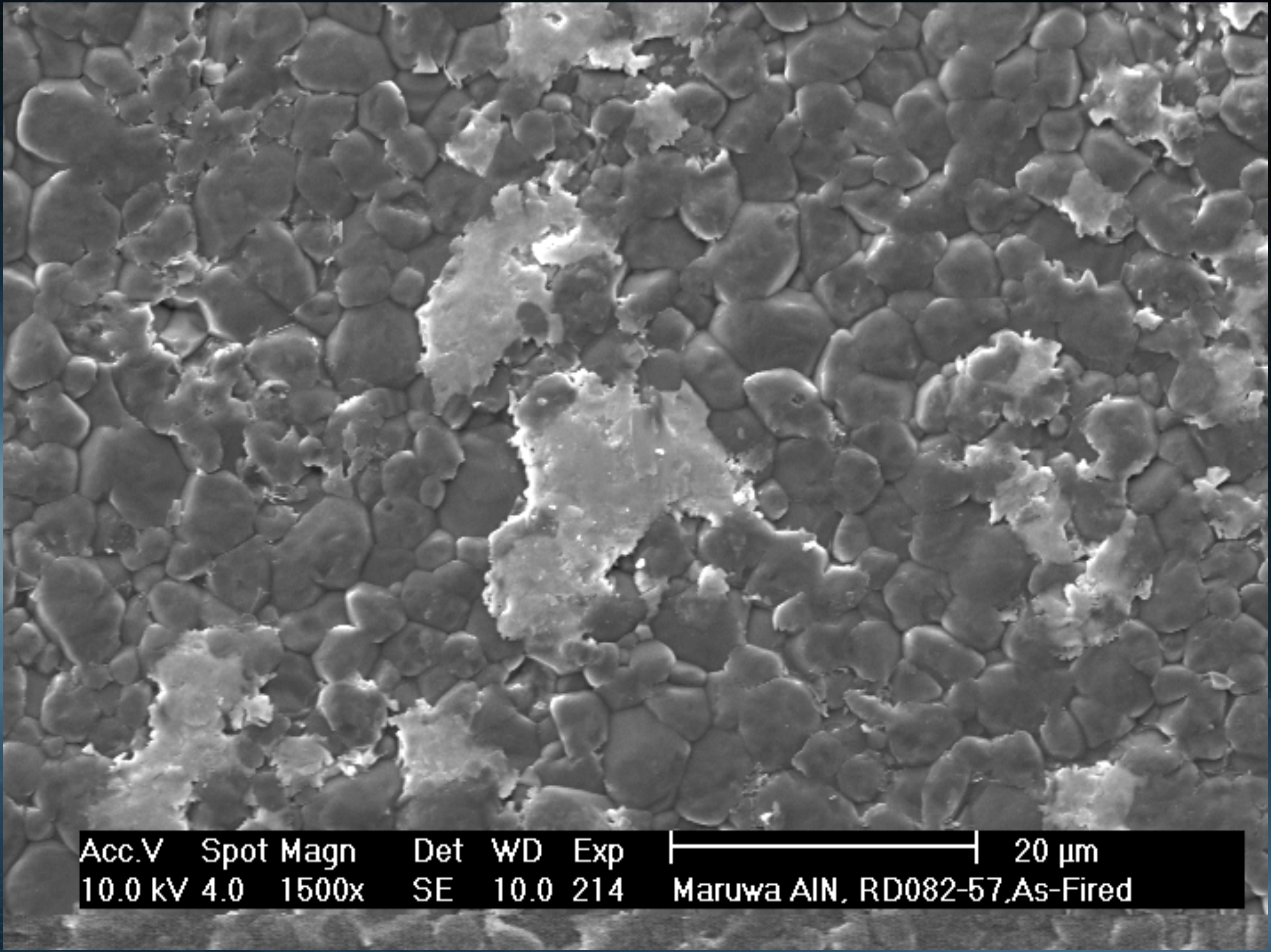
AlN Surface Characterization

- As-fired surfaces
  - No micro-cracks present
  - Surface is contoured but smooth
  - Need to clean off any contaminants
  - Expect good thin film adhesion
- Lapped surfaces
  - Micro-cracks and inter-granular fracture
  - Contamination from lapping media

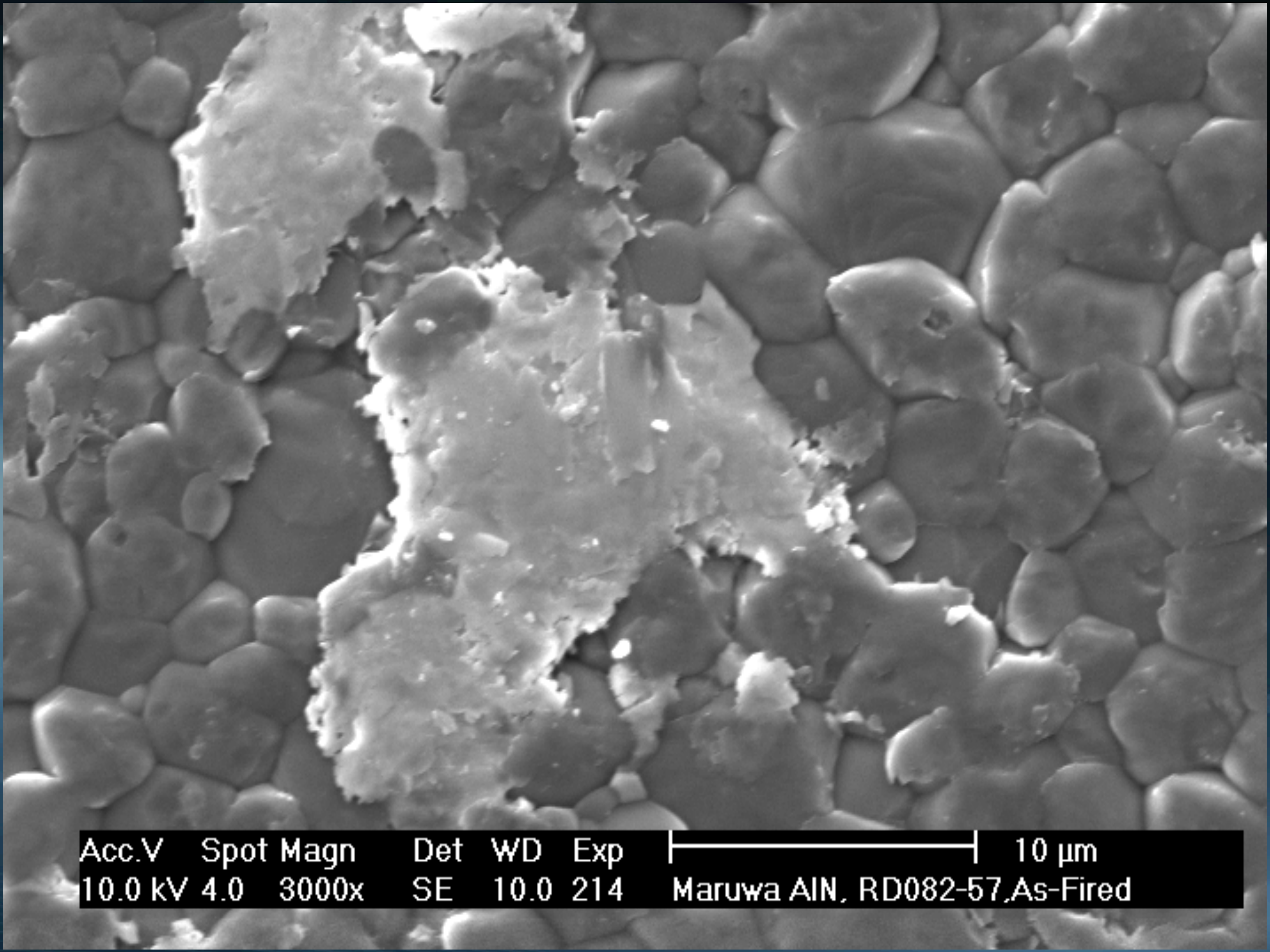
AlN Surface Finish- As Fired



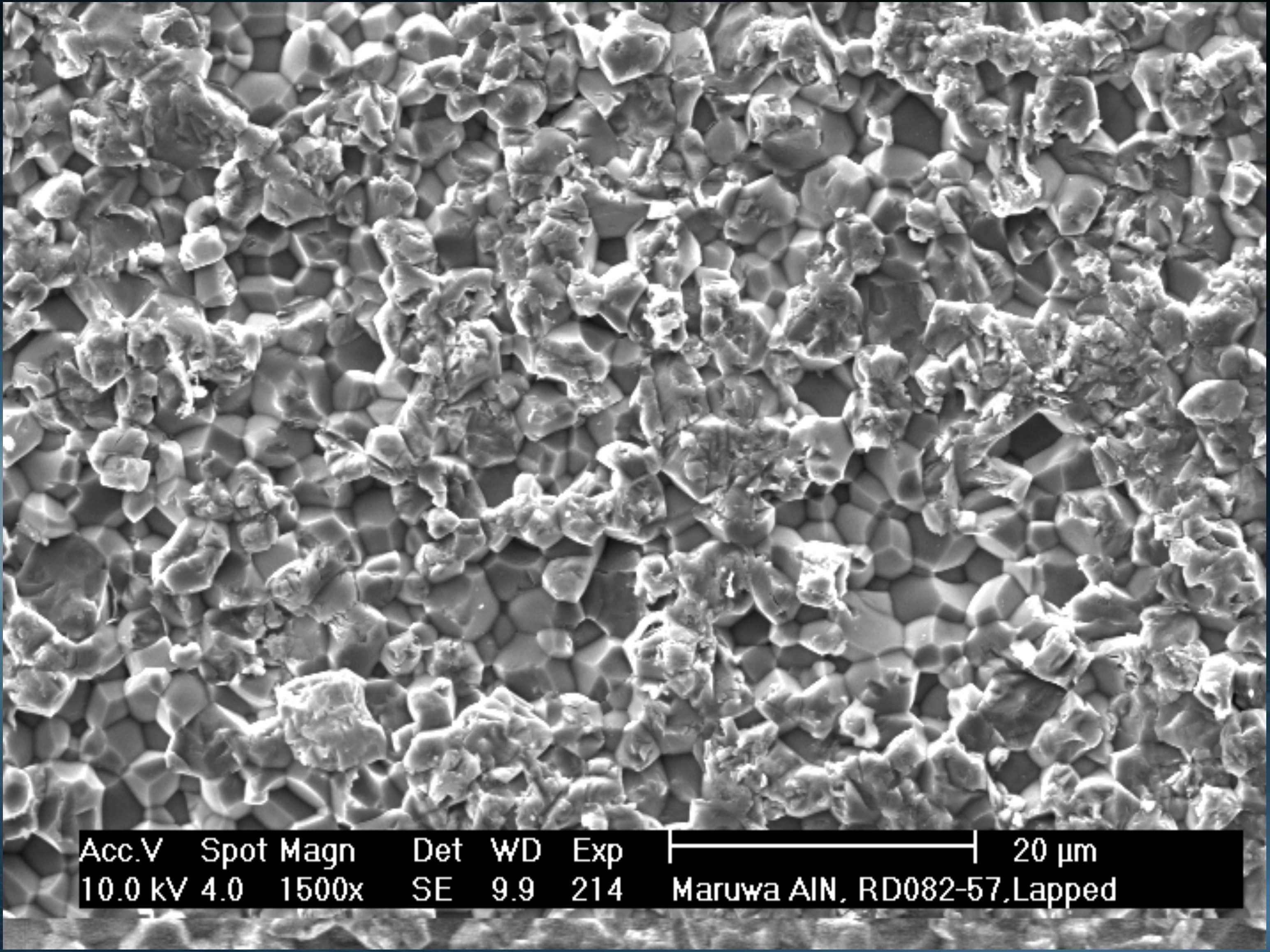
AlN Surface Finish- As Fired



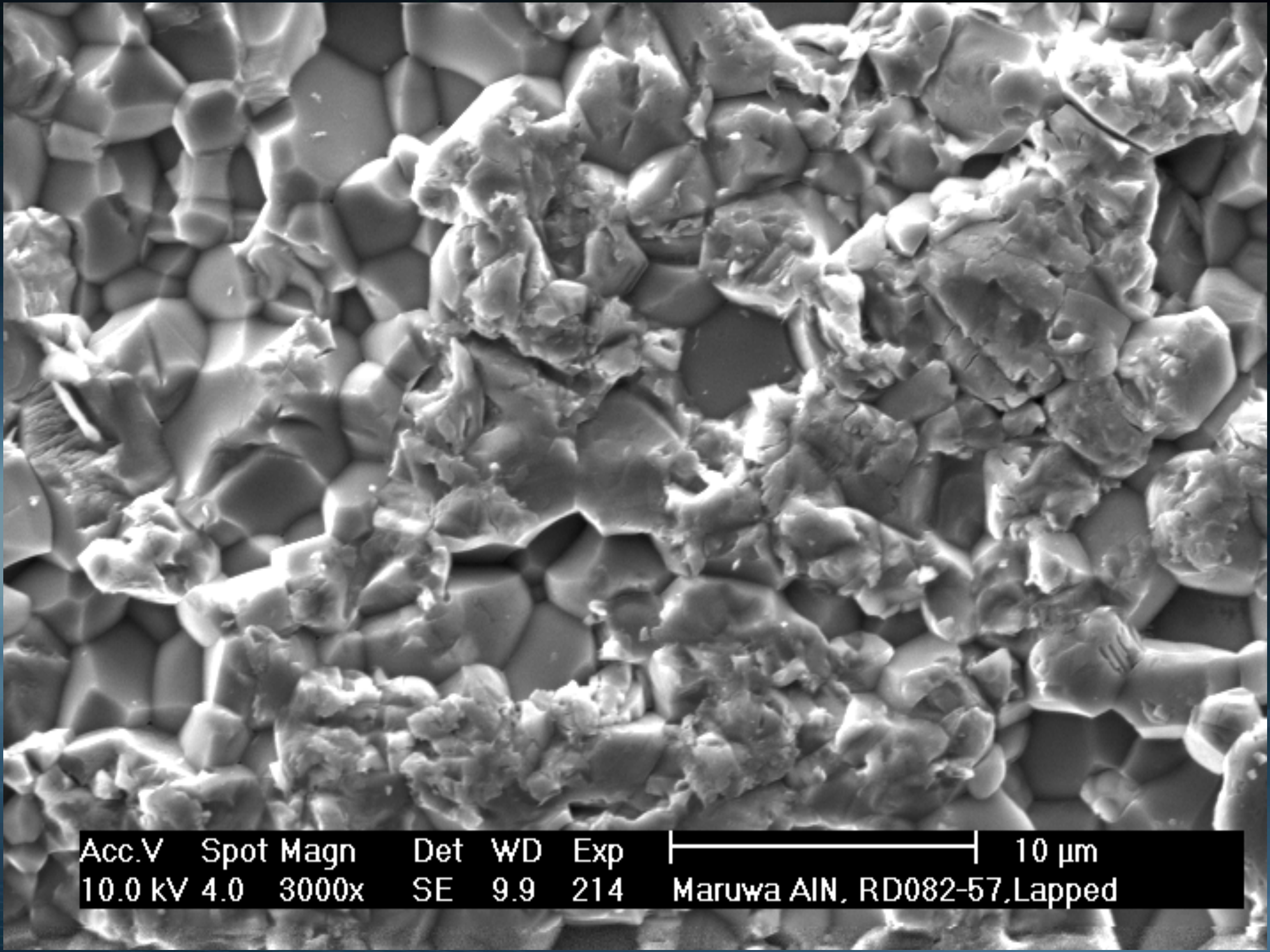
AlN Surface Finish- As Fired



AlN Surface Finish- lapped



AlN Surface Finish- lapped



Adhesion Layer : Al_2O_3 and ALN Thin Film Metallization

CMC INTERCONNECT TECHNOLOGIES

What is Required for Two Materials to Stick Together?

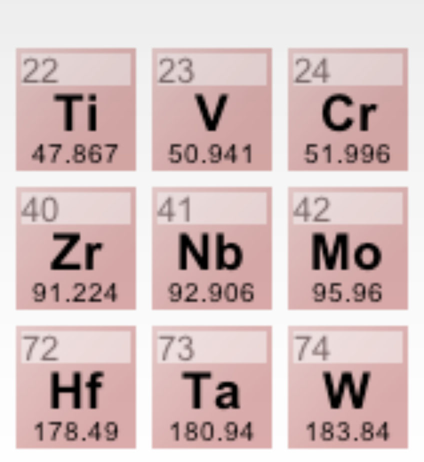
- The two materials must form a bond. This means the total energy of the bonded material is lower than the sum of the energies of the individual non-bonded materials

$$E_{AB} < E_A + E_B$$

- For chemical bonds, this energy difference is the change in Gibbs Free Energy (ΔG). Negative ΔG means bonding is favorable.
- There are two components in ΔG, Enthalpy ΔH which is chemical energy, and Entropy ΔS.
- If ΔG is negative, the next requirement is that the materials are brought into intimate contact with each other so that a bond can form. This involves reacting and mixing on an atomic level. It also requires diffusion which is enhanced by temperature
- Even for negative ΔG, materials require an activation energy for a bond to begin to form. This energy usually comes from applied heat.

Adhesion to Al_2O_3 - ΔG

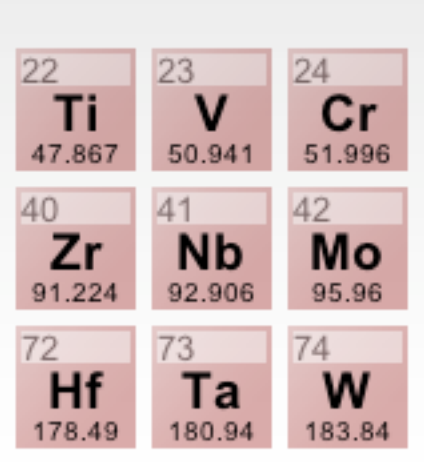
Metal	Oxide	ΔG (Kcal/mole) 200C
Ti	TiO_2	-210
Ti	Ti_2O_3	-225
Cr	Cr_2O_3	-160
V	V_2O_3	-180
Ta	Ta_2O_5	-175
Nb	NbO	-175
Zr	ZrO_2	-240
Hf	HfO_2	-240
W	WO_3	-115



22 Ti 47.867	23 V 50.941	24 Cr 51.996
40 Zr 91.224	41 Nb 92.906	42 Mo 95.96
72 Hf 178.49	73 Ta 180.94	74 W 183.84

Adhesion to AlN - ΔG

Metal	**Nitride**	**ΔG (Kcal/mole) 200C**
Ti	TiN	-140
Zr	ZrN	-140
Nb	Nb_2N	-90
Cr	Cr_2N, CrN	-35
V	VN	-65
Ta	TaN	-100



22 Ti 47.867	23 V 50.941	24 Cr 51.996
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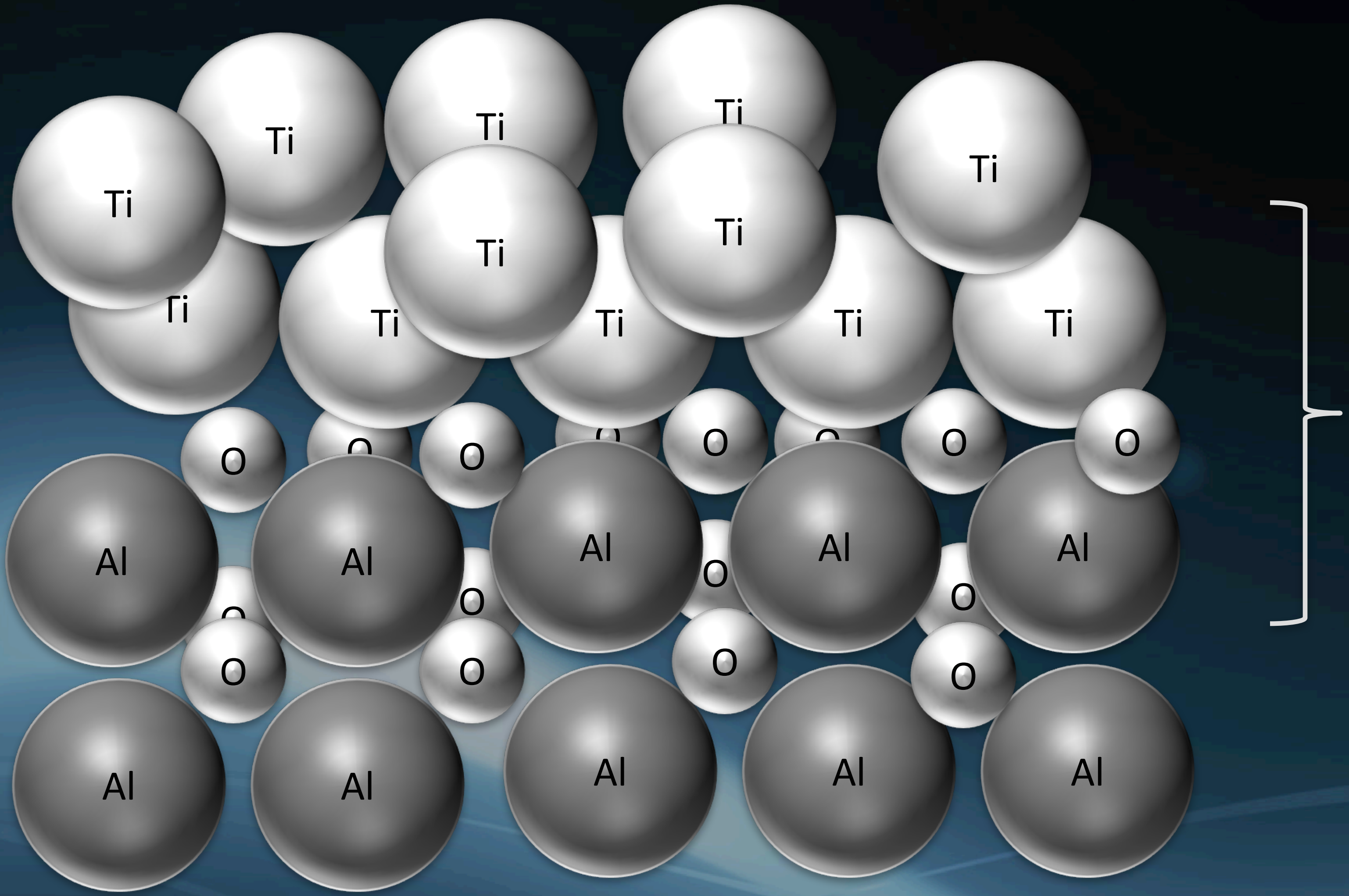
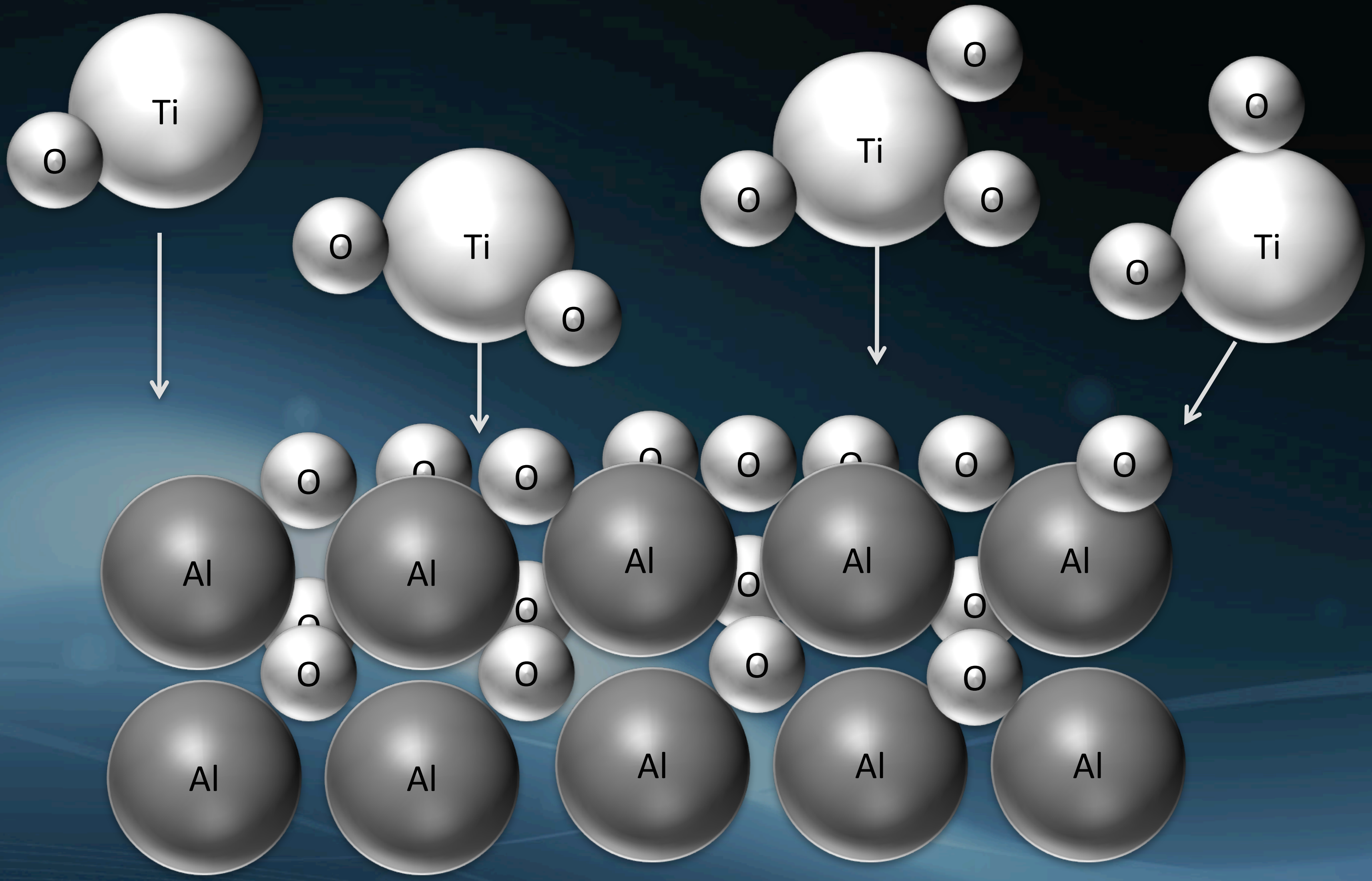
Adhesion Layer Surface Reaction- Ti Reaction with Oxygen and Aluminum



Ti=O=Al

Poor Base Vacuum- Ti Is Oxidized- No Adhesion Reaction



Ti/W and Ti Adhesion Layer Comparison

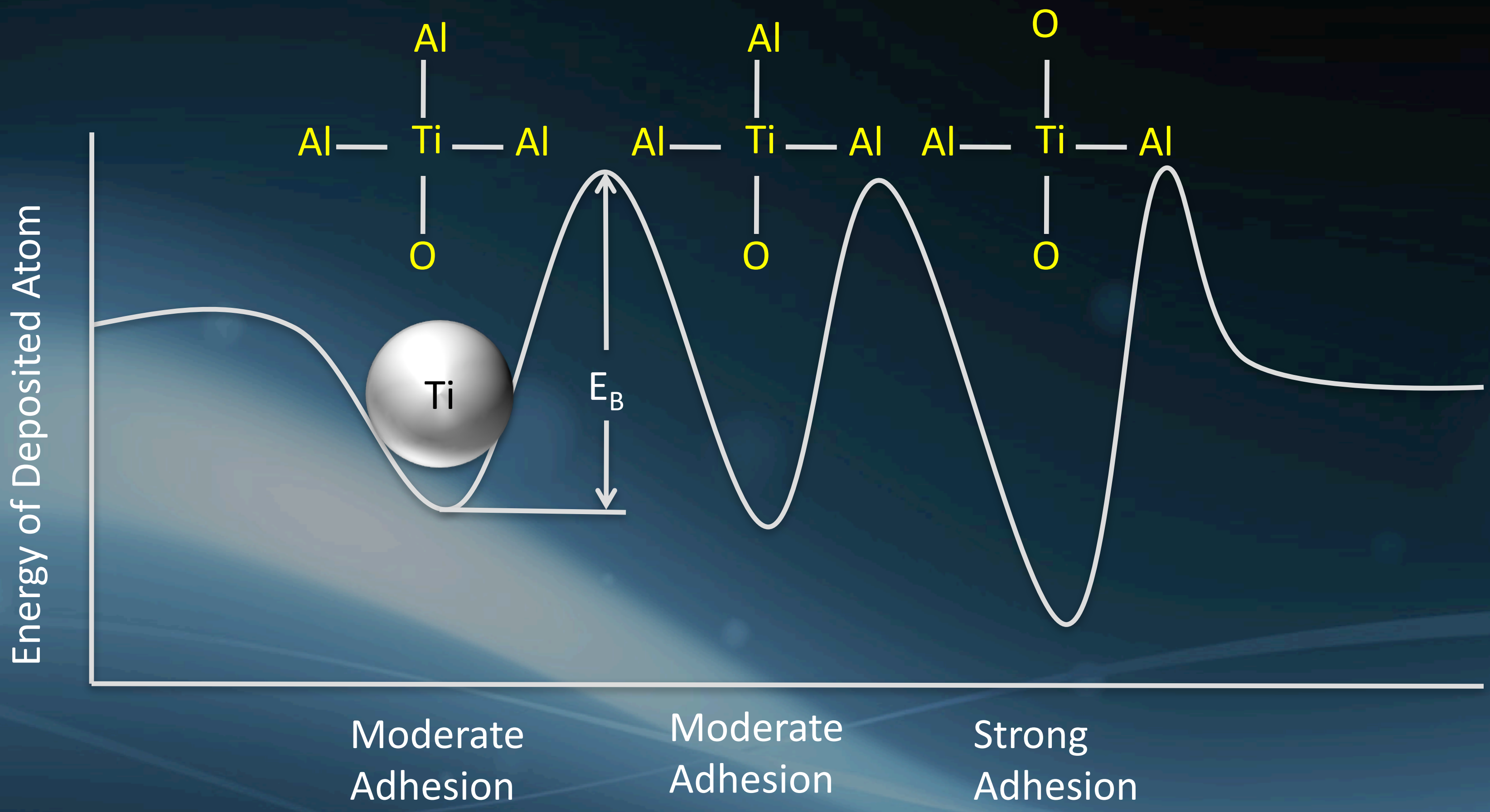
- Ti and TI/W are the most commonly used bonding layer materials
- Ti has stronger chemical bonding (larger -ΔG) with both oxide (Al_2O_3) and nitride (AlN) surfaces
- W has higher atomic mass than Ti (183 gr/mol vs. 47 gr/mol) and thus higher kinetic energy upon impact with the substrate. This provides additional activation energy for bonding.
- Ti films are much more sensitive to base vacuum because of Ti high affinity for oxygen. Adhesion is degraded for vacuums $>10^{-6}$ torr.
- Ti/W films have higher residual stress than Ti and film properties are much more dependent on Argon pressure than Ti films
- Ti/W films can be etch with H_2O_2 and thus do not require HF.

Ti/W and Ti Adhesion Layer Comparison

- Both Ti/W and Ti can be used effectively
- For Ti, base vacuum < 1 x 10^{-6} torr to get good adhesion to Al_2O_3 and to subsequent metal layers
- For Ti/W, Argon pressure must be 7 x 10^{-3} torr for low film stress
- Because Ti/W has both strong chemical bonding and higher deposition energy, the process window is larger

Impact of Substrate Temperature on Ti Adhesion to Al_2O_3

$KT_{substrate} > E_B$ for Ti to diffuse to strong adhesion site



Impact of Substrate Temperature on Ti Adhesion to Al_2O_3

- If $T_{substrate}$ is too low, fraction of Ti at poor bonding sites (few surrounding O or N atoms)
- Adhesion of Ti layer is compromised
- CMC experience, $T_{substrate} > 150C$ (CMC used 200C)
- This is the same for other bonding layers such as Ni to Ti, or Cu to Ti.

Thin Film Barrier Layers on Al_2O_2 and AlN

Barrier Layers

- Role(s) of the barrier layer:
  - Prevent diffusion of the adhesion layer atoms (Ti, W, Cr) to the solder interface
  - Act as a solder interface
  - Act as seed metal for subsequent plated layers
- Common Barrier layer materials: NiV, Pd, Pt
- Common Barrier Seed layer materials: NiCu, Cu
- Key requirements for sputtered barrier layer
  - Bond well to adhesion layer
  - Form a low stress deposit
  - Form a dense deposit (no pores)
  - Wet solder materials well
  - Not form extensive intermetallic compounds when reacting with solder
  - For seed metal applications: provide low electrical resistance

Bonding of the Barrier Layer to the Adhesion Layer

First requirement for bonding, negative Gibbs Free Energy (–ΔG)

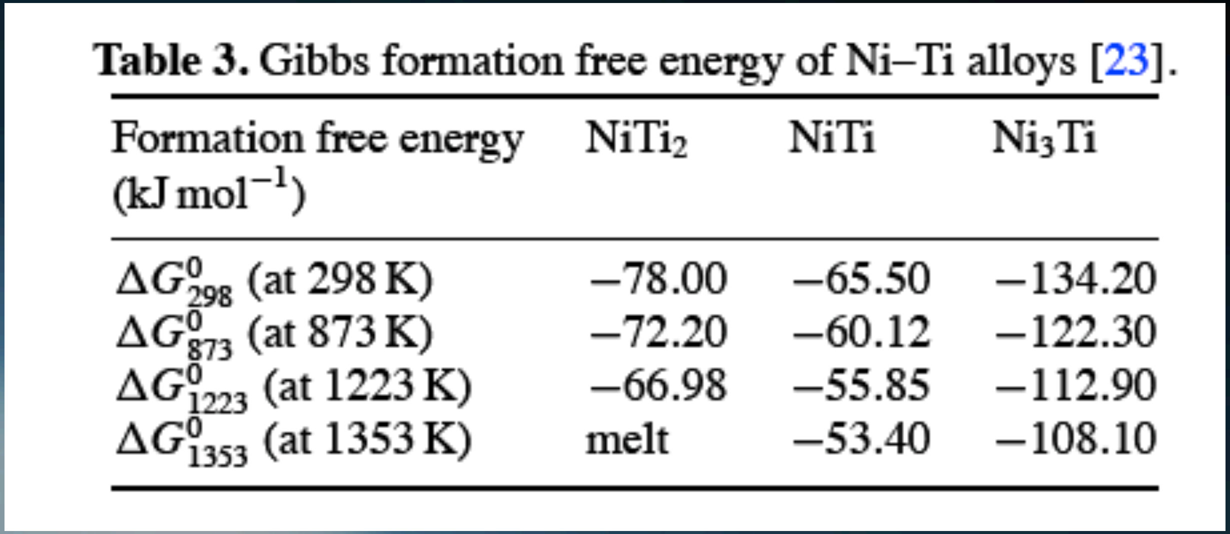


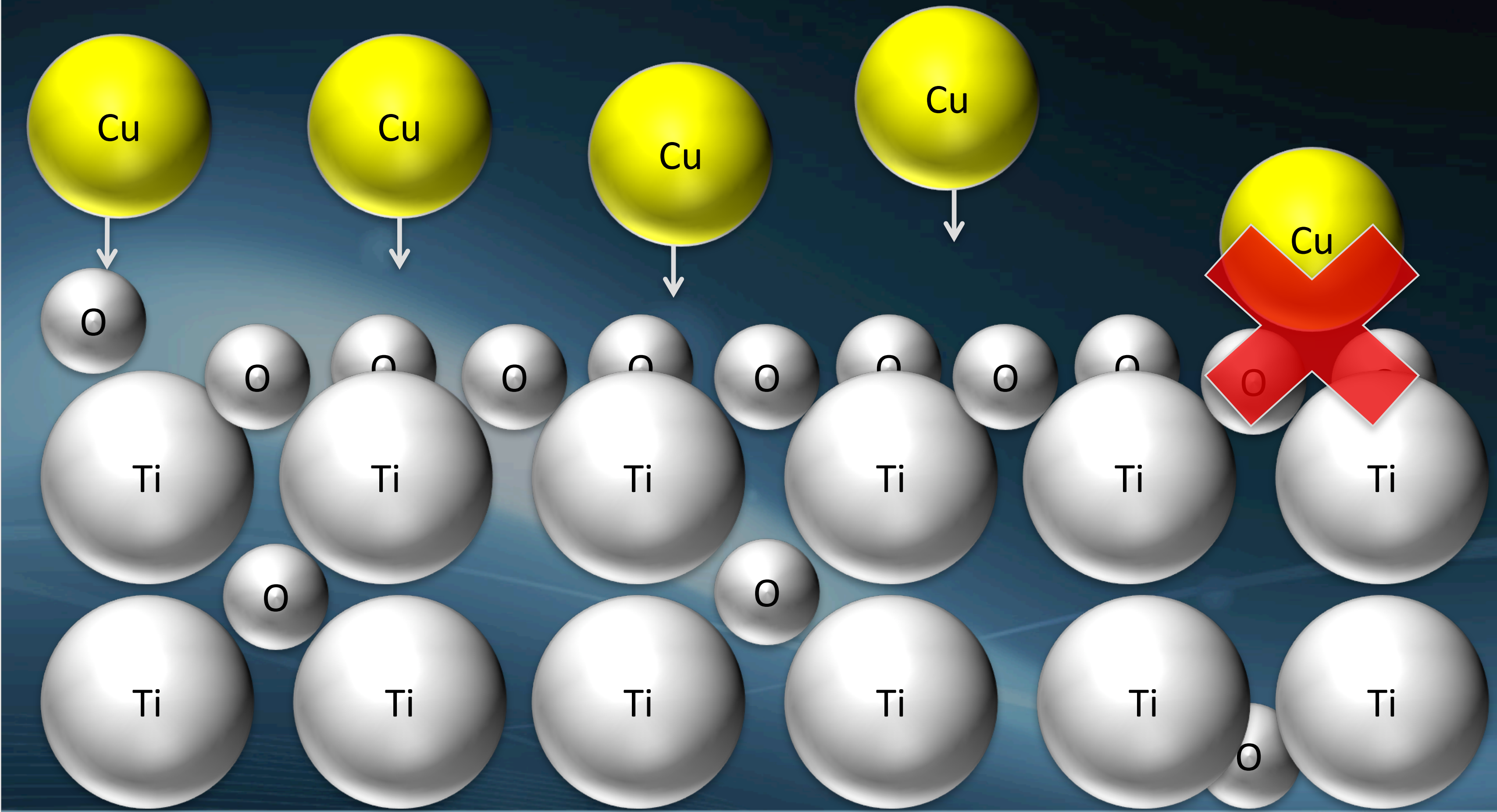
Table 3. Gibbs formation free energy of Ni–Ti alloys [23].

Formation free energy (kJ mol^{-1})	$NiTi_2$	NiTi	Ni_3Ti
ΔG^0_{298} (at 298 K)	−78.00	−65.50	−134.20
ΔG^0_{873} (at 873 K)	−72.20	−60.12	−122.30
ΔG^0_{1223} (at 1223 K)	−66.98	−55.85	−112.90
ΔG^0_{1353} (at 1353 K)	melt	−53.40	−108.10

Ti/Ni, Ti/Cu, Ti/Pd, Ti/Pt all have (–ΔG)

Bonding of the Barrier Layer to the Adhesion Layer

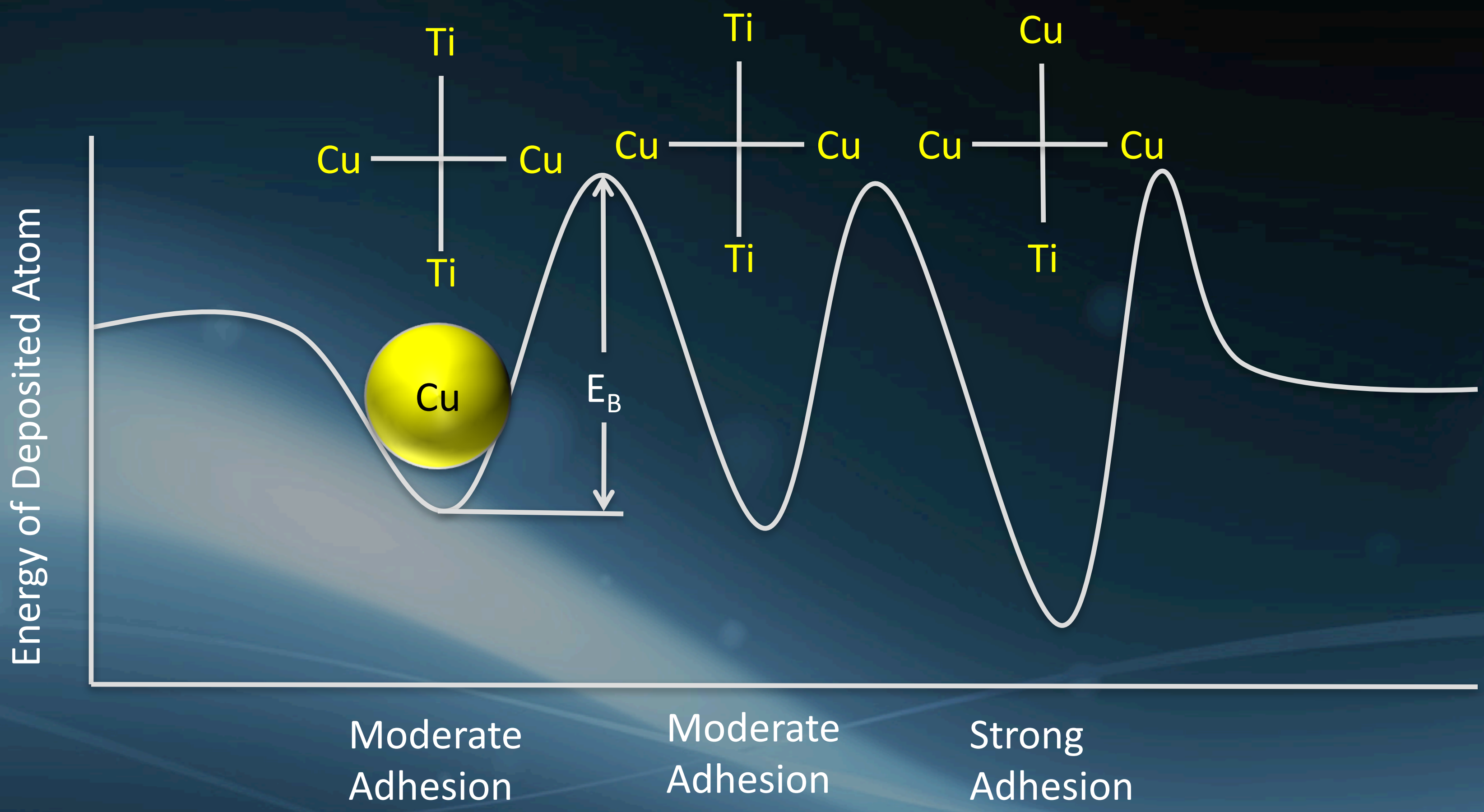
Second requirement for bonding: bonding materials in intimate contact (no contamination). For sputtered layers, this requires a low base vacuum (10^{-7} range for Ti). Case of a poor base vacuum is illustrated below.



Ti is passivated and non-reactive.

Bonding of the Barrier Layer to the Adhesion Layer

Third requirement for bonding materials can inter-diffuse to find optimum bonding sites



$KT_{substrate} > E_B$ Substrate Temperature must be > 150C during deposition for strongest bonds to form

Typical Sputtering Parameters for Al_2O_3 and AlN

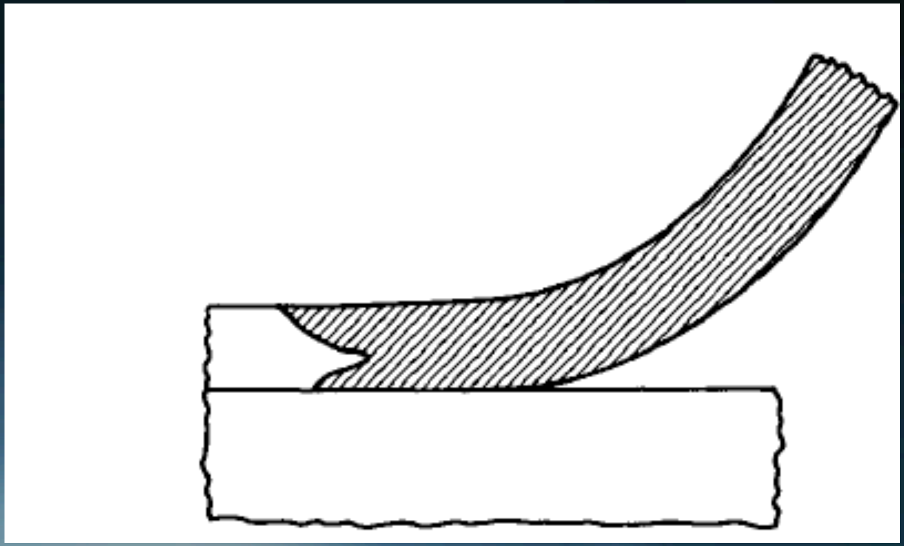
Typical Conditions for Sputtering on Al_2O_3 and AlN

- Base vacuum
  - In the 10^{-7} torr range for Ti adhesion layer
  - Below 2 x 10^{-6} torr for Ti/W
- Argon pressure 3-7 x 10^{-3} torr
  - Less critical for Ti layers
  - For Ti/W, 5-7 x 10^{-3} to reduce stress
- Substrate temperature 150-200C
  - Critical parameter
  - Needs to be optimized for specific materials and sputter system
  - CMC used 200C for Ti/W on AlN with excellent results
  - Needs to be constant during run
- Power 2 KW

Thin Film Adhesion Testing

- There are no universal accepted tests for measuring thin film adhesion
- Most used test is Peel Testing (ASTM D 3167)
- Most adhesion tests infer the adhesive strength by subjecting the film to an external load and measuring the critical value where failure is observed
- Useful for routine quality control
- Most test methods measure adhesion by delaminating a film from the substrate
  - Energy to break the thin film and substrate bonds
  - Energy to plastically deform the thin film layer and substrate layer as the film is pulled away
  - Friction of the thin film surface and substrate surface

Schematic Peel Test



Regions deforming plastically during the peel test are shaded.

Energy to deform thin film layers M_1 is included in the energy required to peel off the thin film . If the thin film is relatively thick, or made of a stiff material, the plastic deformation energy can be comparable to the adhesion term. What is actually measured in the interfacial fracture toughness. Could be a significant factor for DPC adhesion because of thick, ductile Cu layer.

Fundamental Tests for Measuring Adhesion

Measure the strain energy release rate U(J/m^2) or work required to propagate a crack along the interface

Indentation initiates an interfacial crack. Width of resulting delamination blister is measured. Mechanical properties of film materials are used to derive U.

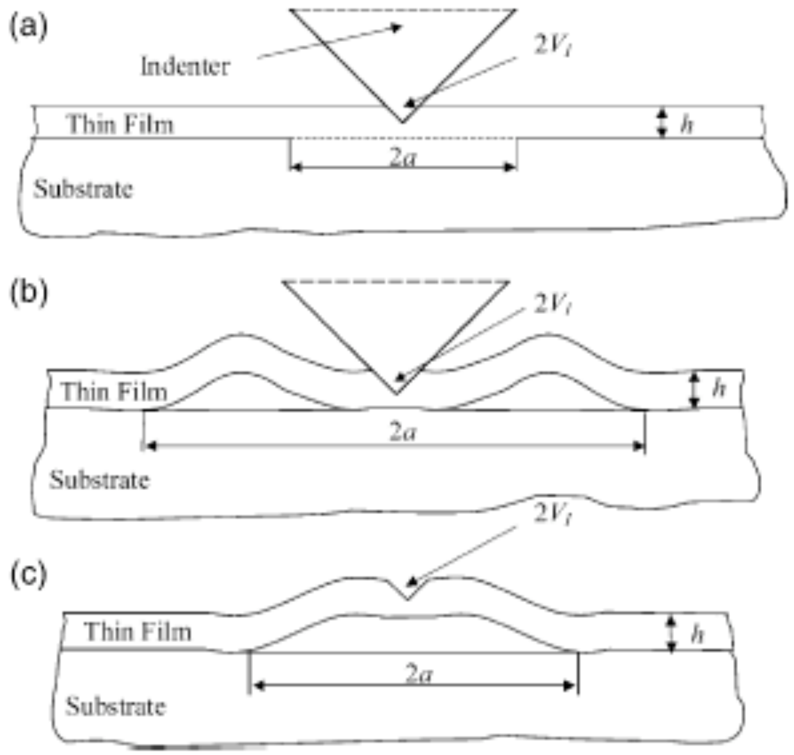
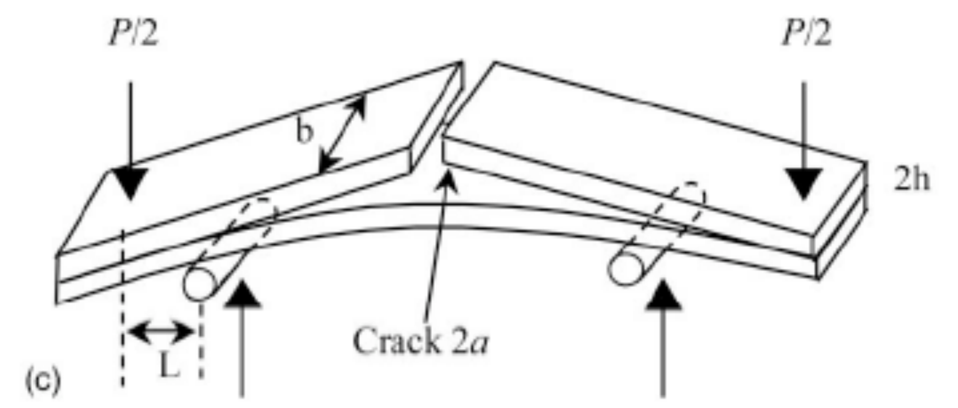
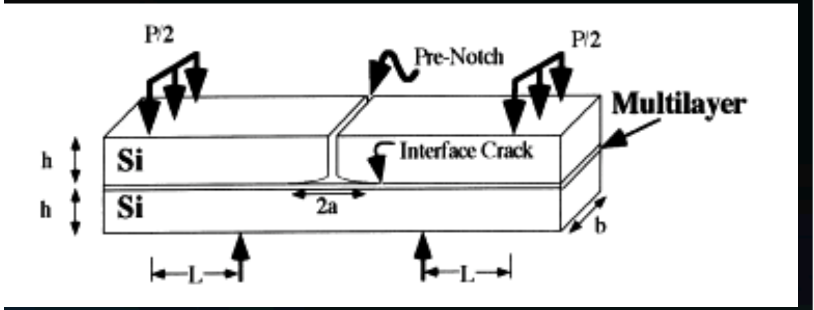


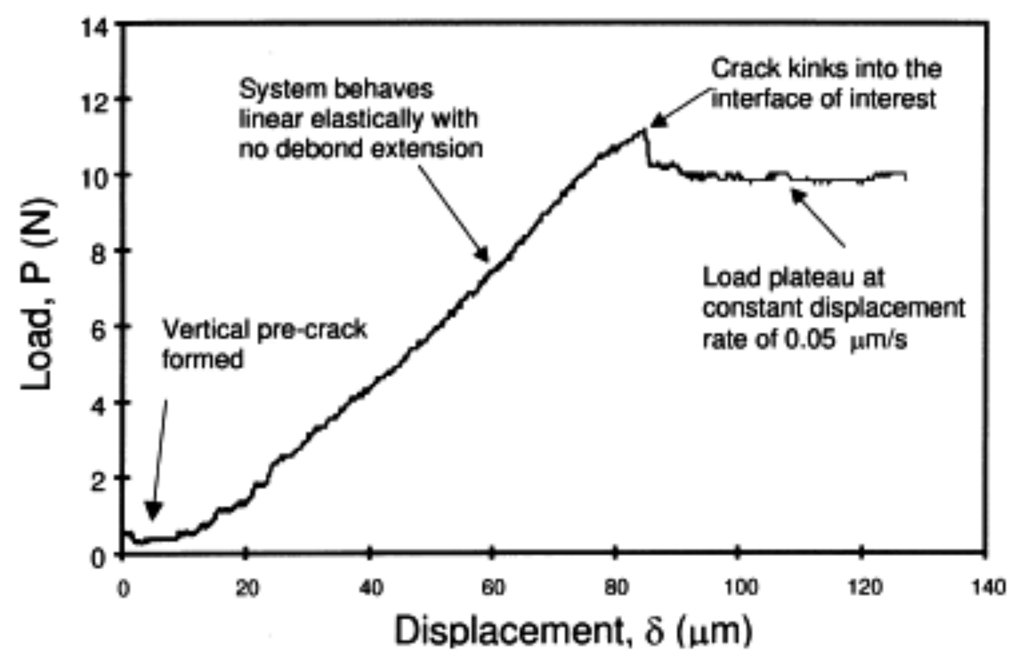
Fig. 6. (a) No buckling during indentation; (b) double-buckling during indentation; (c) single-buckling after the indenter tip removal.

Fundamental Tests for Measuring Adhesion





The substrate layer is cut and a bend test is used to drive a crack along the interface.

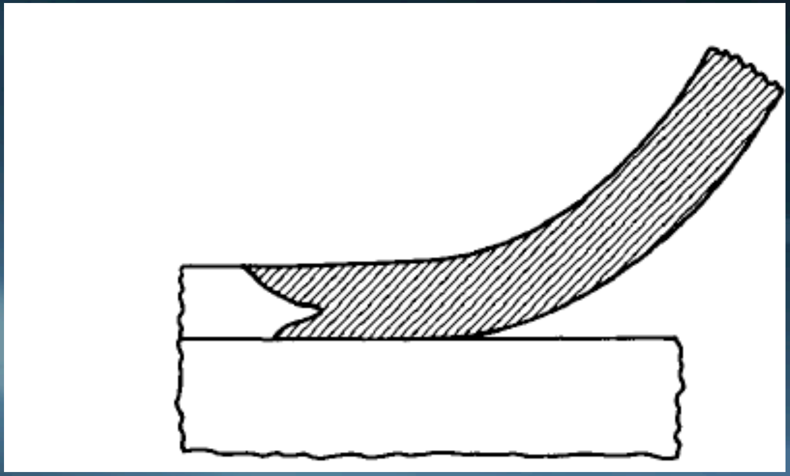


Fundamental Tests

- Developed at Intel, Stanford U and Motorola for evaluating semiconductor metallization schemes
- Requires mathematical models to interpret data
- Value:
  - Allows optimization of adhesion separated from other effects such as film deformation
  - Can provide fundamental information that can be used to predict reliability
  - Allows meaningful comparison of different materials

Practical Adhesion Testing

- Measure adhesion strength + energy to deform layer during testing
- Good relative measure of adhesion if film characteristics are constant
- For example, useful for DPC if Cu thickness is uniform.

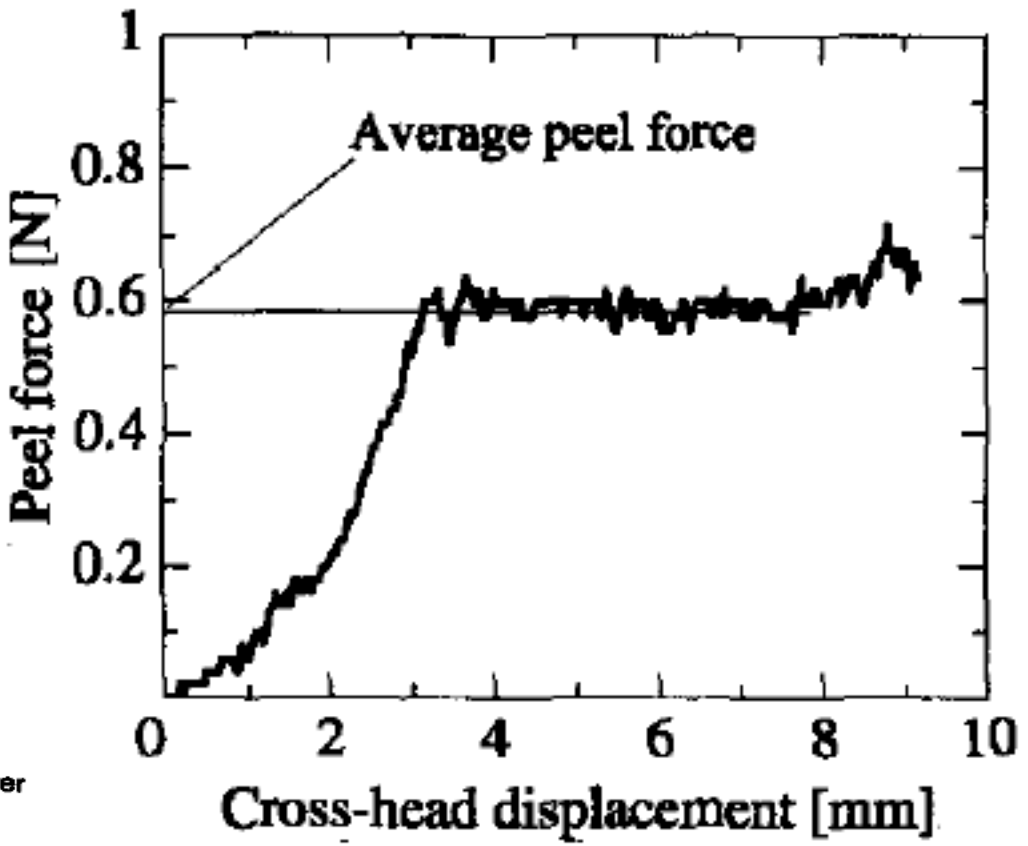


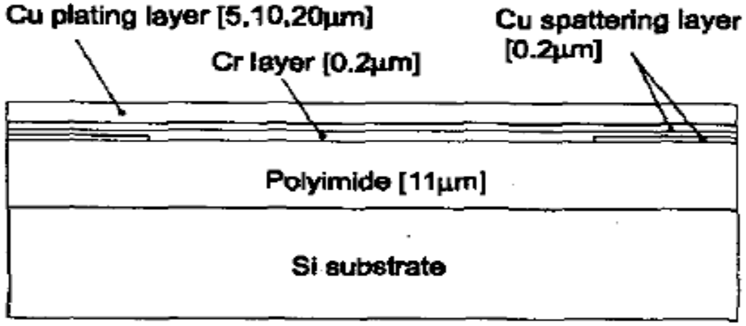
Peel Test Apparatus

- Mechanical tester (Instron)
- Load cell
- Peel fixture designed for constant 90° peel angle
- Peel force vs Displacement

Peel Testing- Typical Metal to Metal Peel

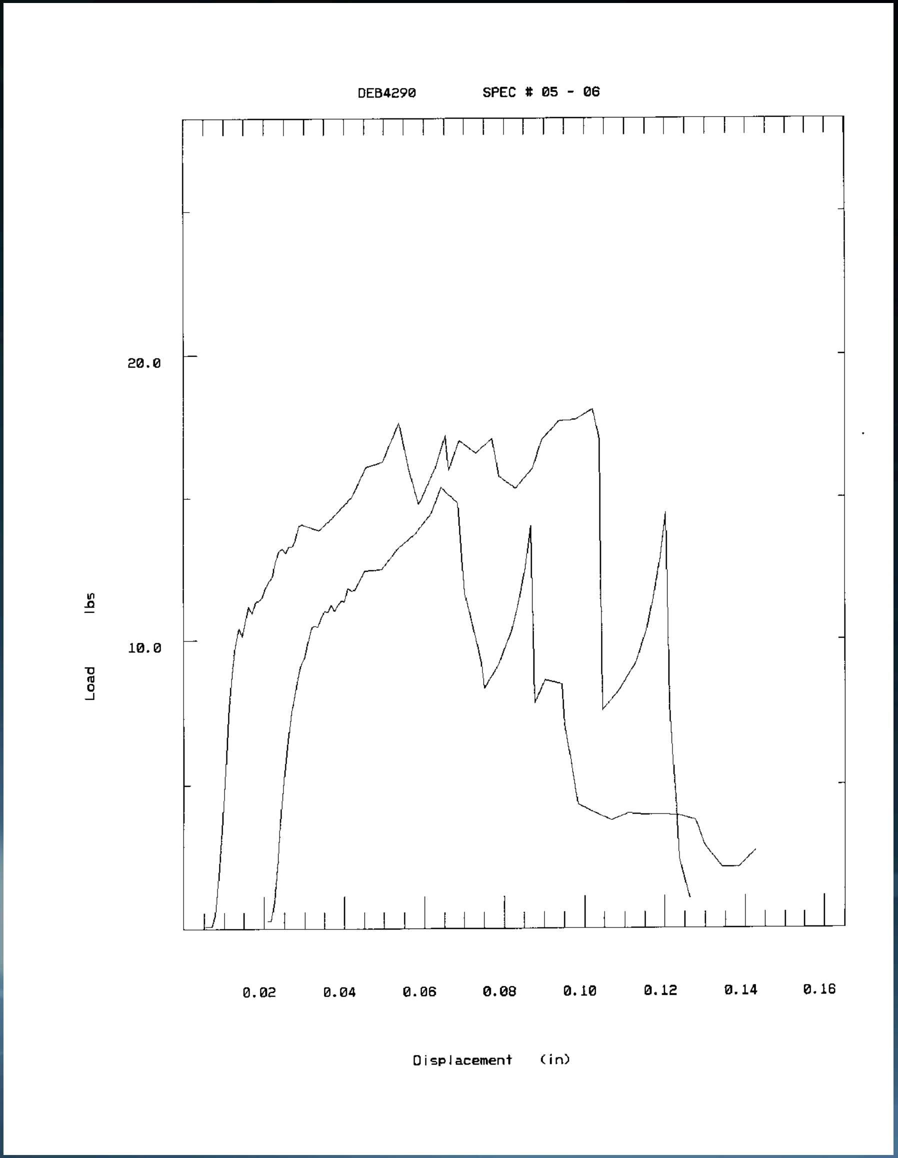
Failure at Cu/Cr Interface





Example from CMC: Ceramic Failure

- CuNi/TiW on AlN
- Brazed Lead Peel
- Ceramic failure
- Ceramic failure is dictated by flaw population



Practical Adhesion Tests- Useful for What?

- Process Optimization
- Specific product specification such as a lead pull value
- Package Qualification
  - Characterize Qualification samples for each customer
  - Adhesion values can be tied to customer established reliability data
  - Can evaluate adhesion at any time relative to qualified samples
  - Can use adhesion data from qualification to establish process changes and also to introduce equipment changes
- Periodic Quality Control

Troubleshooting Guide for Sputtering on Al_2O_3 or AlN

Issue	Location of Issue	Potential Factors to Address
Poor adhesion	Ceramic to metal	Surface contamination on ceramic
		Micro-crack damage layer present on ceramic (intra ceramic and ceramic to metal failure)
		Thin film layer with high stress
		Thin film deposition substrate temperature too low
		Oxygen contamination of adhesion layer during deposition because base vacuum is too high
		Chemical attack from post thin film processing. Pores in thin film.
		High Temperature induced CTE stress. Thin film deposition temperature too high.

Troubleshooting Guide for Sputtering on Al_2O_3 or AlN

Issue	Location of Issue	Potential Factors to Address
Poor adhesion	Adhesion layer to barrier layer	Oxygen contamination of adhesion layer during deposition because base vacuum is too high
		Barrier layer with high stress.
		Thin film deposition substrate temperature too low
		Chemical attack from post thin film processing. Pores in barrier thin film layer.
		High Temperature induced CTE stress. Thin film deposition temperature too high.

Troubleshooting Guide for Sputtering on Al_2O_3 or AlN

Issue	Location of Issue	Potential Factors to Address
Blisters form after exposure to heat > 300C	Ceramic and adhesion layer delamination	Pores in the ceramic surface result in a pore in the thin film layer. After exposure to post-thin film chemicals, these pores trap liquids or gas. Upon heat expose, the liquids or gas expand forming a blister.
		All of the factors in slide 61 that lead to poor overall adhesion may also lead to more localized blistering.
	Adhesion Layer and Barrier Layer Delamination	All of the factors in slide 62 that lead to poor overall adhesion may also lead to more localized blistering