

A photograph of a snowy street scene. The road is covered in a layer of snow with some tire tracks. On the right side of the road, there are several tall evergreen trees, some of which are covered in snow. A red circular traffic sign with a white horizontal bar is visible on the right. In the background, there is a long, low building with a yellowish-brown facade. The sky is overcast and grey.

Introduction to Thin Film Technology

End

Introduction and Overview

What is a "thin film" ?

- thin = less than about one micron (10,000 Angstroms, 1000 nm) film = layer of material on a substrate
 - (if no substrate, it is a "foil")

Applications:

- microelectronics - electrical conductors, electrical barriers, diffusion barriers . . .
- magnetic sensors - sense I , B or changes in them
- gas sensors, SAW devices
- tailored materials - layer very thin films to develop materials with new properties
- optics - anti-reflection coatings
- corrosion protection
- wear resistance
- etc.

Special Properties of Thin Films

different from bulk materials

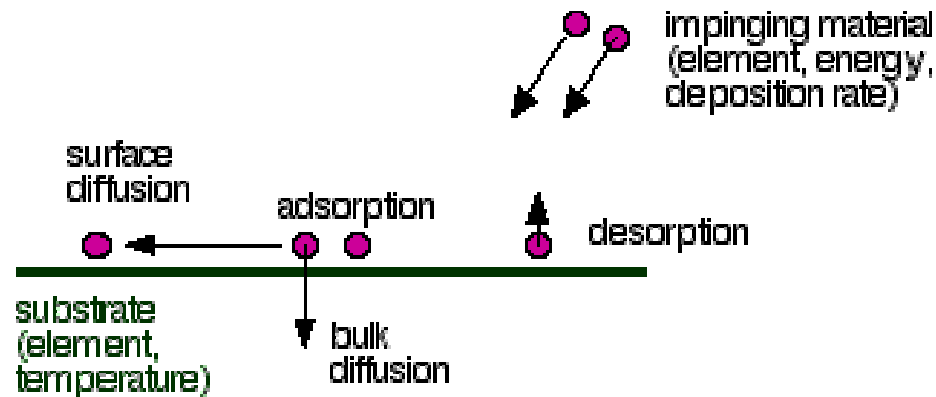
Thin films may be:

- two dimensional (very thin films)
- strongly influenced by surface and interface effects
- This will change electrical, magnetic, optical, thermal, and mechanical property

Typical steps in making thin films

- emission of particles from source (heat, high voltage . . .)
- transport of particles to substrate (free vs. directed)
- condensation of particles on substrate (how do they condense ?)

Simple model:

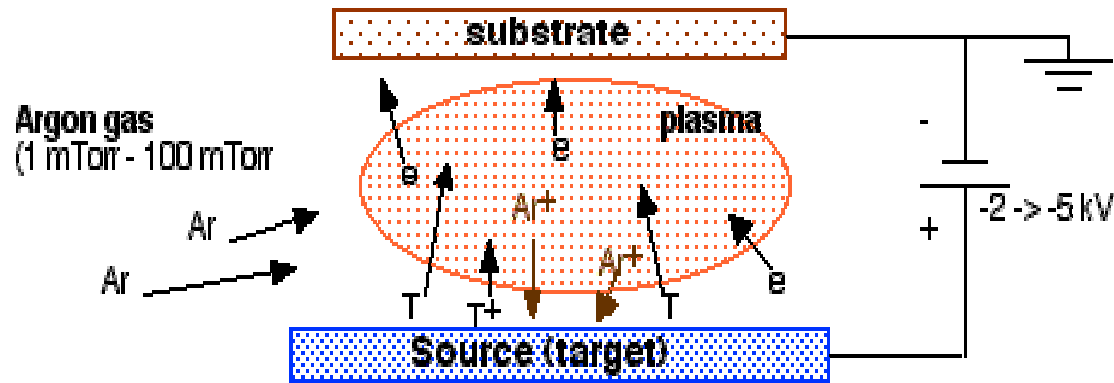


How do the variables effect film structure and properties ?

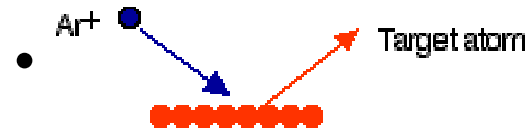
What physics is in all this ?

- thermodynamics and kinetics
 - phase transition - gas condenses to solid
 - nucleation
 - growth kinetics
 - activated processes
 - desorption
 - diffusion
 - allowed processes and allowed phases
- solid state physics
 - crystallography
 - defects
 - bonding
- electricity and magnetism
 - optics
 - conductivity - resistivity
 - magnetic properties
- mechanics
 - stresses in films
 - friction and wear

Sputter Deposition



Sputtering process



momentum transfer process

involves top 10 Å

model as hard sphere collisions

good for energies < 50 keV

95 % of incident energy goes into target

=> COOL the target

5 % of incident energy is carried off by target atoms

typical energies of 5-100 eV

target atoms come off with a non-uniform distribution

more atoms normal to the surface

cosine distribution (like surface source)

Atoms into gas state

at target:

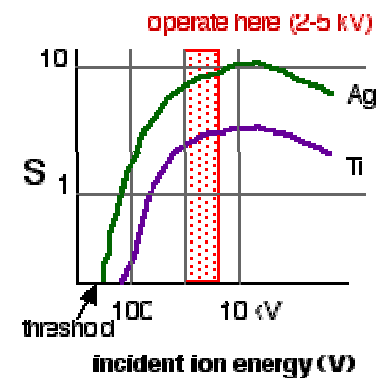
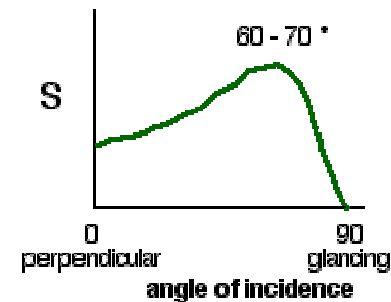
- target atoms ejected
- target ions ejected (1 - 2 %)
- electrons emitted
 - helps keep plasma going
- Ar⁺ ions reflected as Ar neutrals
- Ar buried in target
- photons emitted

We are most interested in the first of these: target atoms going into the gas phase

characterize process by **sputter yield (S)**

for normal incidence sputtering:

- S = number ejected / number incident
- S depends on
- target material
- binding energy
- mass of atoms
- sputtering gas
- mass of atoms (S increases for heavier gasses)
- incident energy (S increases for higher energies)
- geometry
- most efficient 20-30 degrees from glancing





TECHNOLOGY FOCUS

Thin film technology is pervasive in many applications, including microelectronics, optics, magnetic, hard resistant coatings, micro-mechanics, etc.

- ❑ Progress in each of these areas depends upon the ability to selectively and controllably deposit thin films - thickness ranging from tens of ångströms to micrometers - with specified physical properties.
- ❑ its requires control - often at the atomic level - of film microstructure and microchemistry. There are a vast number of deposition methods available and in use today.
- ❑ all methods have their specific limitations and involve compromises with respect to process specifics,
- ❑ substrate material limitations, expected film properties, and cost. This makes it difficult to select the best technique for any specific application.

Coating Processes

تکنولوژی لایه نازک



Electro deposition

Chemical coating

Conversion coating

Vapour deposition

Chemical vapour deposition

Physical vapour deposition

Spraying

Welding

Molecular beam epitaxy

Electrochemical deposition



- **Advantages**

- Low temperature treatment
- High hardness
- Low friction
- Applicable to a wide range of metal substrates
- Thick layers possible

- **Disadvantages**

- Poor thickness uniformity on complex components
- Hydrogen embrittlement
- Not applicable to insulating substrates
- Possible environmental concerns with plating baths

Chemical Coatings



- **Advantages**

- Low temperature treatment
- More corrosion resistant than electrodeposited chromium
- Can coat complex shapes uniformly
- Hard particles can be incorporated to increase hardness
- PTFE can be incorporated to reduce friction
- Can coat most metals and some insulators

- **Disadvantages**

- More expensive than electroplated chromium
- Heat treatment is needed to develop optimum properties

Conversion Coatings



- Thin compound layers can be produced by reacting a metal surface with an acidic solution. e.g. Thin ($10\mu\text{m}$) coatings of metal phosphates are formed on steel substrates exposed to phosphoric acid. These provide low friction surfaces with some resistance to adhesive wear. Often used to help components run-in.
- **Advantages**
 - Cheap and simple to perform
 - Low temperature treatment
- **Disadvantages**
 - Restricted range of materials can be treated
 - Thin treated layer
 - Poor treatment durability
 - Difficult to control treatment quality on heterogeneous materials

Chemical Vapour Deposition (CVD)



- Gaseous compounds react to form a dense layer on a heated substrate. The most widely deposited wear-resistant coatings are TiC, TiN, chromium carbide and alumina. Deposition temperatures are generally in the range 800-1000°C which restricts the range of materials which can be coated and can lead to component distortion. Thicknesses are limited to about 10µm due to the thermal expansion mismatch stresses which develop on cooling which also restrict the coating of sharp edged components.
- **Advantages**
 - High coating hardness
 - Good adhesion (if the coating is not too thick)
 - Good throwing power (i.e. uniformity of coating)
- **Disadvantages**
 - High temperature process (distortion)
 - Sharp edge coating is difficult (thermal expansion mismatch stresses)
 - Limited range of materials can be coated
 - Environmental concerns about process gases

Physical Vapour Deposition (PVD)

- **Advantages**

- Excellent process control
- Low deposition temperature
- Dense, adherent coatings
- Elemental, alloy and compound coatings possible

- **Disadvantages**

- Vacuum processes with high capital cost
- Limited component size treatable
- Relatively low coating rates
- Poor throwing power without manipulation of components

- low pressure coating processes in which the coating flux is produced by a physical process. There are two main types:-

- Evaporation

- Sputtering

- In both cases the source material is a solid (metal or ceramic). A reactive gas may be used in the deposition chamber to deposit compound coatings from an elemental source or maintain the stoichiometry of coatings from compound sources. Typical coating thicknesses range from 1-10 μ m for wear-resistant coatings, though thinner layers are used in microelectronics and thicker layers are used for high temperature corrosion protection of gas turbine components.

Evaporation Processes



The vapour pressure of most materials increases with temperature and if it exceeds the ambient pressure the material will rapidly evaporate into the environment. In a coating chamber the pressure is reduced and the source material heated until a desired vapour flux is maintained which is controlled by the source material, the source temperature and the system pressure.

Heating can be performed in several ways:-

- Resistive heating (e.g. aluminium evaporation from TiB_2 boat)
- Electron beam evaporation (e.g. metals such as tungsten)
- Cathodic arc evaporation (e.g. titanium evaporation for TiN coatings)

The vapour pressures of different metals vary over several orders of magnitude so it is difficult to evaporate alloys and control composition.

As-deposited evaporated coatings are porous due to the limited mobility of coating atoms on component surfaces. This can be controlled by heating or ion plating (see later)

Spatter from localised boiling can lead to droplet formation which affects coating performance

Sputtering Processes



- **Main sputtering processes:-**

- DC diode sputtering
(for conducting targets)
- RF sputtering
(for insulating targets)

- When energetic ions strike a surface, material is ejected by the transfer of momentum from the ion to the target atoms (akin to billiard ball collisions at the atomic scale). This can be conveniently achieved in a low pressure glow discharge of an inert gas such as argon.
- In such a process the target material is made the cathode and is raised to a potential of several hundred volts. Electrons leaving the cathode stream out into the gas phase where they can impact with argon atoms, ionising them. The positively charged argon is then accelerated to the cathode where it impacts and sputters away material.
- The sputtering yields of different elements for given impact conditions do not vary very much so target alloy compositions can be maintained in the coating except in cases where there are large differences in the atomic weights of alloy constituents.

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The coating rate scales with the electrical power used to sustain the discharge. The coating rate also depends on the plasma density, so techniques to increase this (e.g. by confining the electrons close to the target using magnets) will increase the coating rate. However, as much as 95% of the power is dissipated as heat in the target so good cooling is essential.

Ion Implantation



- **Advantages**

- Low temperature process
- Very versatile - every stable element in the periodic table can be implanted into any vacuum compatible target
- Highly controlled
- No distortion - can be applied to finished components
- Not a coating process
- **Disadvantages**
- Line of sight process
- Expensive vacuum equipment needed
- Very thin treated layer

- A vacuum process in which a beam of ions is directed at the surface and injected into it. The ions lose energy in collisions with the target atoms and come to rest in the surface layer of the material with an approximately Gaussian distribution. The ion penetration depth depends on the ion species, ion energy and target material, but is generally less than $1\mu\text{m}$. For steels the main ion used is nitrogen, which hardens the surface by forming nitride precipitates and solid solutions. The damage introduced by the implantation process also introduced a compressive residual stress which improves fatigue performance.

• Ion implantation is routinely used for semiconductor doping and treatment of expensive plastics injection moulding tools where any wear is detrimental.

Welding Processes



- **Advantages**

- Cheap
- Applicable to large components
- Localised coating possible
- Excellent adhesion

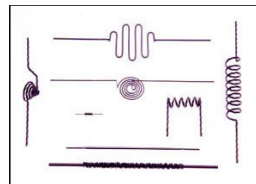
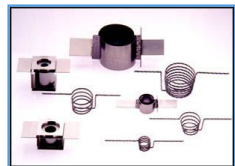
- **Disadvantages**

- Limited range of coating materials
- Minimum thickness limits

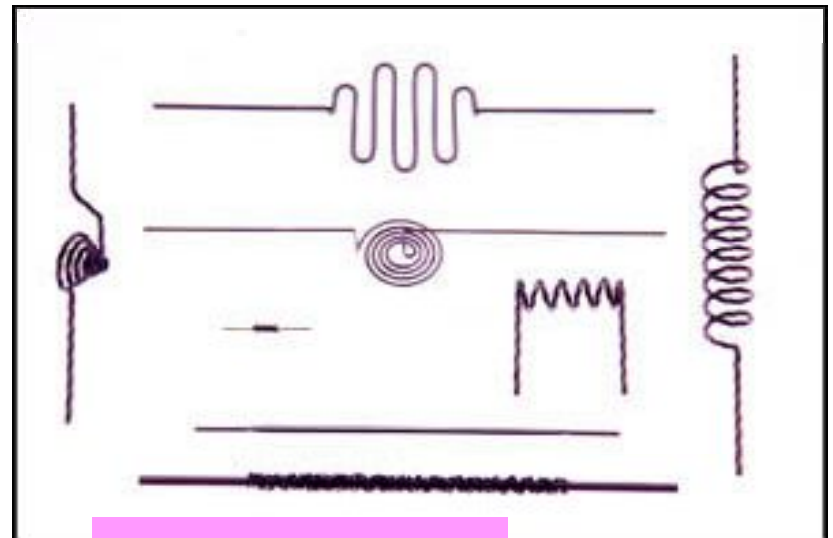
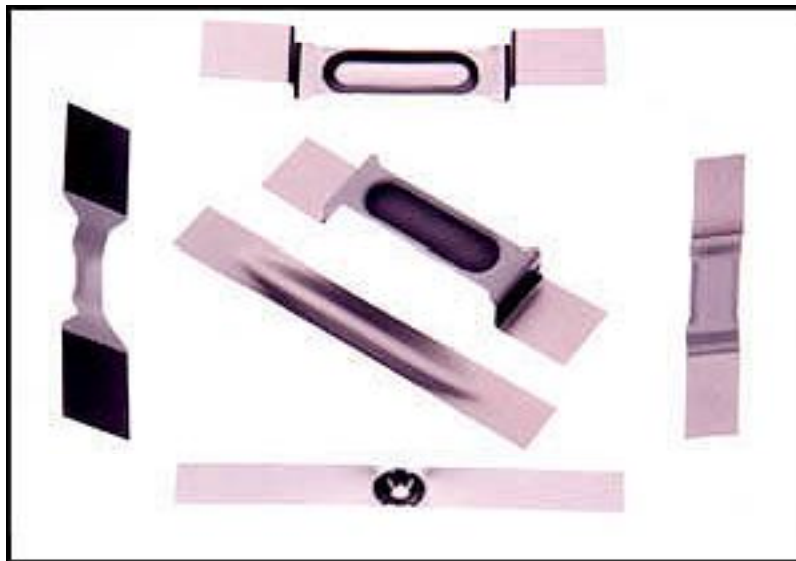
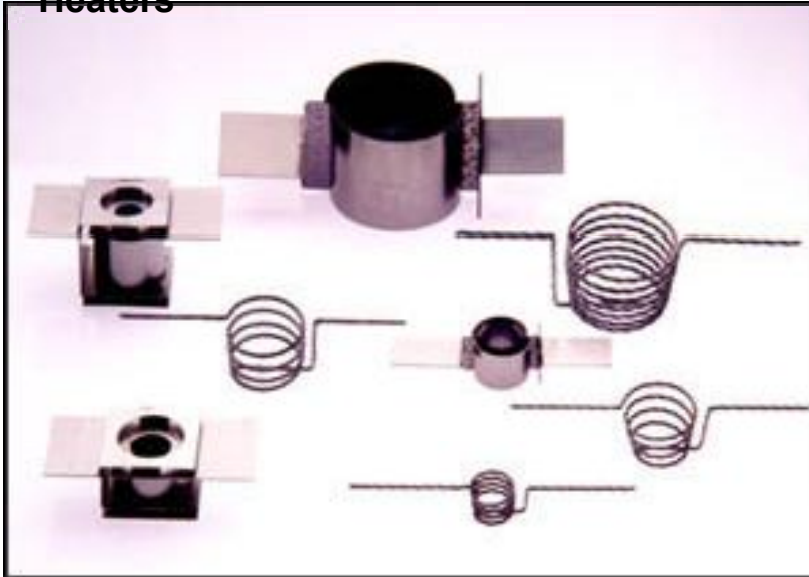
- The same methods which can be used for joining materials can be used to deposit wear resistant coatings (hardfacings). Coating materials range from low alloy steels to tungsten carbide composites.
- High deposition rates are possible and very thick coatings can be produced. It is impractical to produce layers less than 2-3mm thick.
- There can be problems with cracks in weld deposits

Thermal Evaporation in Vacuum: From Source to Substrate

we will first discuss the thermal evaporation parameters, such as vapor pressure, evaporation rates and directionality. This will be followed by a technical description of various types of evaporation sources, both resistively heated and electron beam heated. We will then discuss how to monitor and control the evaporation processes, with emphasis especially on in-process techniques.



Crucible Heaters



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Filaments
Boats

Introduction

Three key elements in a solar cell form the basis of their manufacturing technology. The first is the semiconductor, which absorbs light and converts it into electron-hole pairs. The second is the semiconductor junction, which separates the photo-generated carriers (electrons and holes), and the third is the contacts on the front and back of the cell that allow the current to flow to the external circuit. The two main categories of technology are defined by the choice of the semiconductor: either **crystalline silicon** in a wafer form or **thin films** of other materials.

سلولهای خورشیدی نوع سیلیکون

1- سلولهای خورشیدی از تک بلور سیلیکان

2- سلولهای خورشیدی از پلی کریستال سیلیکون

3 - سلولهای خورشیدی نوع آمورف سیلیکان

4- سلولهای خورشیدی نوع سیلیکون از طریق لایه های نازک

- سلولهاي خورشيدى ازتك بلور سيليكان

ساخت سلولهاي خورشيدى ازتك بلور سيليكان بدون
عيوب كريستالي و با كمترين مقدار مجاز

Dislocation (محل خالي) حائز اهميت مي باشد .

روشهاي رايج در اشل صنعتي

1- روش چكرالسكي

2 - بريجمن

3- ذوب منطقه اي

سلولهاي خورشيدى ازتك بلور سيليكان

در اين روش سيليكون در ظروفى از جنس ريخته و توسط كوره هاي RF ○ كوارتز الكتريكي يا

(راديو فرکانس) ذوب و به كمك تخمك سيليكون (Seed) تمام ذوب را به آهستگي به سمت بالا كشند

روشهاي رايج در اشل صنعتي

سلولهاي خورشيدى ازپلي كريستال سيليكون

اگر سيليكون را در شرايط نرمال در بوته هاي گرافيت ذوب و همراه گاز آرگون خنك نمايند. سيليكون از كريستالهاي ريز ميكروسكوپيك تشكيل ميشود ، اگر شرايط خنك كردن به تدريج و خيلي آهسته باشد كريستالهاي ريز امكان بزرگ شدن را پيدا مي نمايند كه به اين محصول پلي كريستال سيليكون مي گويند كه اندازه كريستاليزه شدن دانه ها بستگي به شرايط خنك شدن انجماد دارد.

پلي كريستال سيليكون را مي توان بصورت هاي گوناگون از قبيل استوانه اي، مكعب مستطيل، نواري و يا ودقه اي نازك از حمام مذاب باكيفيت عالي رشد داد.

سلولهاي خورشيدى ازپلي كريستال داراي بازدهي 10-12% مي باشند

سلولهاي خورشيدى نوع آمورف سيليكان

سيليكون به صورت آمورف (بي شكل) مي توانند يكي از پروسه هاي ساخت سلولهاي خورشيدى باشد.

آمورف سيليكون داراي خواص فيزيكي كريستالين نمي باشد و همين علت در موقع تشكّل، اتمهاي آن بصورت اتفاقي و بي نظم و ترتيب توزيع و قرار مي گيرد.

آمورف سيليكون را توسط سيستمهاي تبخير و پلاسما اسپري تهيه نمود.

Electron beam Deposition System



Vacuum : 5×10^{-7} torr

Multi source: 4 – 6 S

Power : 4 – 6 Kev

In-situ electrical measurements



- سلولهاي خورشيدى نوع سيليكون از طريق لايه هاي نازك

اين نوع سلولها خورشيدى با خلوص بالا از طريق سيستمهاي تبخير از فاز مذاب به روشهاي

1. [Molecular beam Epitaxy](#) and
2. [Electron beam Evaporation](#) ,
3. Chemical Vapour Deposition
4. Vapour Phase Epitaxy, etc. under UHV condition

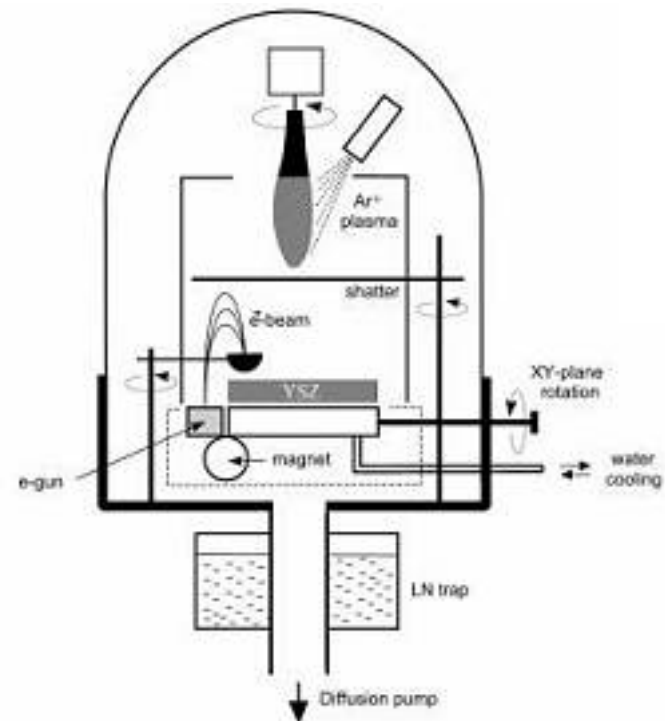
بروي بالشتكهاي تك كريستال با صفات كريستالي مشخص رشد داد

Electron beam Deposition

Schematic Sputtering



Schematic Electron beam



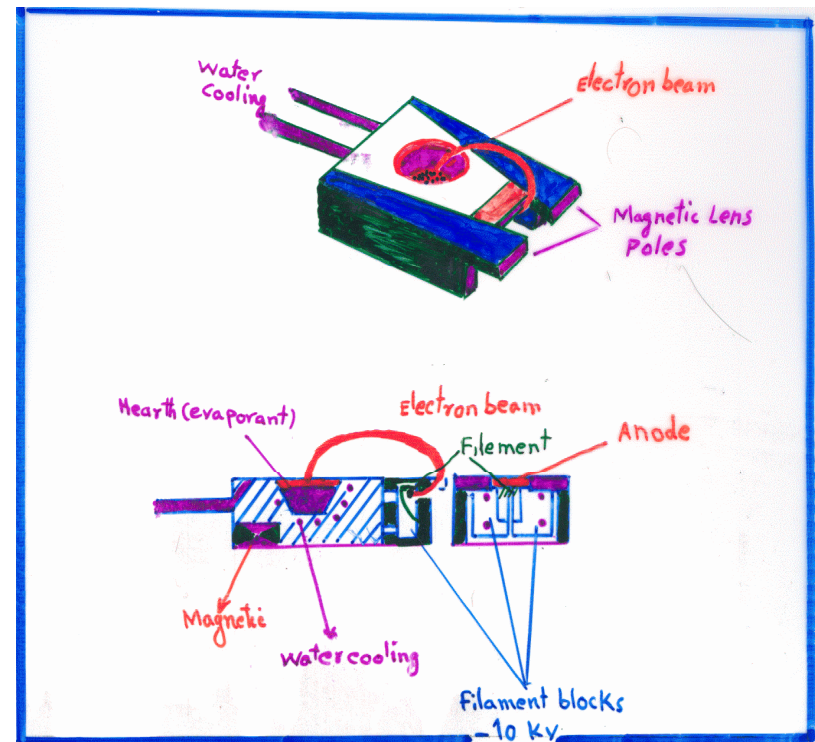
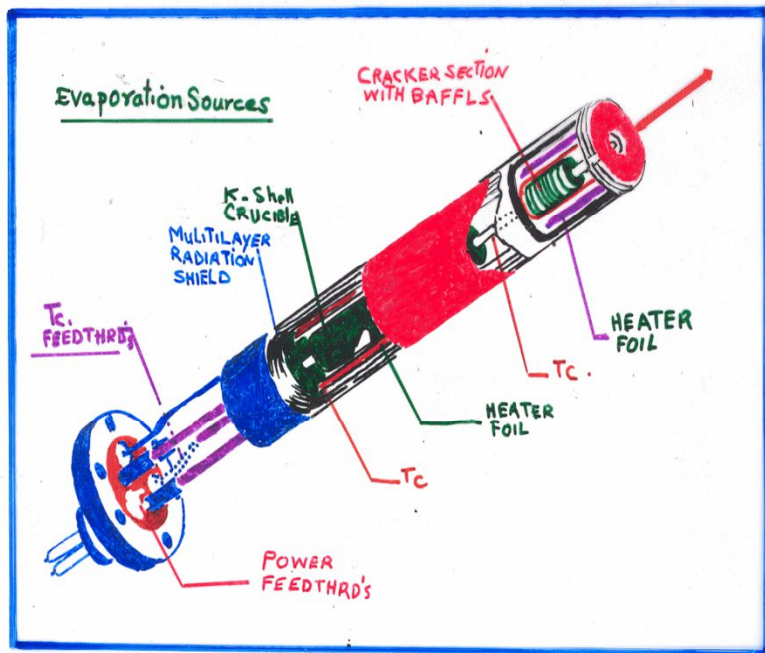
Gas Sensor

Electrical properties of SnO₂ films deposited using various techniques.

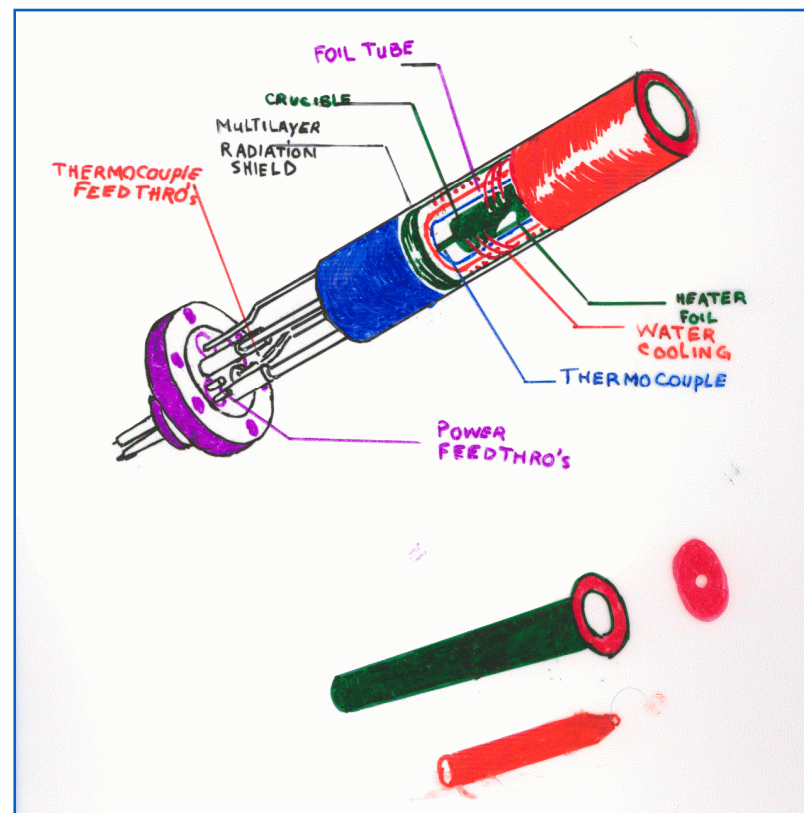
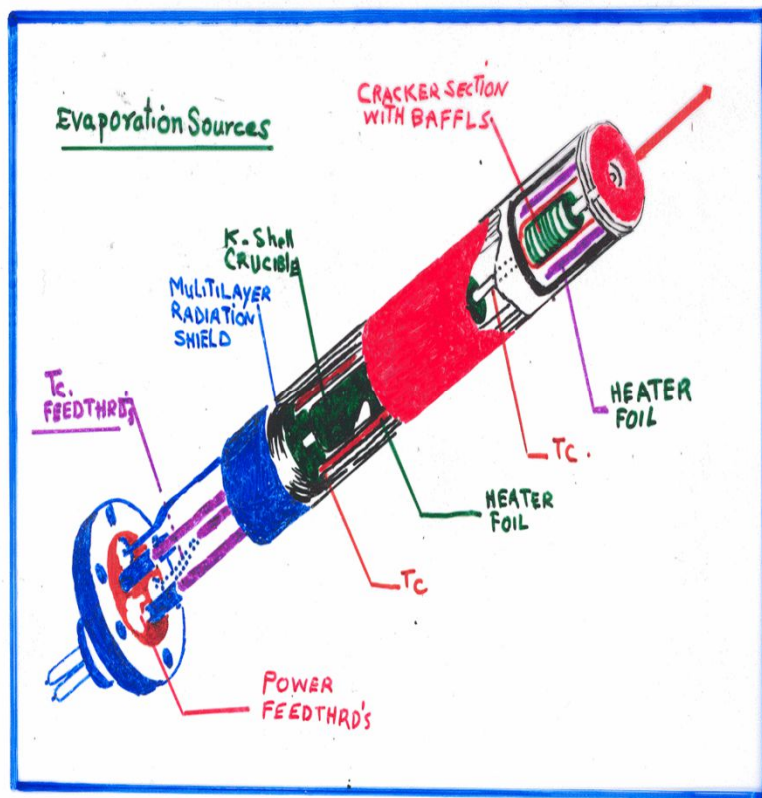
Method	Ref	ρ (Ω -cm)	n (cm ⁻³)	μ (cm ² /v.sec)
Spray Pyrolysis	47	1.46×10^{-3}	0.80×10^{21}	10
CVD	48	1.42×10^{-3}	6.00×10^{20}	7.8
RF Sputtering	49	2	1.60×10^{18}	1.1
Re active evaporation	50	7.20×10^{-3}	9.00×10^{19}	9.6
Electron beam evaporation	51	7.00×10^{-2}	7.70×10^{19}	11.6
Reactive electron beam evaporation	52	7.50×10^{-4}	3.20×10^{20}	26

Merits and demerits of various thin films deposition method used for sensor fabrication

Method of preparation	merits	Demerits
Sputtering	Mostly used method at low deposition temperatures. No post deposition heat treatment required. Fine thickness control. Easy to dope with noble metals.	Difficult to accommodate multiple targets. In-situ masking not possible. Complex operation.
Electron beam evaporation	Often used. Reasonable uniformity over large areas. Reasonable thickness control. Easy to accommodate multiple boats. In-situ masking is possible, easy operation .excellent to dope with noble metals.	Post deposition heat treatment is necessary. Few way to control the quality of the films.
Resistive evaporation	Often used.reasonable uniformity.is possible.	No thickness control. Difficult to dope with noble metals.post deposition heat treatments is necessary.
Activated reactive evaporation	Scarcely used. Tachometric films at low temperature.reasonable uniformity	Complex operations. Difficult to dope with noble metals.in-situ masking difficult.no thickness control.
Spray pyrolysis / CVD Back	Cheap and easy technique no post deposition heat treatment.	No thickness control. Poor uniformity. Difficult to dope metals. No in-situ masking.

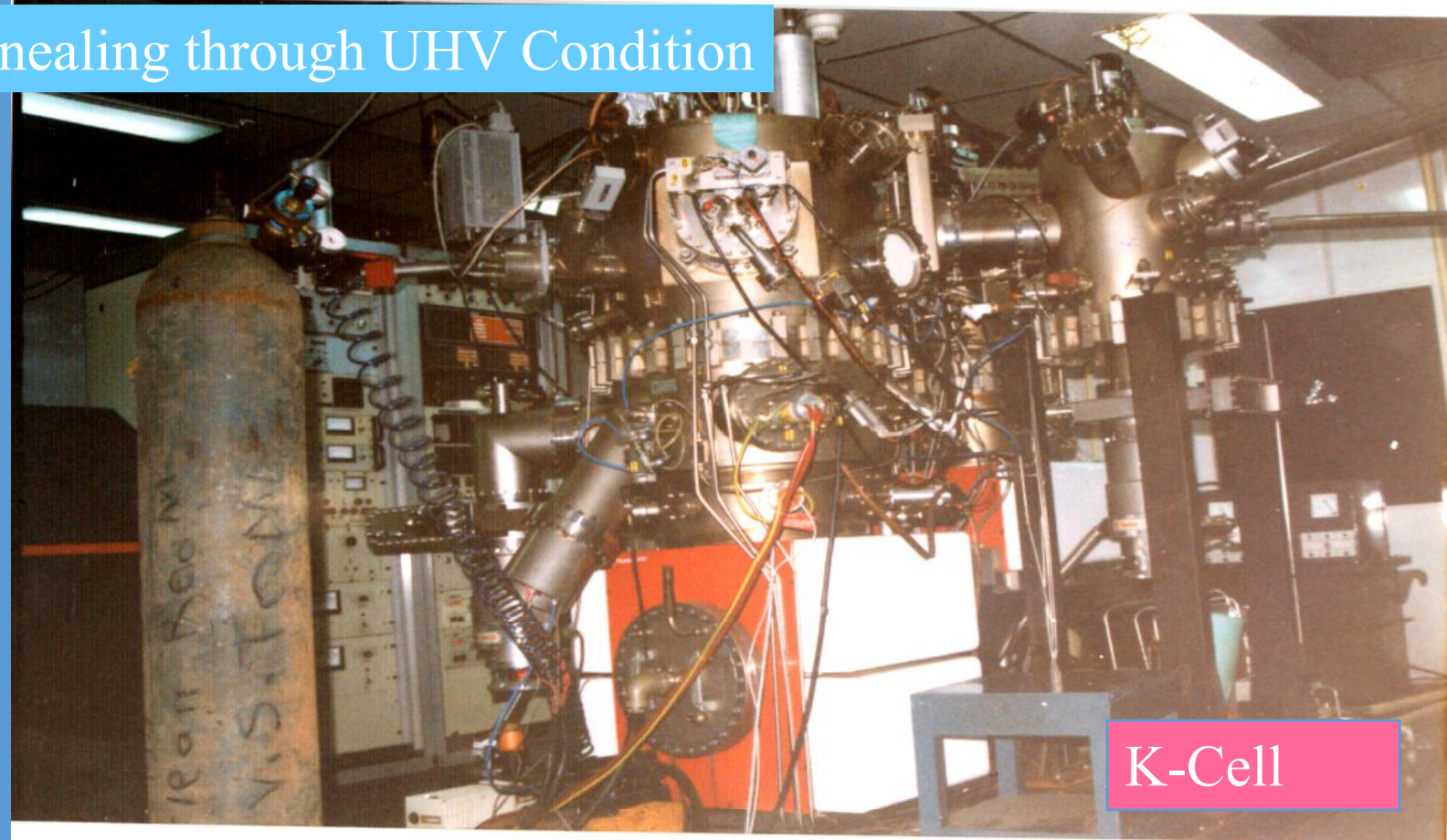


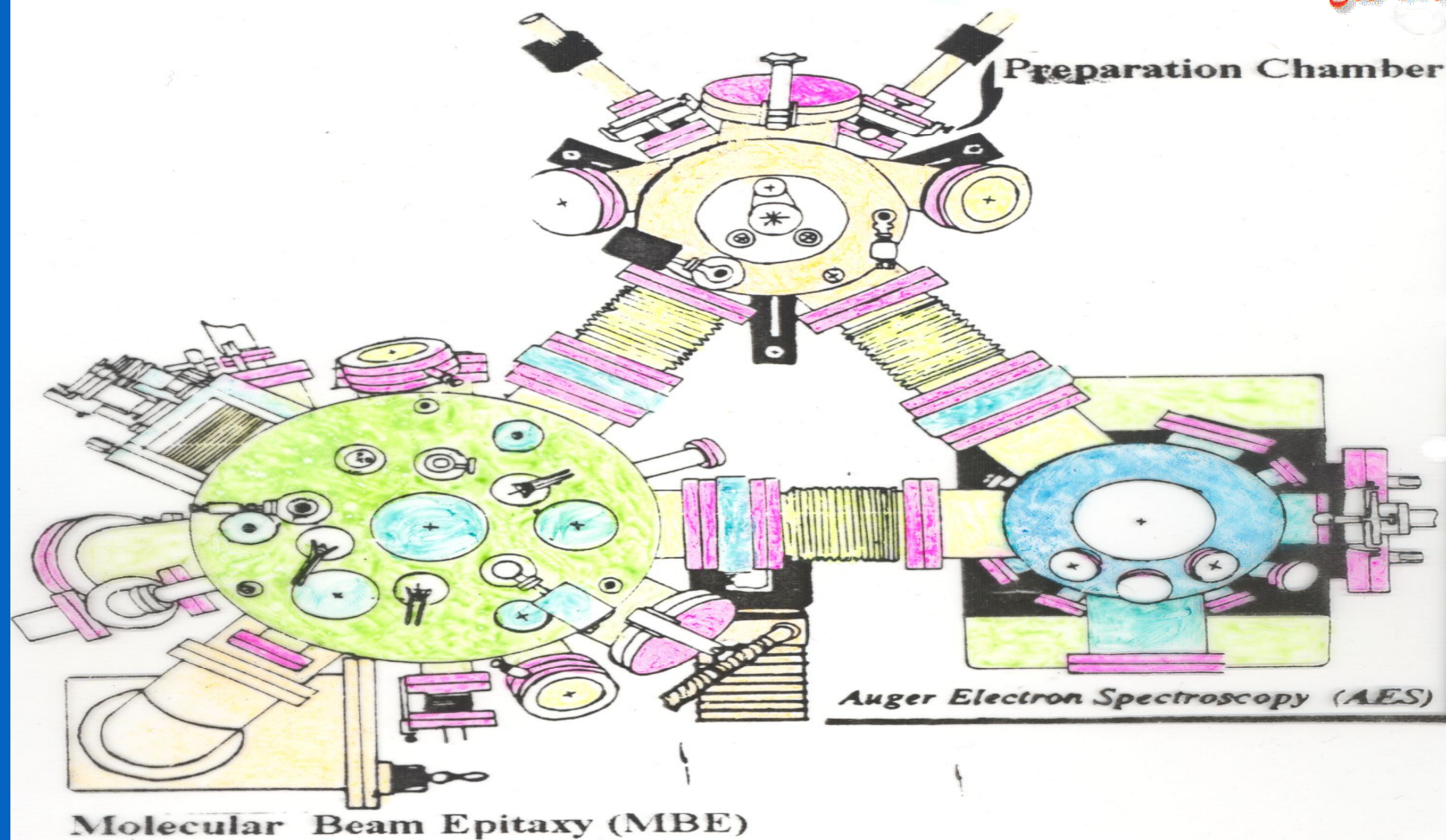
K-Cell



Molecular beam epitaxy

Annealing through UHV Condition



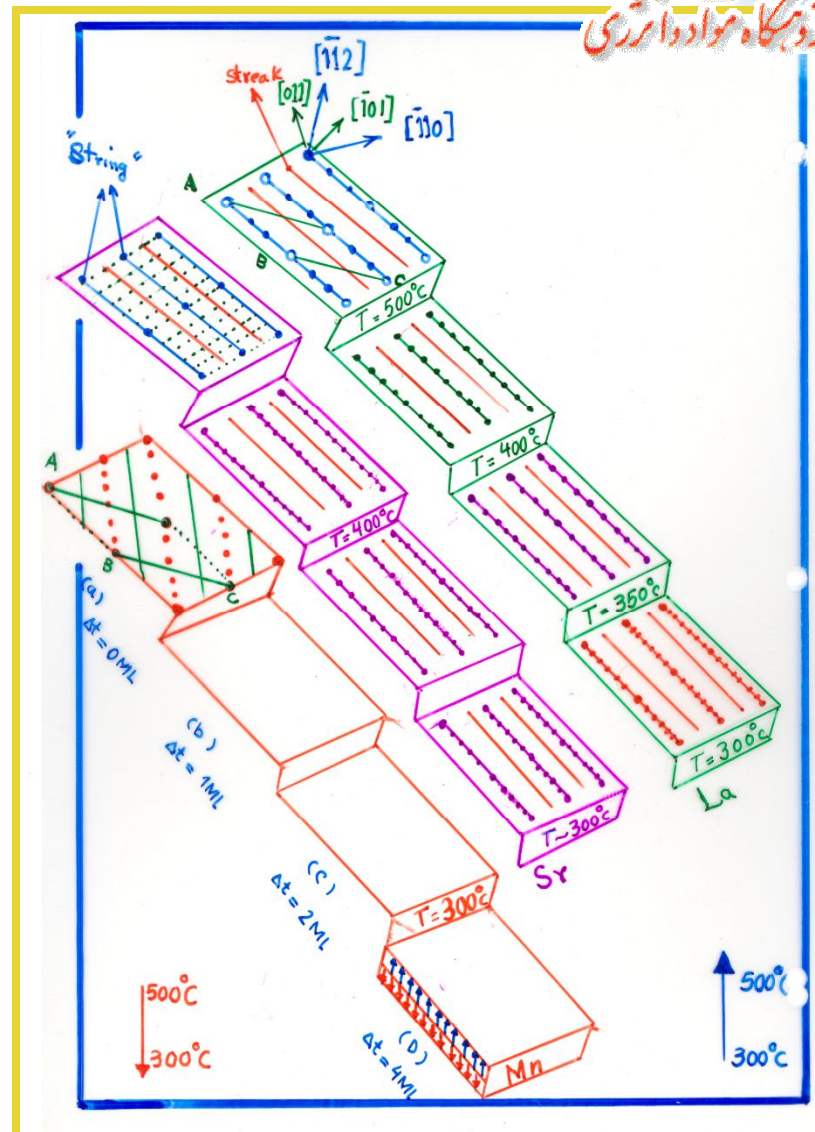


Annealing through UHV Condition

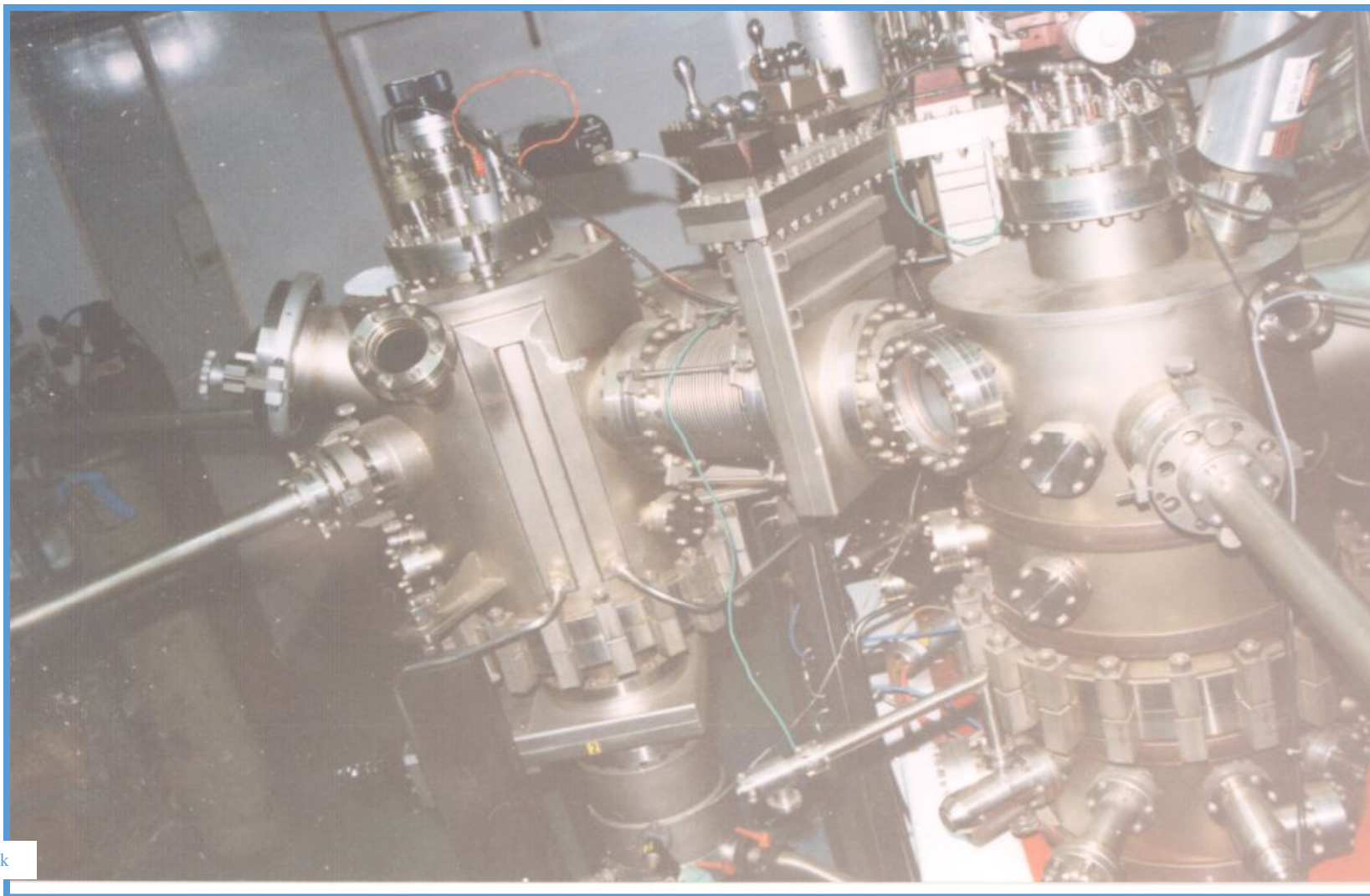


Sample preparation and characterization

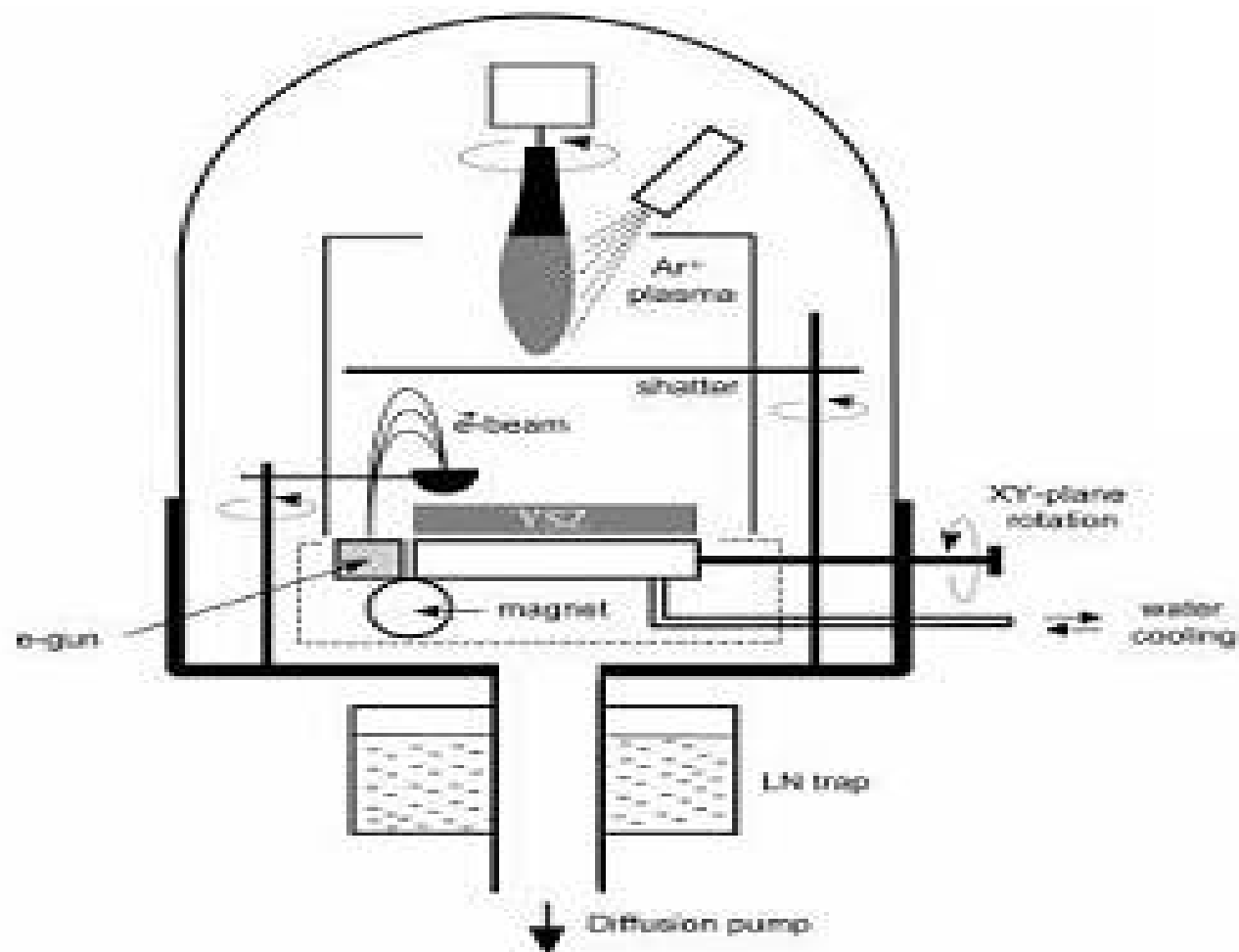
1. Substrate Cleaning(wafer) :Org/ Shiraki process
2. Substrate Cleaning Monolayer : Ion Source etching
II Gridless ion source
3. Growth Chamber : MBE
4. Vacuum MBE system : 3×10^{10}
5. Vacuum during evaporation : 5×10^{-9}
6. Confirmation atomic adsorption gases: Mass spectroscopy
7. The technique evaporation deposition : CVD
8. Target Substrate deposition : Si (WAFER)
9. Substrate Temperature : 500°C
10. Sample thickness composition analysing : Quartz Crystal monitor
mean & two as coarsen and coalesce into uniformity layers.
11. Surface a uniform layer growth : RHEED
12. Interaction Energy level bond : AES
13. The deposition rates in order
are generally appropriate : $0.01\text{-}\text{\AA}_s^{-1}$



Annealing through UHV Condition



Schematic Electron beam



Schematic Sputtering

