50 YEARS ANNIVERSARY OF STARTING THE IN SITU UHV TEM STUDIES OF THIN FILM GROWTH IN BUDAPEST

Plenary lecture

Dedicated to the memory of late Professor J.F. Pócza* on *the occasion of the 100 years anniversary of his birthday

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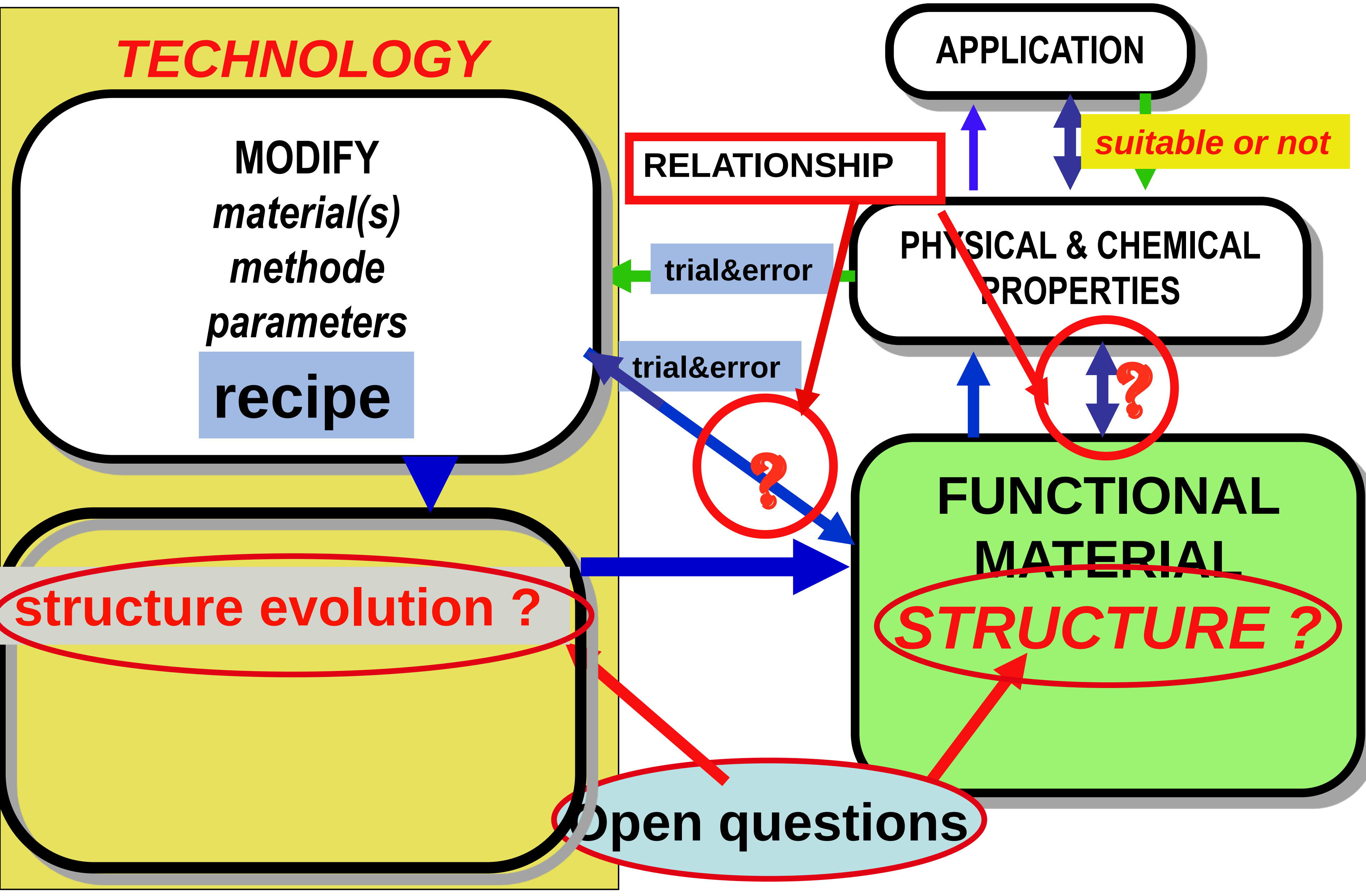
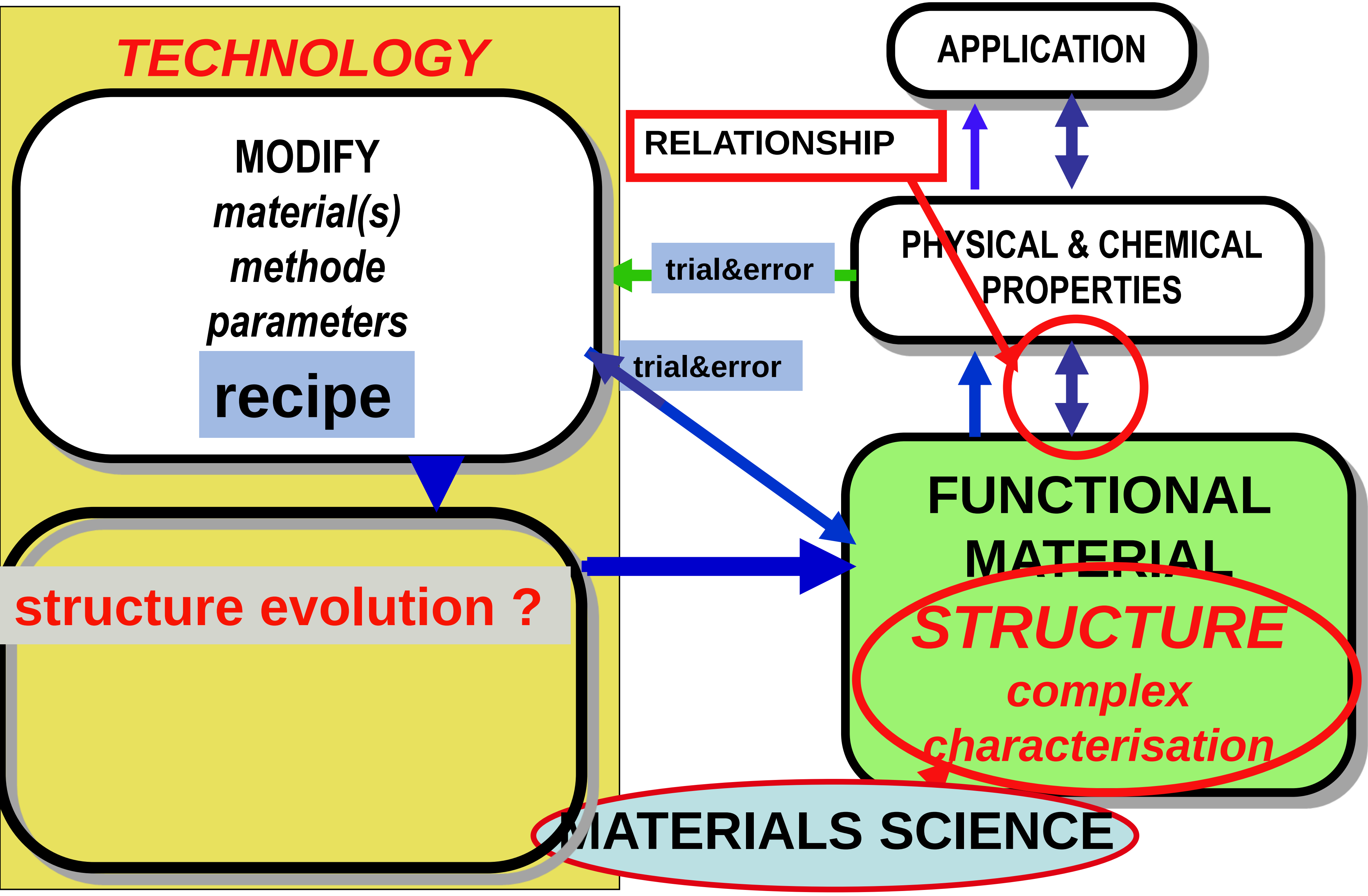
People dealing with functional materials were greatly excited in the middle of 20th century

- **commercialization of materials structure investigation methodes and devices (*X-ray diffraction, electron microscopy*..........) provided the possibility to characterize the materials structure at submicroscopic (nm) level (opening the golden age of materials science)**

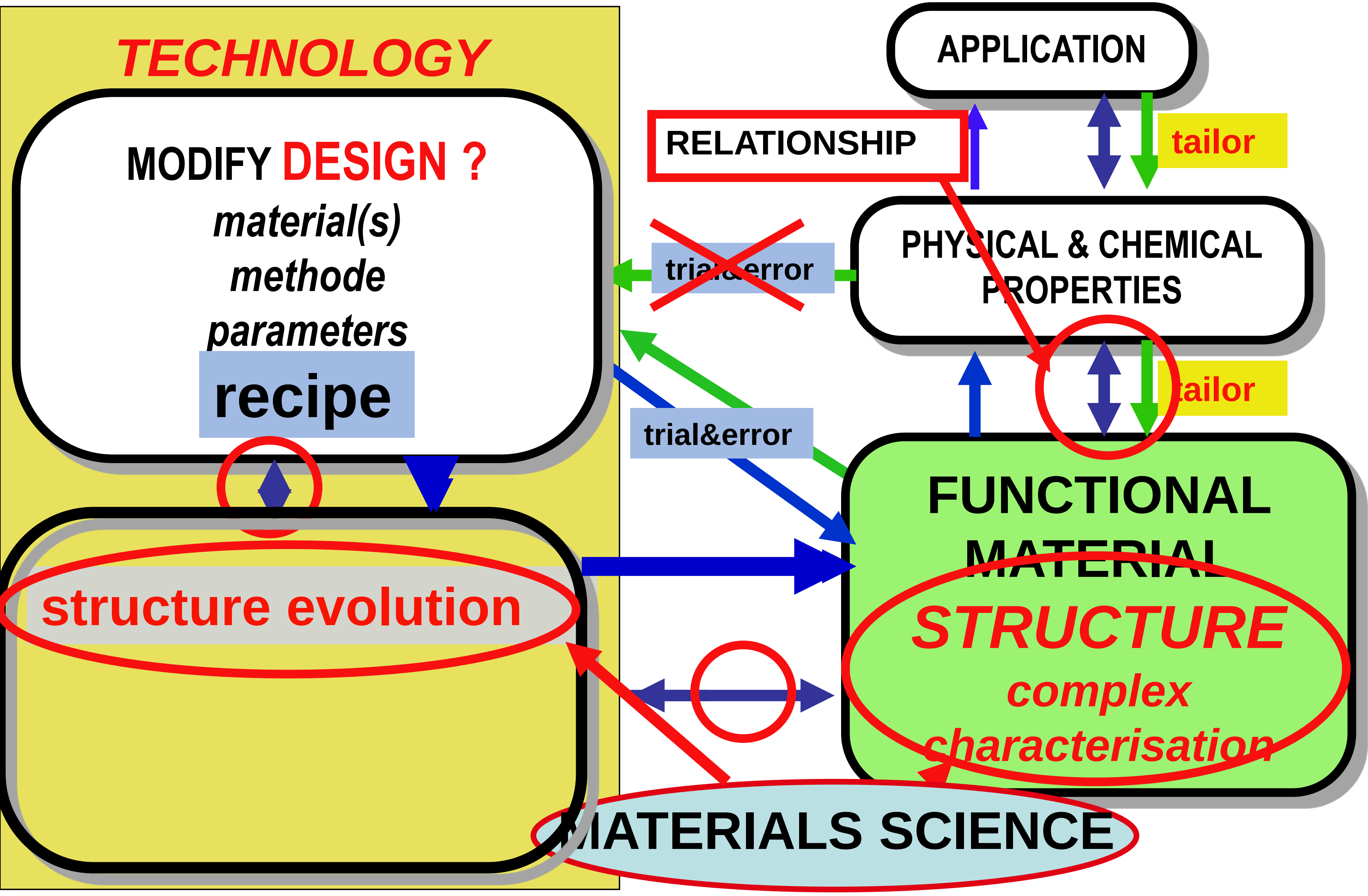
CONVENTIONAL way to discover new functional materials and improve their quality was the TRIAL&ERROR



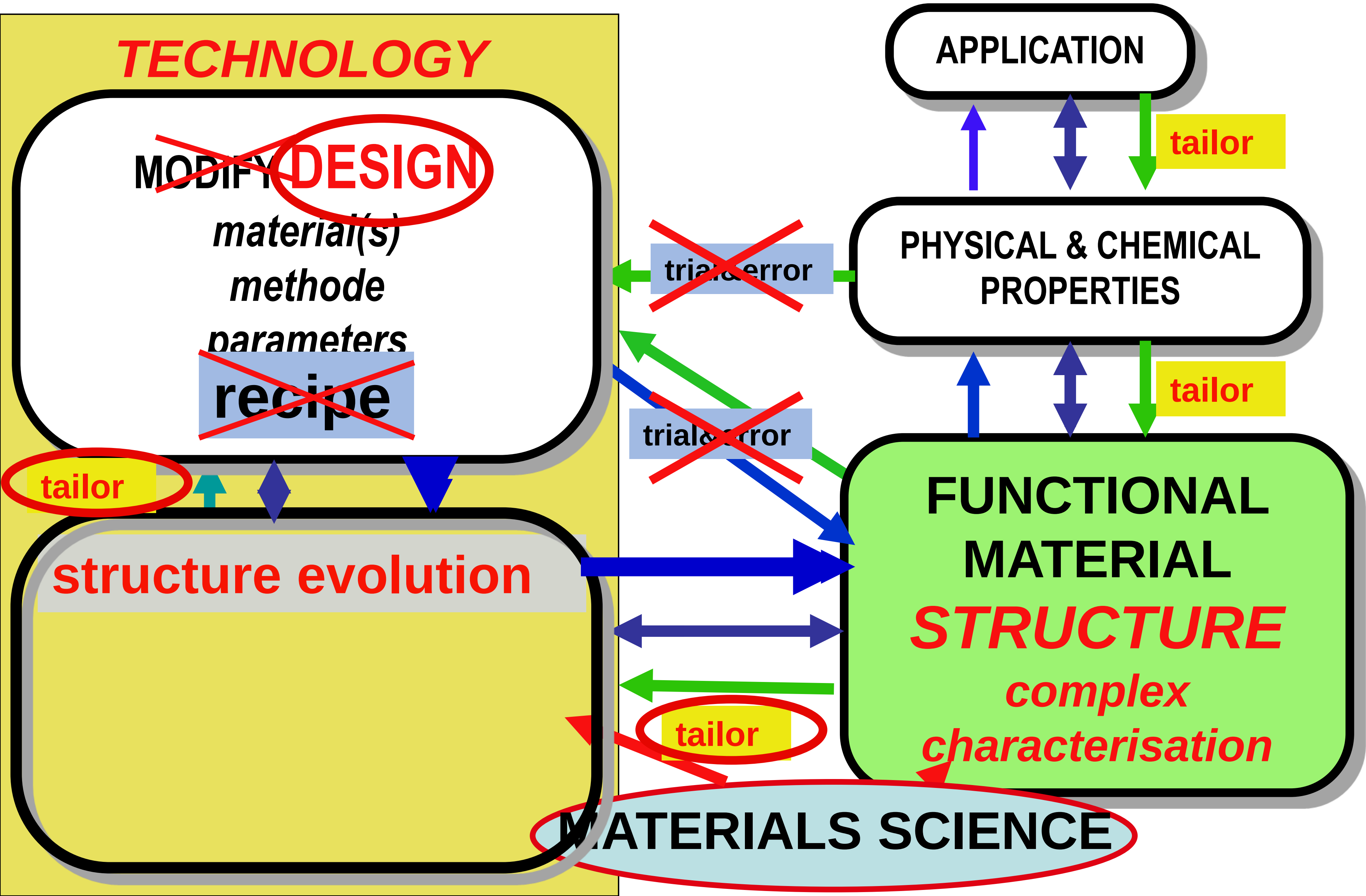
STRUCTURE CHARACTERISATION made possible to determine the fundamental relationship



STRUCTURE CHARACTERISATION make possible to determine the fundamental relationship



MATERIALS SCIENCE makes possible the tailoring of structure and technology to develop and improve advanced functional materials



People dealing with functional materials were greatly excited in the middle of 20th century

- commercialization of materials structure investigation methodes and devices (*X-ray diffraction, electron microscopy*..........) provided the possibility to characterize the materials structure at submicroscopic (nm) level (opening the golden age of materials science)

- Discovery of single crystal and polycrystalline thin films (from monolayer to µm thicknesses) constituting of submicroscopic particles provided the possibility to prepare designed sophysticated material structures with new properties by the atom-by-atom deposition

MATERIALS SCIENCE in THIN FILM RESEARCH

Preparation of thin films:
atom-by-atom deposition in a vacuum system

PRODUCTION
(source)
of
material(s) species
to be deposited

⇩

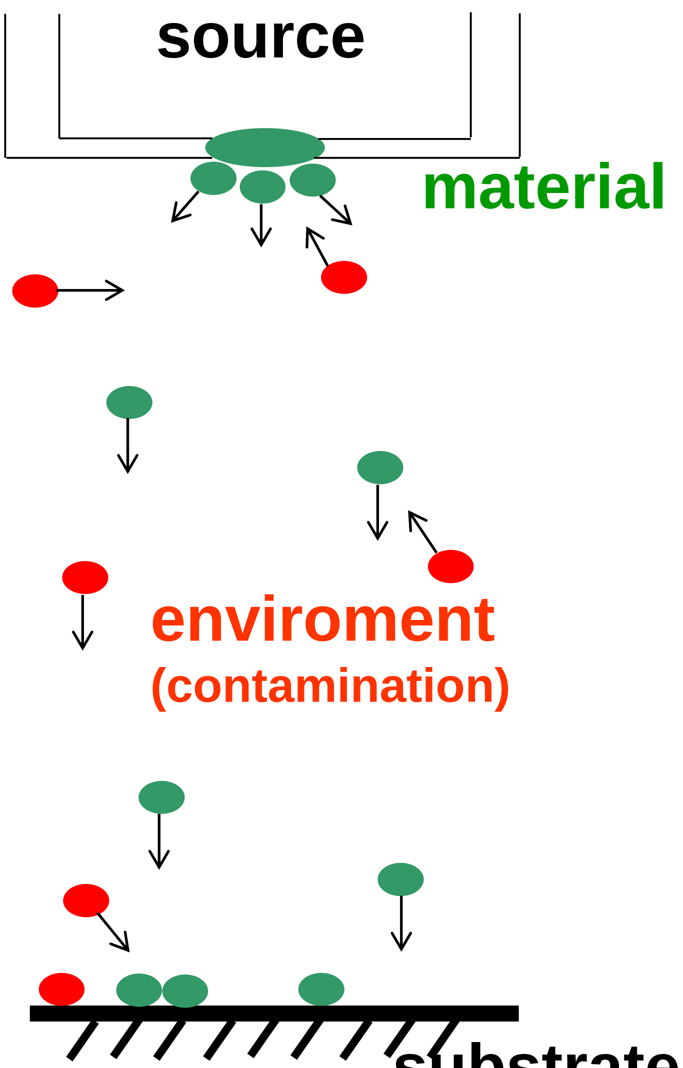
TRANSPORT
of
material species
to
the substrate
(enviroment)

⇩

DEPOSITION
of
material-enviromental (impurity) species
on
the substrate

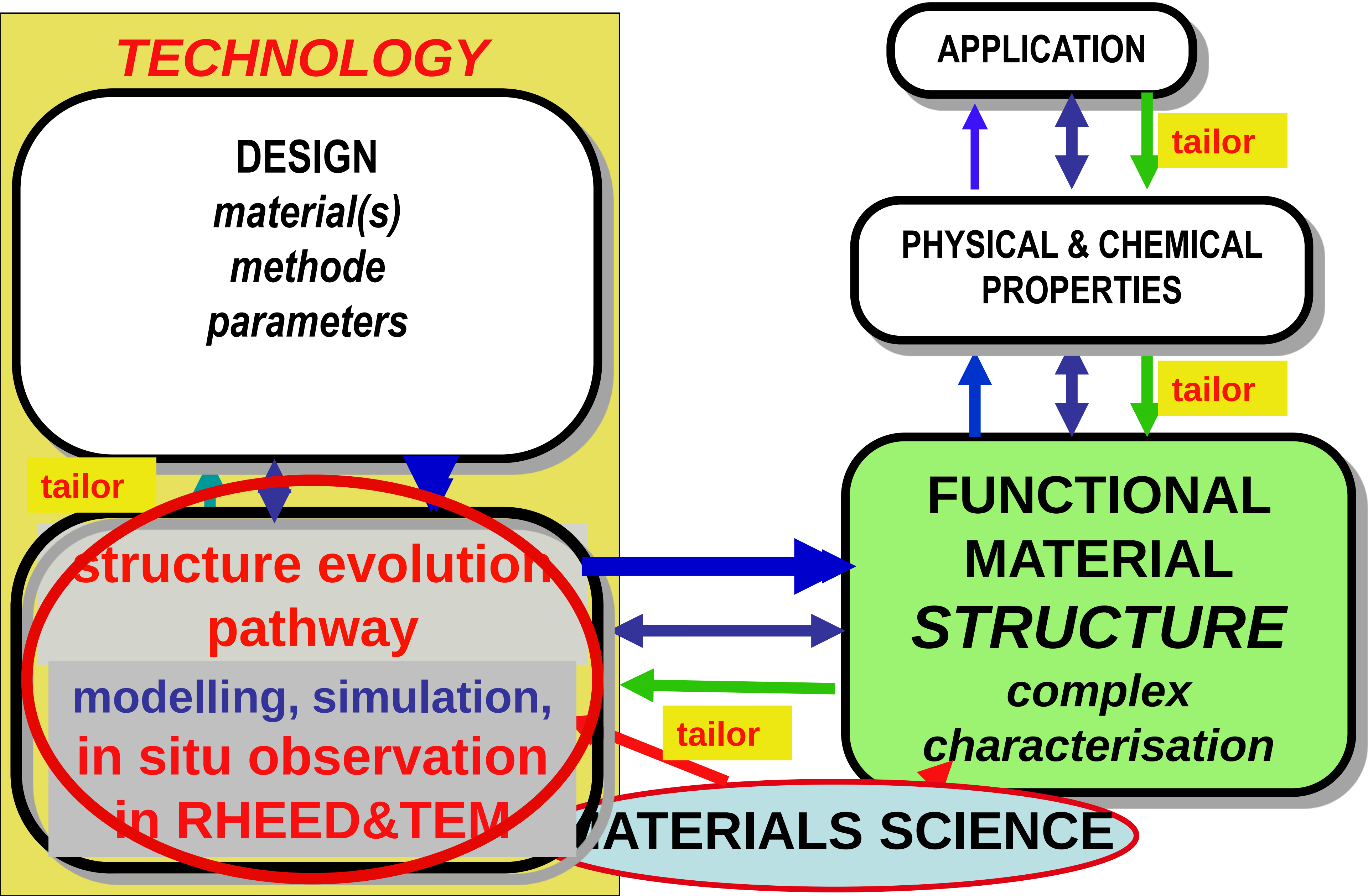
⇩

EVOLUTION
of
the thin film structure



P_3_1_3

Vapour deposition of thin films could be realized in electron diffraction units and TEM



Research teams doing pioneering work to study thin film growth in RHEED and TEM (dates of publication)

T. A. McLauhlan, 1950 (HV - RHEED)
M. Takagi, 1954 (HV - RHEED)
K.J. Hanszen, 1954 (HV -RHEED)
G. A. Bassett, 1958 (HV - TEM)
D. W. Pashley, M.J. Stowell 1964 (HV - TEM)
H. Poppa, 1965 (HV+ cryo - TEM)
J. F. Pócza, Á. Barna, P. B. Barna, 1967 (UHV – TEM + electrical measurements)
G. Honjo, K. Jagi, 1969 (UHV - TEM)
H. Valdre, D.W. Pashley, M.J. Stowell 1970 (UHV - TEM)

Review

Helmut Poppa, ***High resolution, high speed ultrahigh vacuum microscopy, J. Vac. Sci. Technol. A 22 (2004)1931 - 1947***

OUTLINE

- initiatives of Prof. Pócza in thin film research (1950/1960)

- the UHV in situ TEM experimental system constructed in the Budapest research team

- fundamental phenomena and pathway of structure formation of polycrystalline films: case studies of pure In and C doped In thin films

**** deposition on amorphous substrate: vapour-liquid-solid phase transformation, texture evolution (VIDEO),***

**** fundamental phenomenon of nano-composite structure formation: encapsulation of crystals and repeated nucleation (VIDEO)***

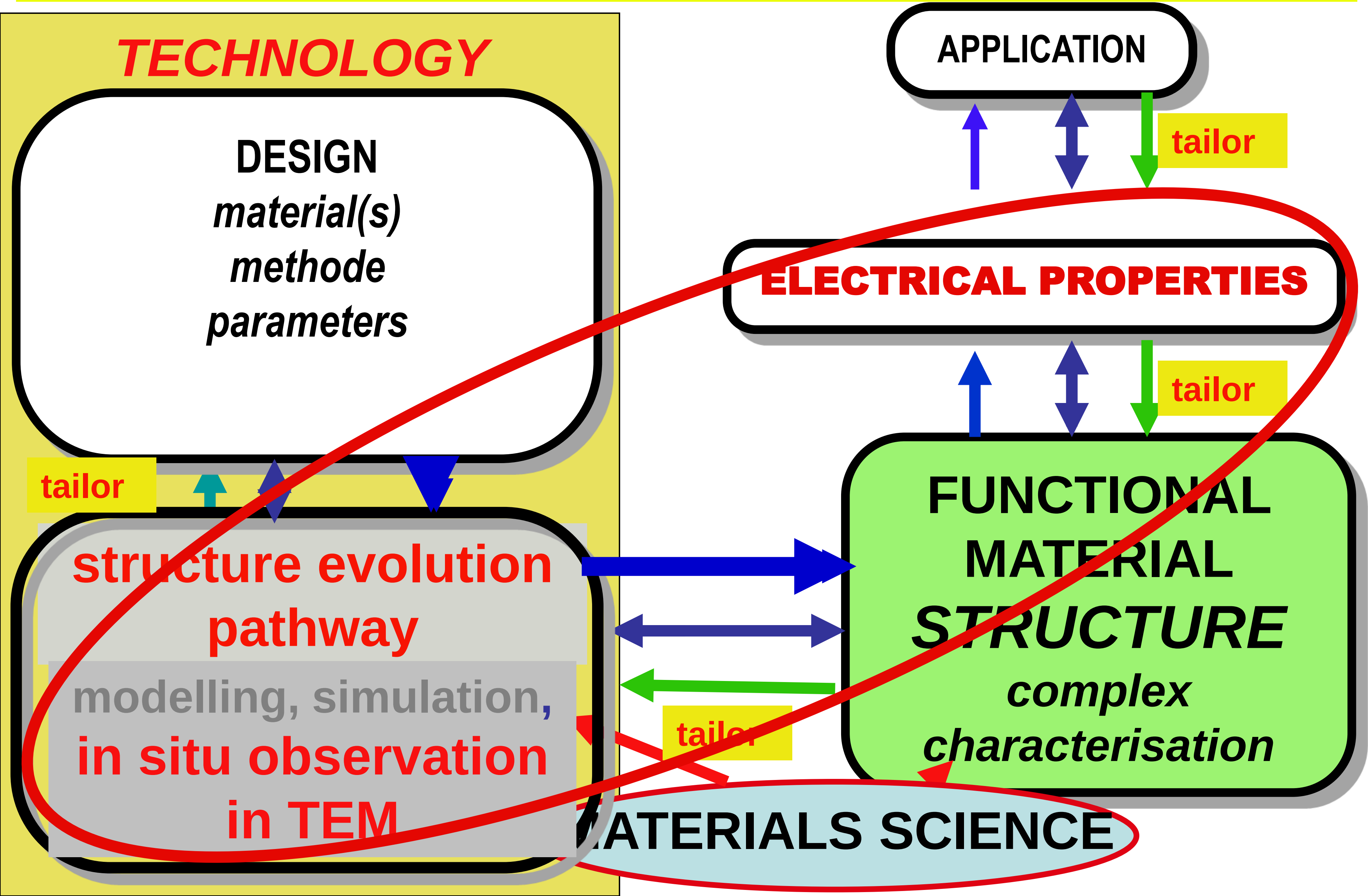
epitaxial growth of In, effect of substrate surface contamination, deposition on single crystal MoS_2 substrate (VIDEO)

- conclusions

In 1950th the RESEARCH PHILOSOPHY of
Prof. J.F. Pócza (1915-1975),
fouder of the Budapest research team was:

Understanding
the structure evolution
and
the structure - property relations
are the key issues of thin film research and technology

Research phylosophy of Prof. J.F.Pócza
realize experiments in UHV TEM



FACILITIES of the In-SITU TEM EXPERIMENTAL SYSTEM developed and constructed in the frame of a PhD work (1963-1978)

Á. Barna: ***UHV in situ TEM experimental system for simultaneous investigation of structure evolution and electrical properties of thin films (1982)***

parameters	structure	electrical measurements

during deposition

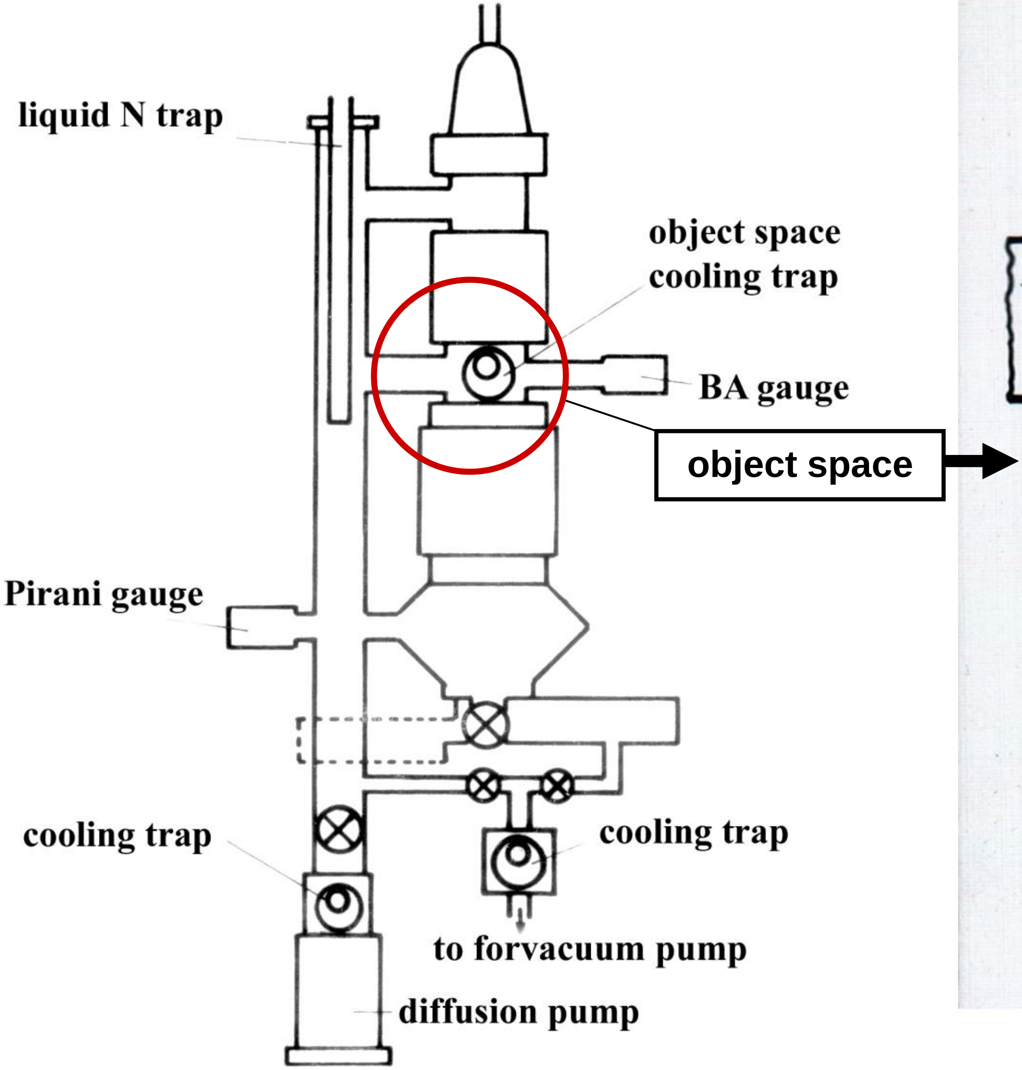
parameters	structure	electrical measurements
deposition rate 0,05 - 2 nm/s **substrate temperature** -150^0C - $+500^0C$ **residual pressure** 10^{-8} Pa plate, 10^{-6} Pa film **gas atmosphere** < 10^{-1} Pa	**TEM images** **selected area ED** **low angle TED**	**resistivity** (thermo power)(1-3 sec)

during post-deposition treatment

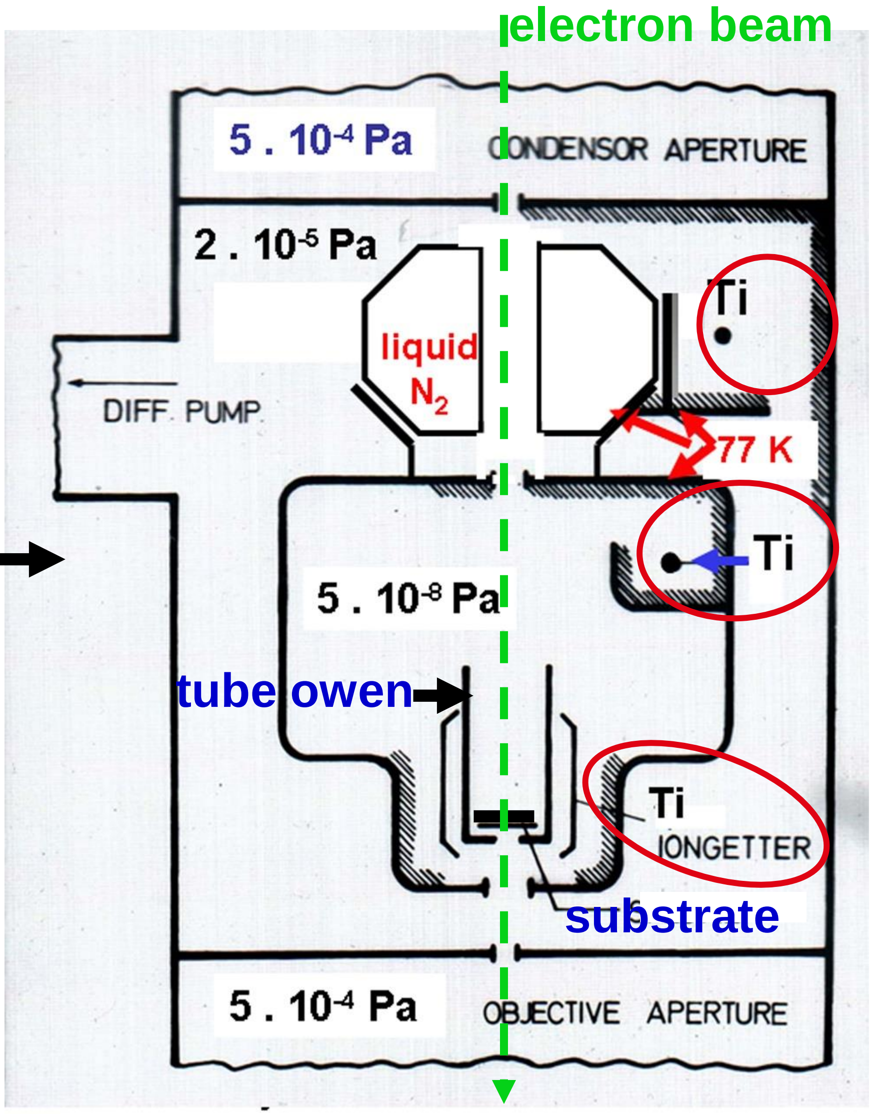
parameters	structure	electrical measurements
heat treatment **gas interaction**	**TEM images** **selected area ED** **low angle TED**	**resistivity** **thermo power** **Hall voltage** **magneto-resistivity**

The vacuum system of the UHV in situ experimental system

Á.Barna, P.B.Barna, J.F.Pócza: Vacuum, 17, (1967) 219-221



Vacuum system of JEM 6A TEM



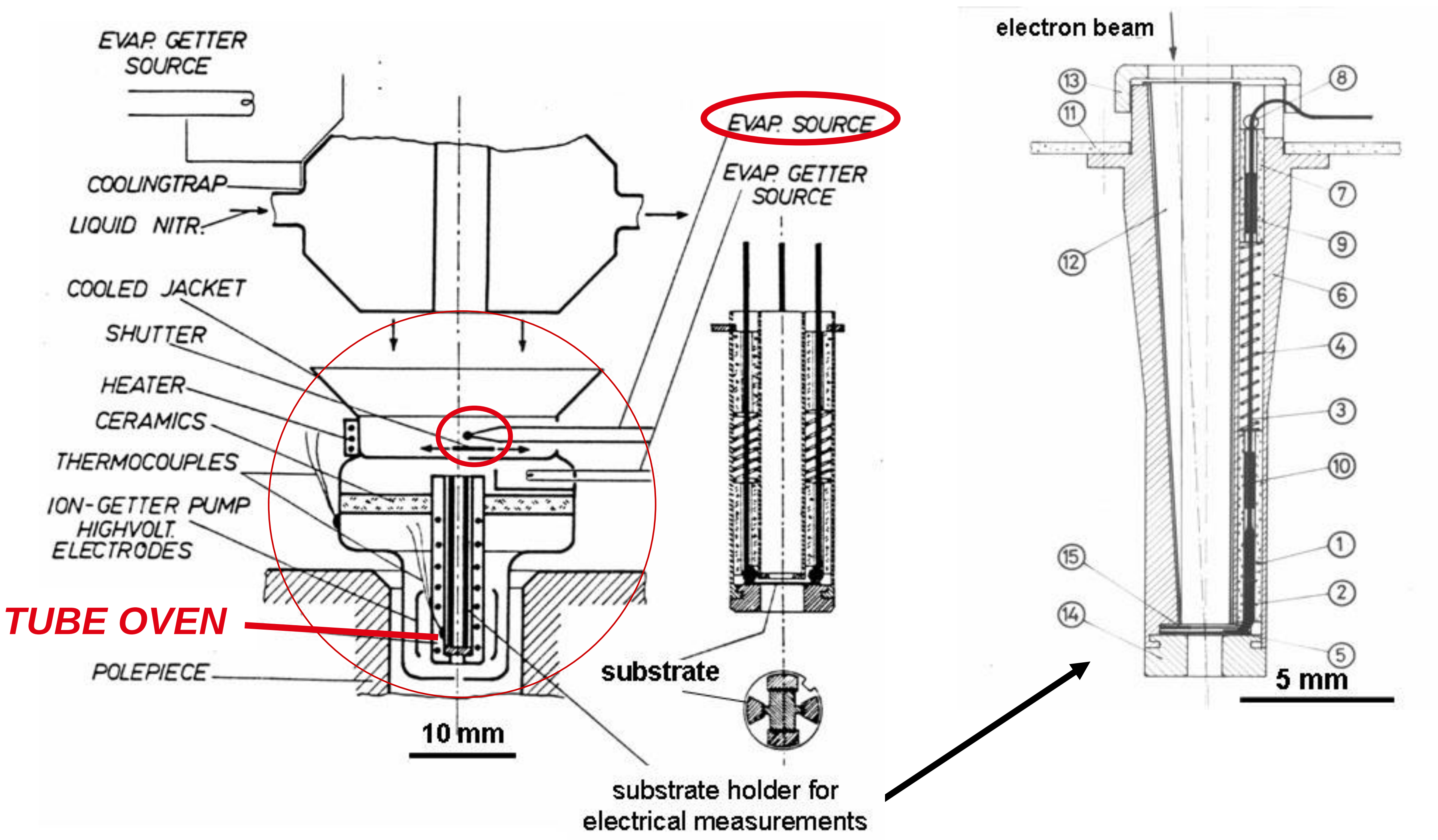
Vacuum system of the in situ experiments

Scheem of the UHV in-situ experimental system dedicated to electrical measurements

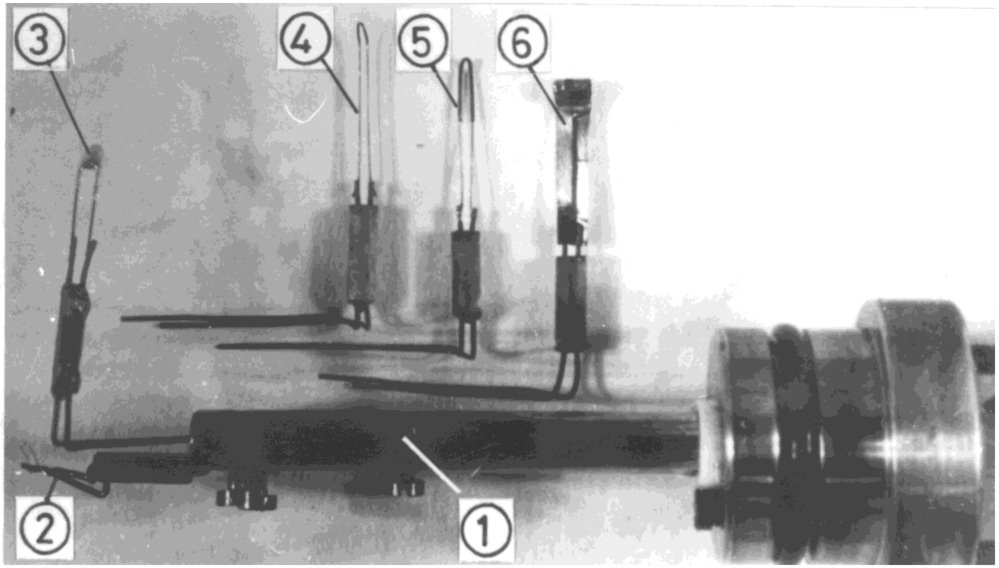
Á.Barna, P.B.Barna, J.F.Pócza: Vacuum, 17, (1967) 219-221

Á. Barna, P.B. Barna, J.F. Pócza: Septiéme Congr. Int. Micr. Électronique, Grenoble, (1970) pp. 445-446.

Á. Barna, P.B. Barna, G. Stark, P. Thomas, L. Tóth: Proc. 7th Int.Vac.Congr. Vienna, 1977. Vol. II. p. 1635

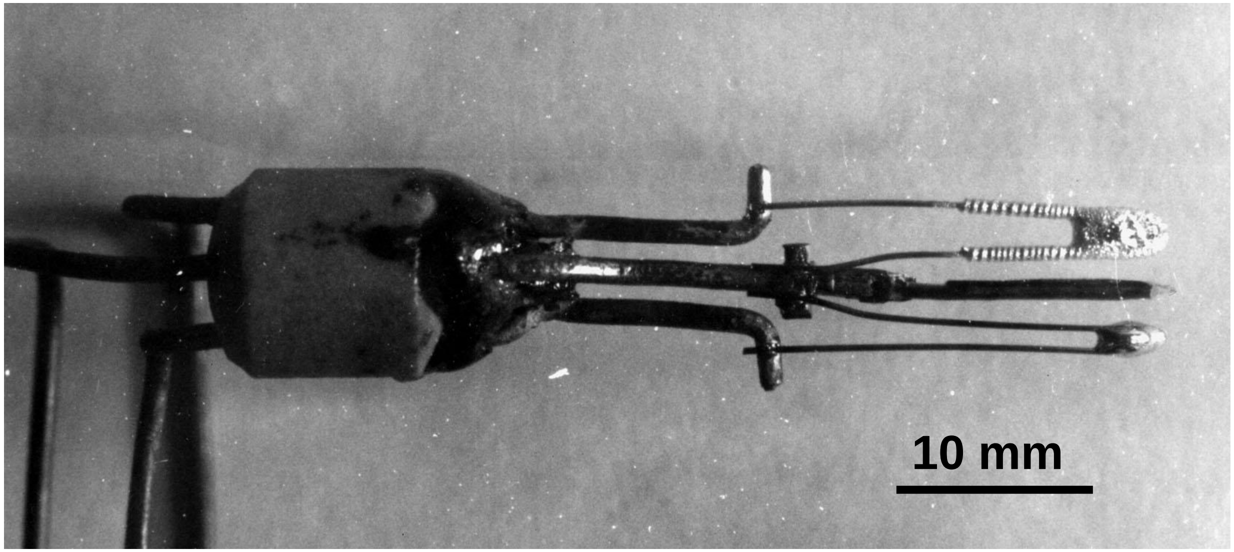


The evaporation sources for various materials

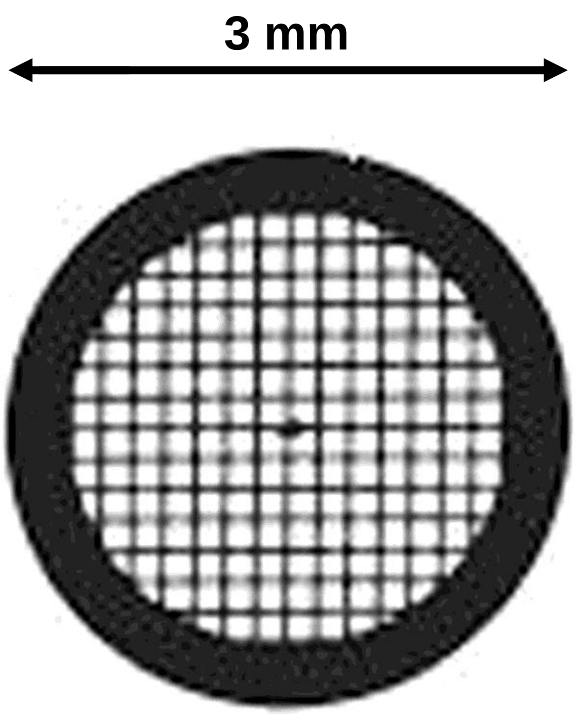


1 source holder
2 optical pyrometer
3 Al
4 Au
5 Ge
6 miniature crucible for Sn, Bi

source for sequential deposition of Al and Au

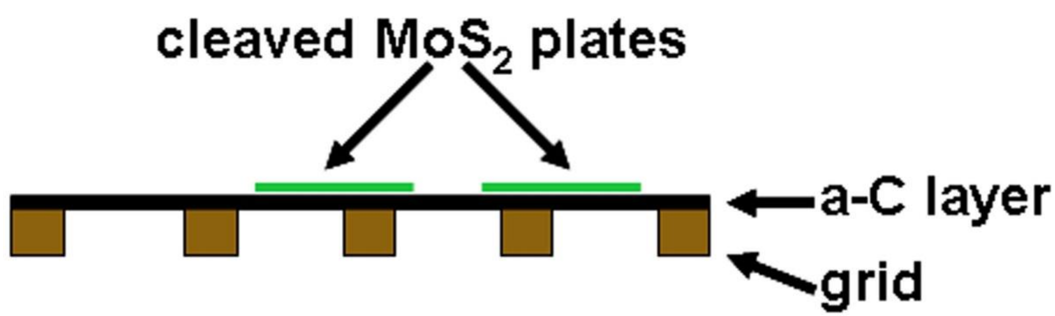


a-C or SiO_2 layer substrates were prepared on TEM microgrids



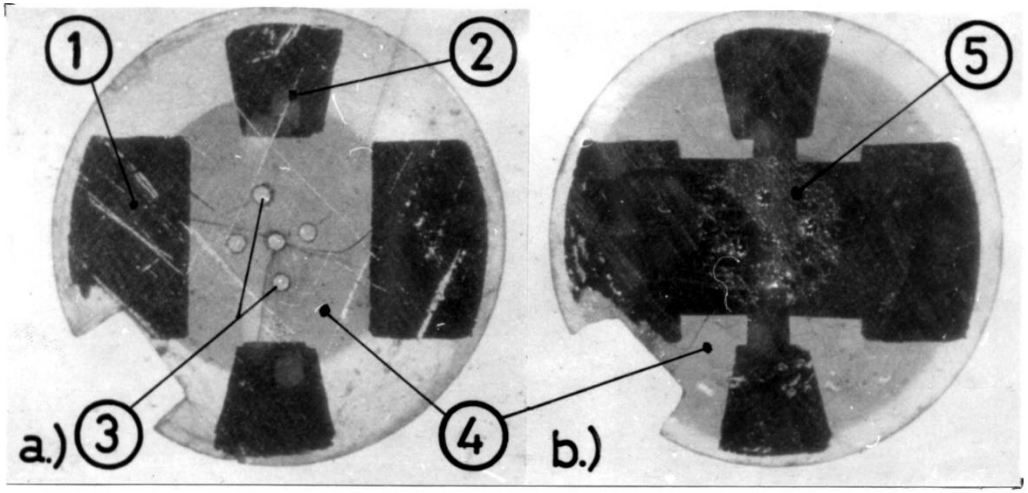
The metall microgrid

Substrate for polycrystalline film growth and texture evolution onamorphous substrates

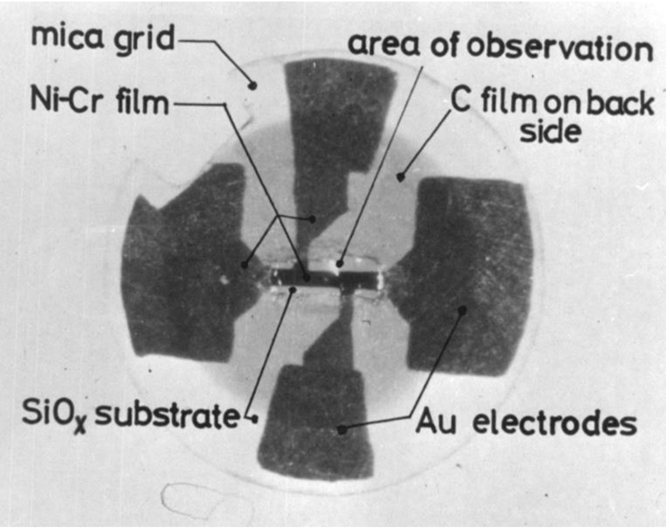


Substrate for epitaxial growth experiements

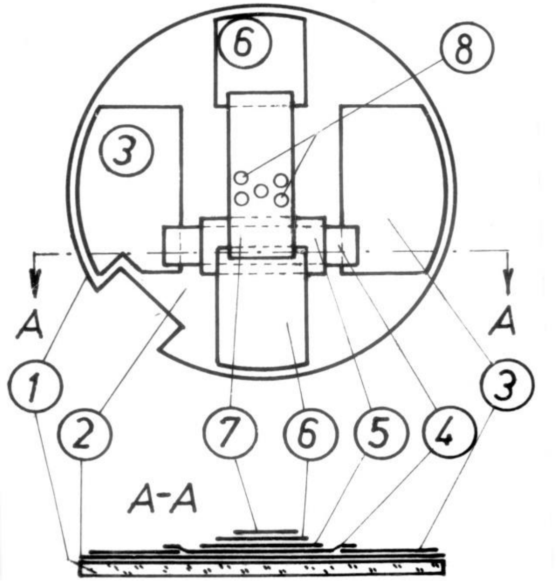
SUBSTRATES DEDICATED to ELECTRICAL MEASUREMENTS on mica microgrids

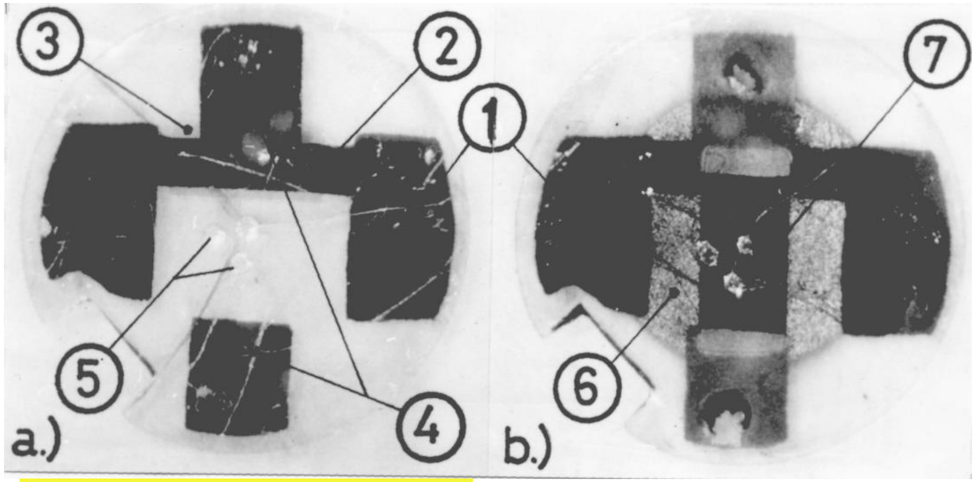


Hall voltage

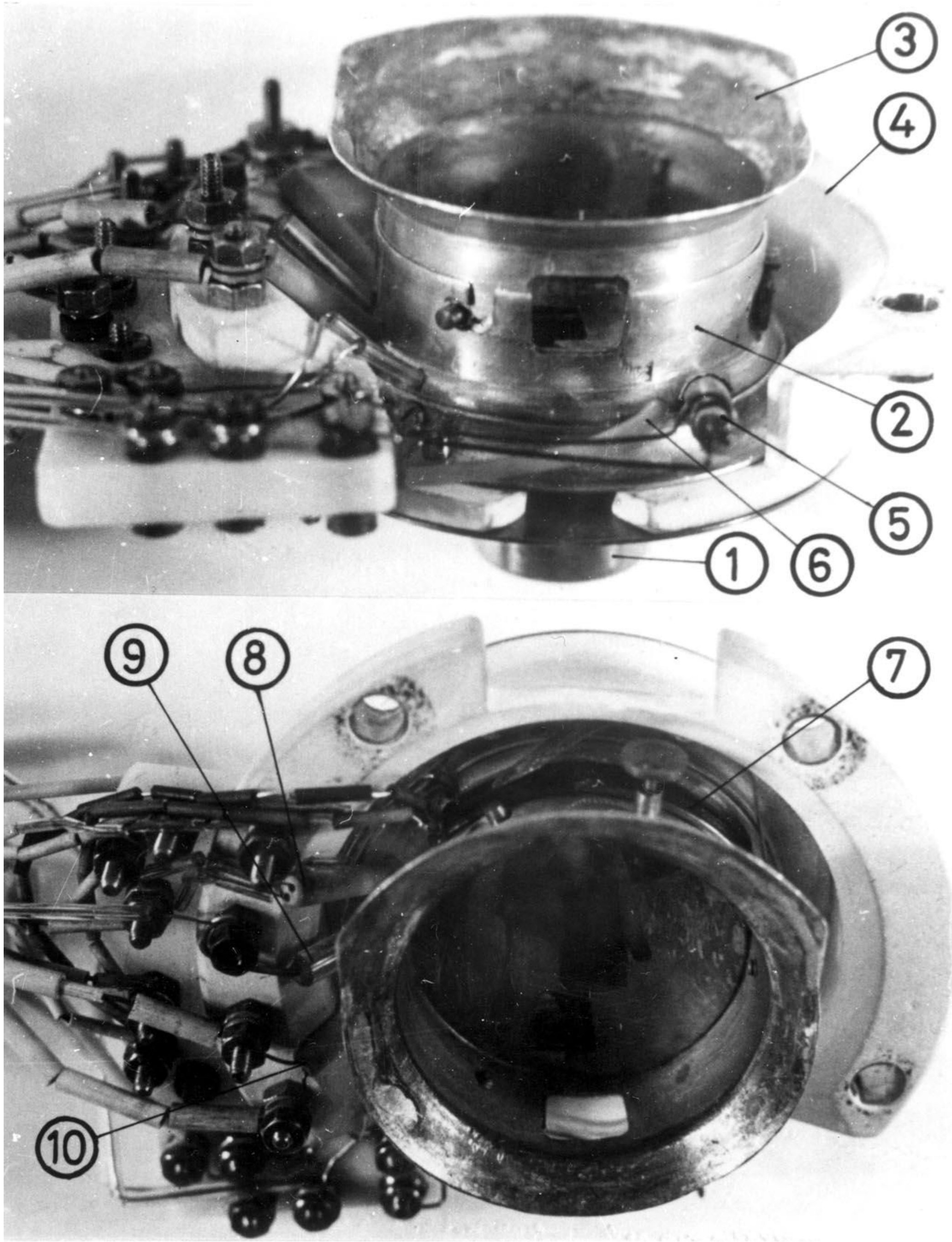


Resistivity

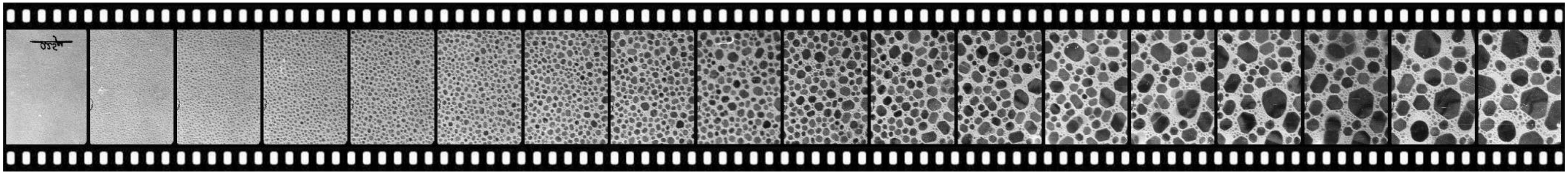




Thermo power



The cartridge of the experimental system



Members of the research team realizing the in situ TEM experiments in 1963 – 1984

J.F. Pócza (leader - 1975)

Á. Barna, P. B. Barna (leader 1975-1984), Z. Bodó, G. Sáfrán, I. Pozsgai, G. Radnóczi, L. Tóth,

Guest researchers:

Á. Csanády (Hungalu, Aluterv-FKI, Budapest)

L. Lomniczy (Res, Inst. Development of Sci. Instr. Budapest)

A. Dévényi (Institute of Physics, Bucharest)

I. Rechenberg (Humboldt Universität, Berlin),

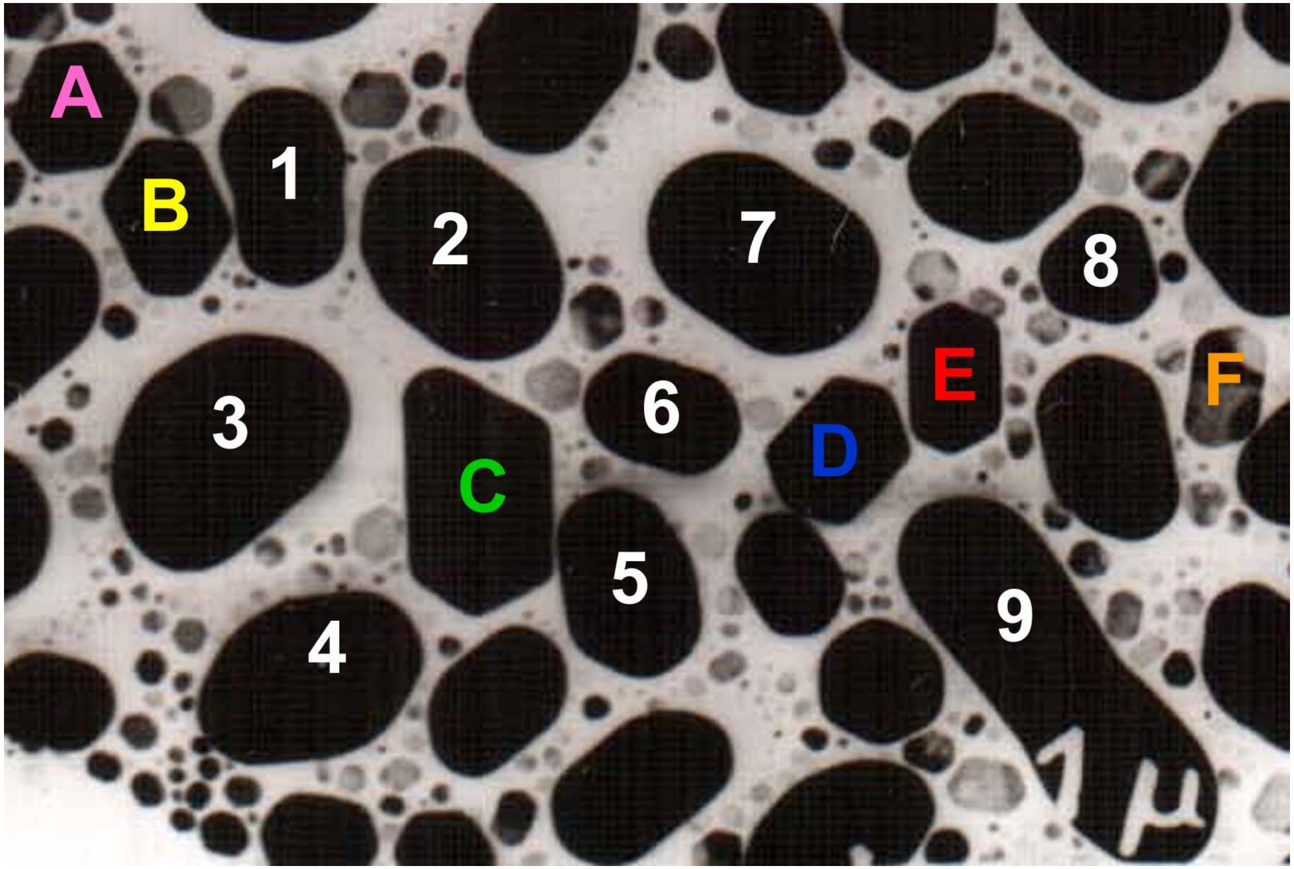
H. Sugawara (Tokyo University),

P. Thomas (Phillips Universität, Marburg)

Assistants

K. Raffay, É. Hajmássy, L. Puskás, G. Barcza, Gy. Glázer

Dumfounding structures shown up in earlyTEM studies



30 nm thick In film deposited on a-C layer substrate at 60ºC ($T_S = 0,77\ T_m$)

* COEXIST

- large and very small grains
- well faceted crystals of various sizes A, B, C, D, E, F, and more rounded ones (marked with numbers)

* ROUNDED CRYSTALS are situated in bare substrate surface domains 2, 3, 7

Stimulating question: How this unusual structure can develop?

Topics investigated by in situ UHV TEM 1963 - 1984

Indium : fundamental phenomena of structure formation

- amorphous-single crystal substrates: vapour-liquid-solid phase transformation, melting point-size dependence
- nucleation, coalescence, crystal growth, development of texture
- effect of inhibitor additive, fundamental process of the evolution of nanocomposite structure
- epitaxiaal growth on MoS_2: effect of substrate contamination

-Ge, Sb films

- evolution and restructuring of amorphous structure during heat treatment, dependence of the conduction mechanism on structure
- crystallisation of amorfous thin films during heat treatment, effect of contamination: nucleation, crystal growth, activation energy
- conduction mechanism of crystallized films, dependence of grain boundary barrier on level of contamination

NiCr resistor films

- restructuring during heat treatment: chemical reactions, formation-segregation of phases, their effects on the electrical conduction mechanisms

Oxidation of Al films

Formation of phases at sequential/multilayer deposition

- nucleation and growth of intermetallic phases at solid surface - vapour phase reaction, effect of surface contamination (sequential deposition of Al and Au)

EXPERIMENTS

Growth of In films on a-Carbon substrate

*** effect of substrate temperature**

dependence of melting temperature on crystal size

vapour-liquid-solid phase transition

coalescence of liquid droplets and crystals, texture evolution

growth of crystals

*** co-deposition of carbon: encapsulation of In crystals by a-C layer**

- Á.Barna, P.B.Barna, J.F.Pócza: Proc.Czechoslovak Summer School on Thin Films, Ledec, 1967, 71-126
- Á.Barna, P.B.Barna, J.F.Pócza: Vacuum, 17, (1967) 219-221
- J.F.Pócza: Proc. II. Coll. on Thin Films, (ED. E.Hahn, Akadémiai Kiadó, Budapest) (1967) pp.93-108
- Á. Barna, P.B. Barna, J.F. Pócza: J.Vac.Sci.Techn. 6(1969)472-474
- J.F.Pócza: Proc. Int.Conf. on Phys. and Chem. of Semiconductor Heterojunctions and Layer Structures (Akadémiai Kiadó, Budapest) (1971)Vol. III, pp.61-82
- J.F.Pócza, Á.Barna, P.B.Barna, I.Pozsgai, G.Radnóczi, Japan JAP, Suppl. 2. Part 1, (1974) 525)
- P.B. Barna:Diagnostics and Application of thin films (Ed. L.Eckertova, I.Ruzicka) Inst. of Physics Publishing, Bristol, (1992) pp. 295-309.
- P.B. Barna, M.Adamik: Science and Technology of Thin Films, eds.: F.C.Matacotta and G.Ottaviani, World Scientific Publishing Co., (1995) p. 1-28.
- P.B.Barna, M.Adamik: Protective Coatings and Thin Films: Synthesis, Characterisation and Applications, NATO ASI Series, 3. High Technology, Vol.21, (1997) (Eds.: Y.Pauleau and P.B.Barna, Kluwer Academic Publishers, Netherlands) pp.279-297.(Proc. NATO HTECH.ARW 950730, 1996, Portimao, Algarve, Portugal)
- P.B.Barna, M.Adamik Thin Solid Films 317 (1-2) (1998) pp. 27-33.
- I.Petrov, P.B.Barna, L.Hultman, J.E.Greene: J.Vac.Sci.Technol. A 21(5) (2003) S117-S128
- P.B. Barna, G. Radnóczi, Structure formation during deposition of polycrystalline metallic films, in K. Barmak&K.Coffey (Ed), Metallic films for electronic, optical and magnetic applications, Woodhead Publishing Series in Electronic and optical Materials 40(2014)67-120

Nucleation, growth and coalescence of liquid In droplets

T_S = + 90^{0}C, 0,85 T_m

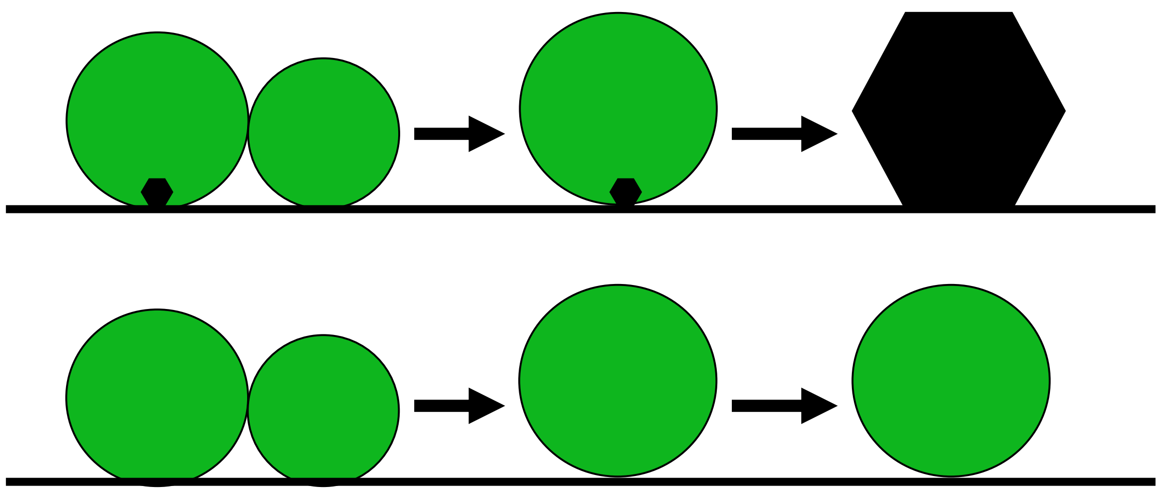
5 . 10^{-5} Pa

0,5 μm

$T_S = +80^0C$, $0{,}82\ T_m$

$P = 4\ .\ 10^{-5}\ Pa$

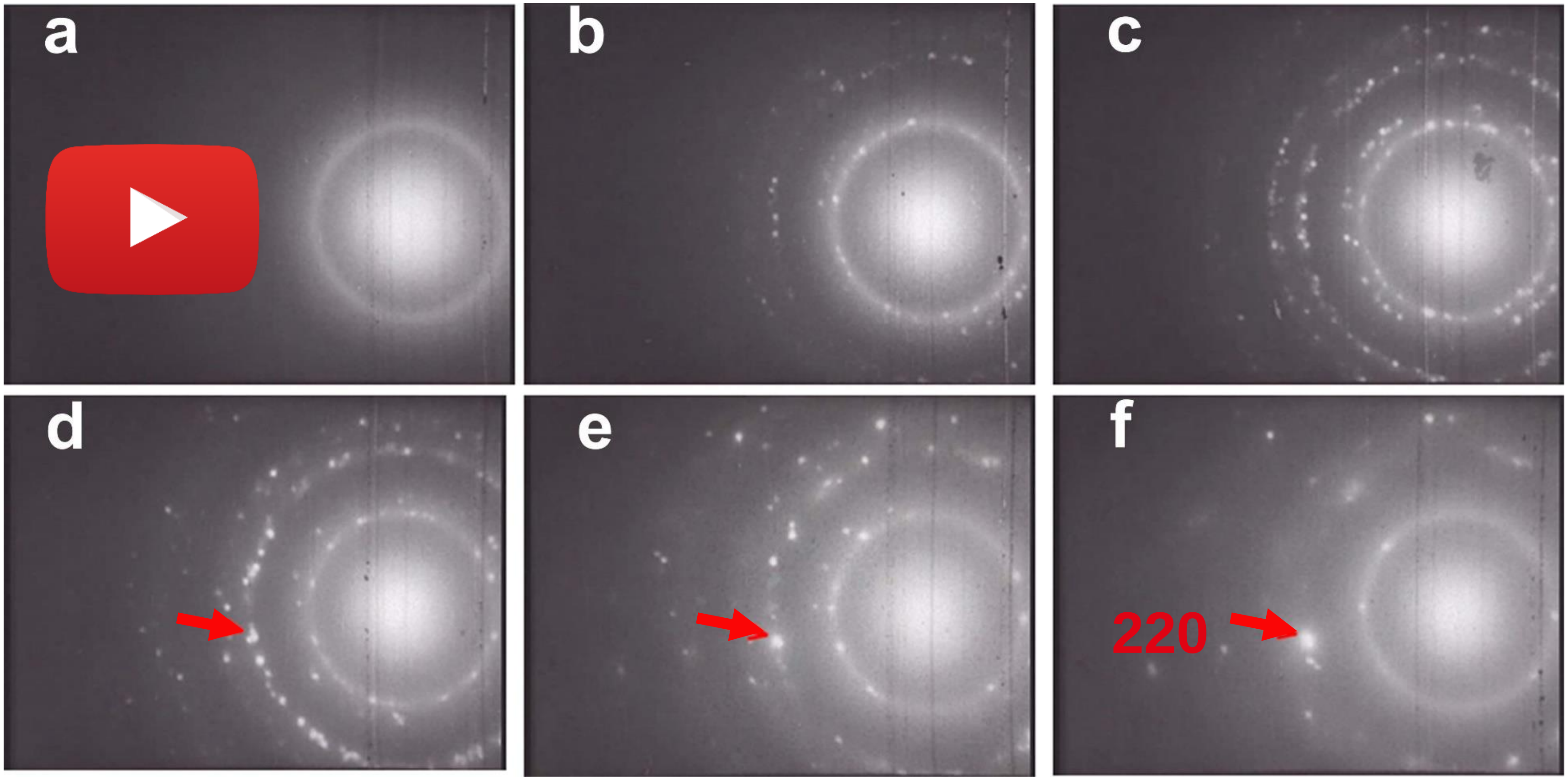
- Nucleation in liquide phase
- Growing or coalescing liquid droplets containing „active nuclei” crystallize beyond a critical size




THE FIRST CRYSTAL WILL SHOW UP

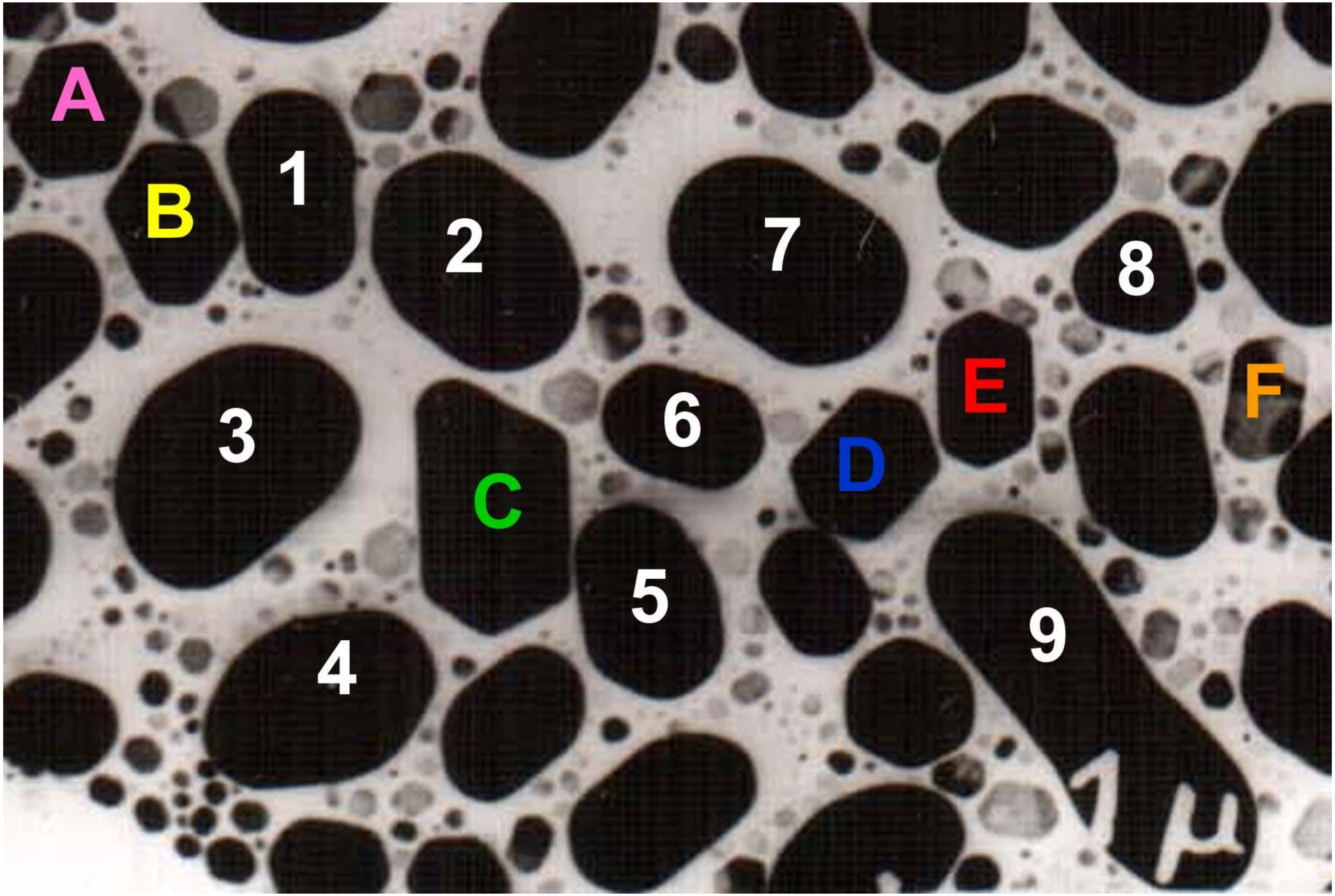
The same process is followed by selected area electron diffraction: pathway of structure evolution *development of texture by coalescence*

- nucleation of liquid droplets
- crystallization of liquid droplets: the number of randomly oriented crystalls increses,
- coalescence of liquid droplets/crystals: the material is accumulated stepvise into the crystals with the lowest interface energy and the [111] texture develops



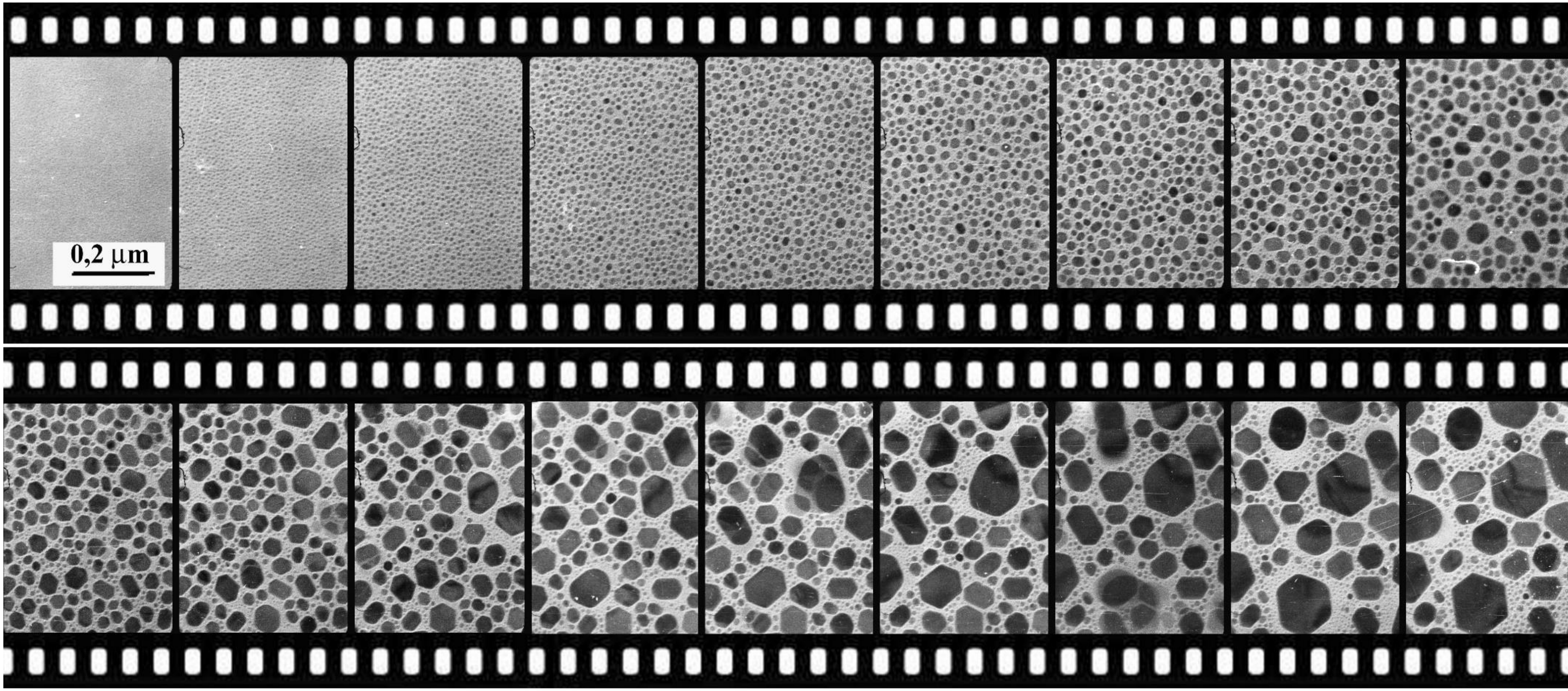
Development of the unusual structure

In deposition at 60°C (0,77 T_m) and 10^{-7}Pa, depositon rate 2 nm/s,

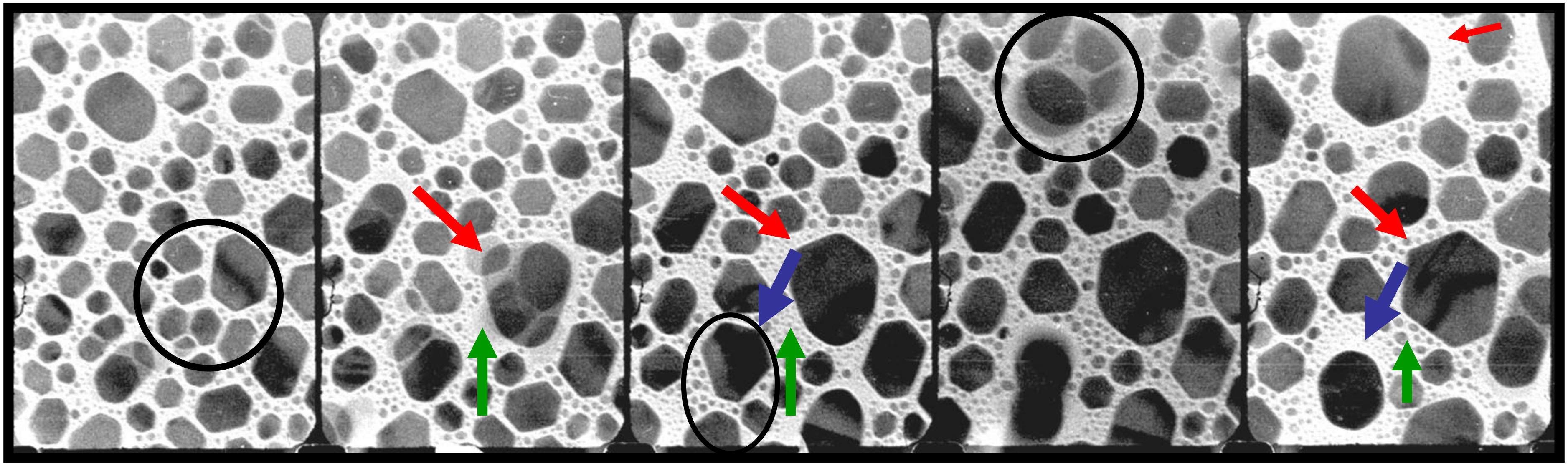


The fundamental phenomena of structure evolution:

In deposition at 60°C (0,77 T_m) and 10^{-7}Pa, depositon rate 2 nm/s,



- **nucleation: primary and secondary on the substrate**
- **crystal growth**
- **grain growth by liquid-like coalescence**



- Grain growth by liquide-like coalescence:
  * rounded crystals develop
  * bare substrate surface area
  * secondary nucleation

- Crystal growth develops faceted crystals !!!

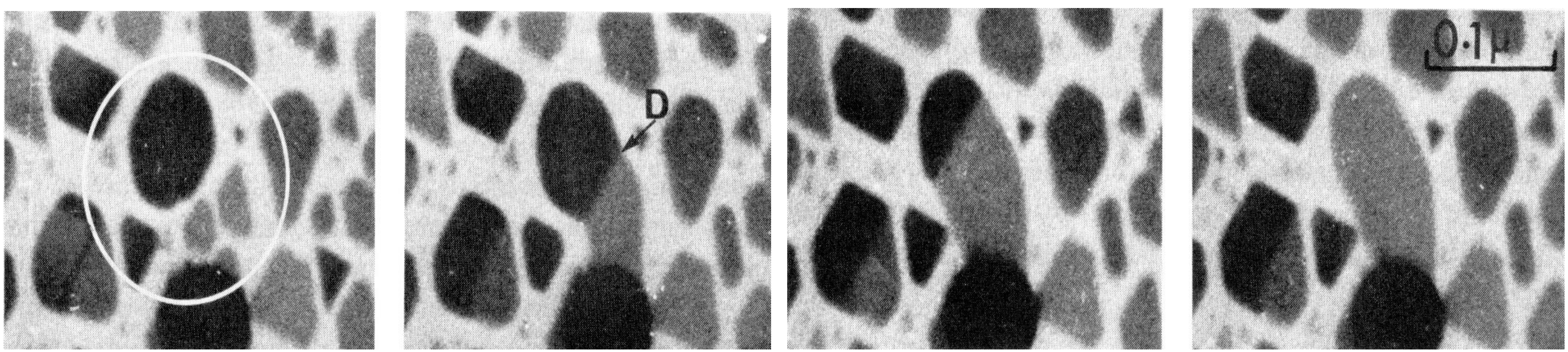
J.F.Pócza: Proc. II. Coll. on Thin Films, (ED. E.Hahn, Akadémiai Kiadó, Budapest) (1967) pp.93-108

Solid phase coalescence

Au deposition on cleaved 0002 MoS_2 surface at 670 K (T_S/T_m = 0.5)

(Stowell, M.J., in Dauglis E, Gretz R.D., Jaffee R.J. Molecular Processes on Solid Surfaces, McGraw –Hill, 1969, pp.461-477)

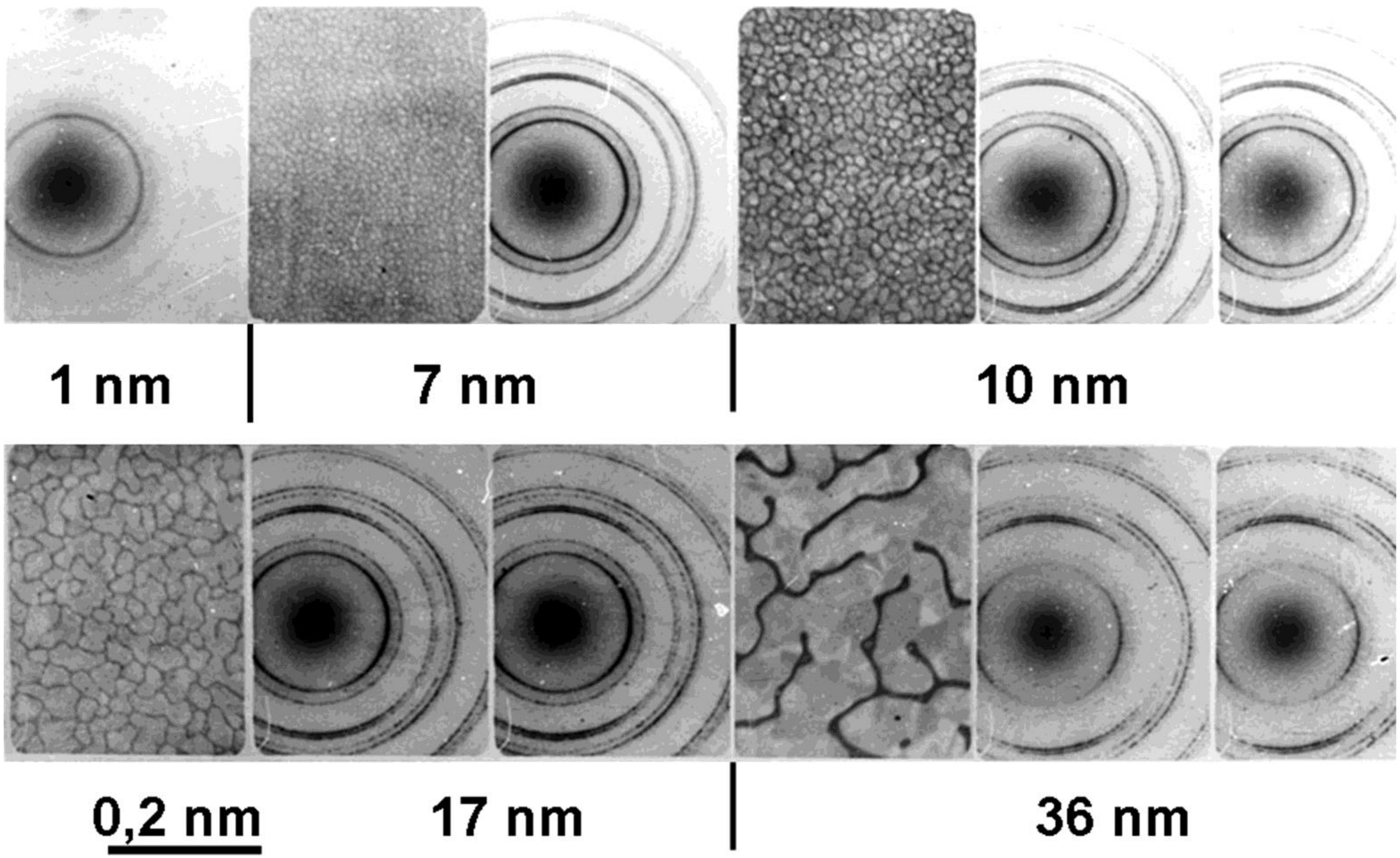
- **grain boundary migration (slow)**
- **reorientation**
- **no secondary nucleation**



Development of the [111] texture in In film during growth

T_S = - 70ºC, 0,47 T_m, 10^{-5}Pa, 0,2 nm/s

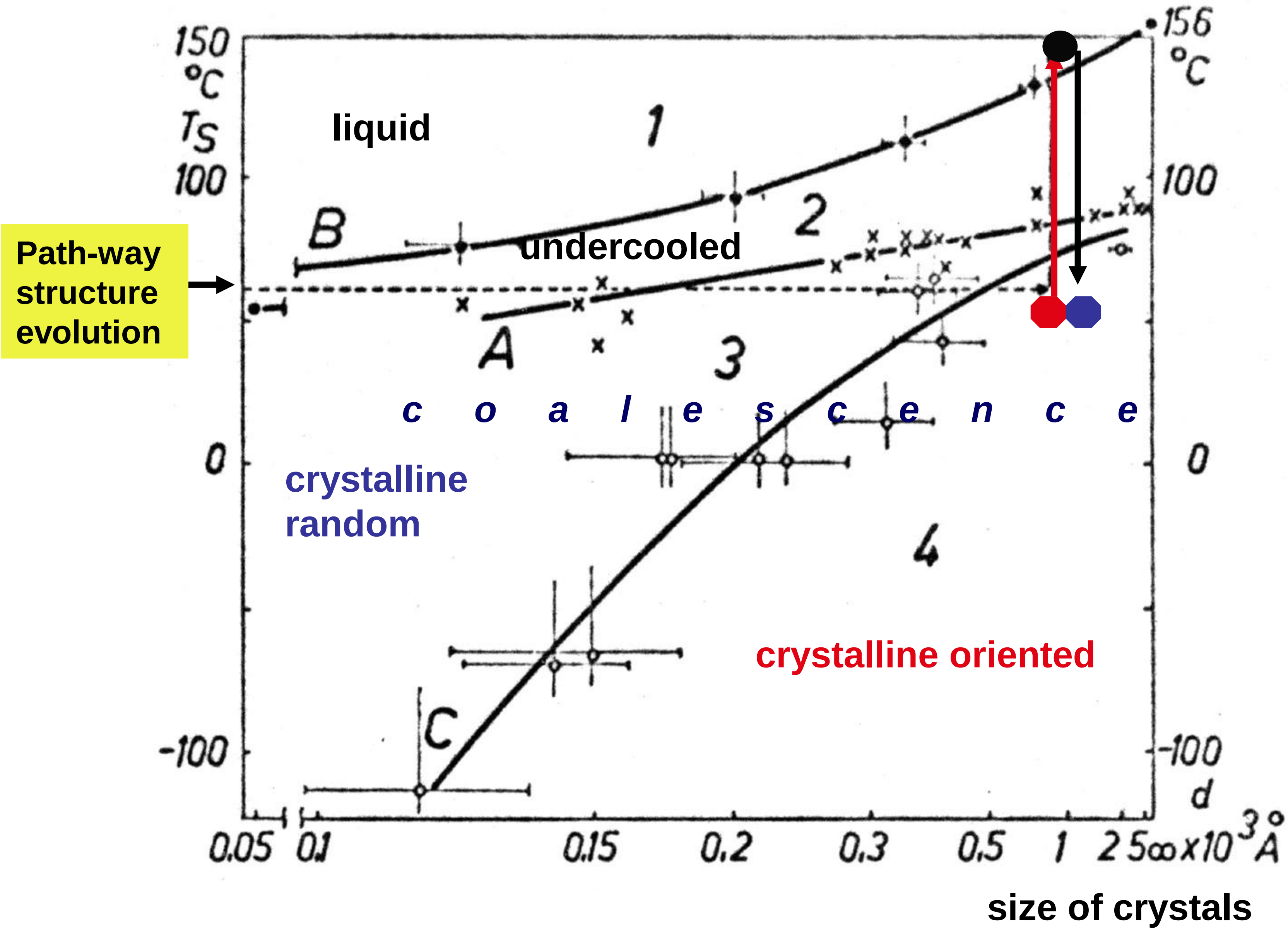
In situ experiment on tilted substrate, alternately taken images and SAED patterns



Video: T_S = -150^{0}C, 0,29 T_m

Results made possible to develop the phase diagram of In crystals and the path-way of structure evolution

Á. Barna, P.B. Barna, J.F. Pócza: J.Vac.Sci.Technol. 6(1969)472-474



CO-DEPOSITION of indium & carbon

$T_S = +75\ ^{o}C$

$P_{total} = 5x10^{-6}$ Torr without COOLING TRAP

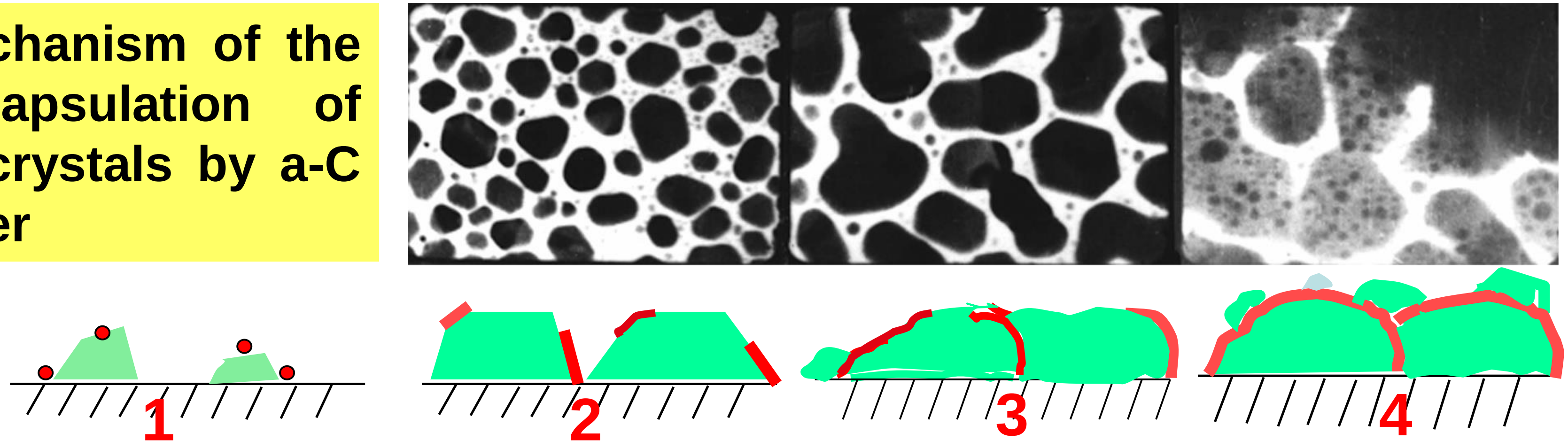
$E = 5$ Å/s

$N = 14\ 000$ x

0,5 µm

Fundamental mechanism of the nanocomposite structure evolution:
encapsulation of the In crystals by C layer developed by segregation

Mechanism of the encapsulation of In crystals by a-C layer



1 In crystals nucleate at first and grow on the substrate

-the condensing **C** species are segregated by the crystal growth processes to the surface of the In crystals

2 segregated C species nucleate an a-C phase on the In crystal's surface

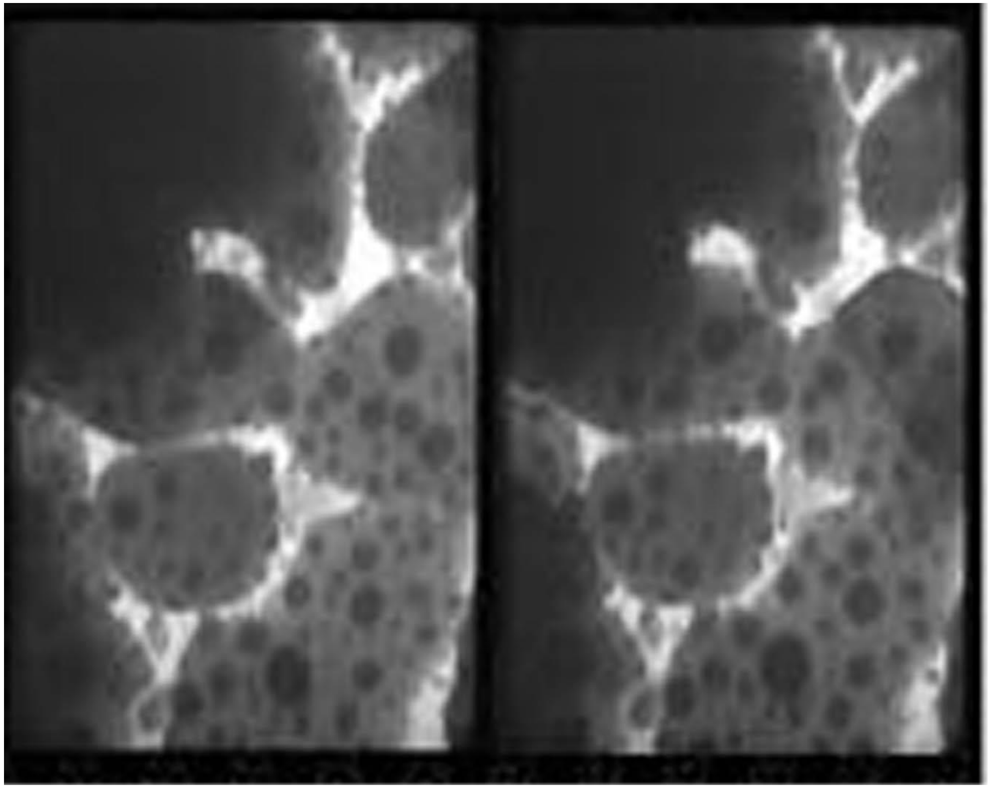
-the **C phase is growing in a 2D layer (tissue phase)** along the surface of In crystals blocking the crystal growth locally (rounding the crystal shape), **encapsulation starts**

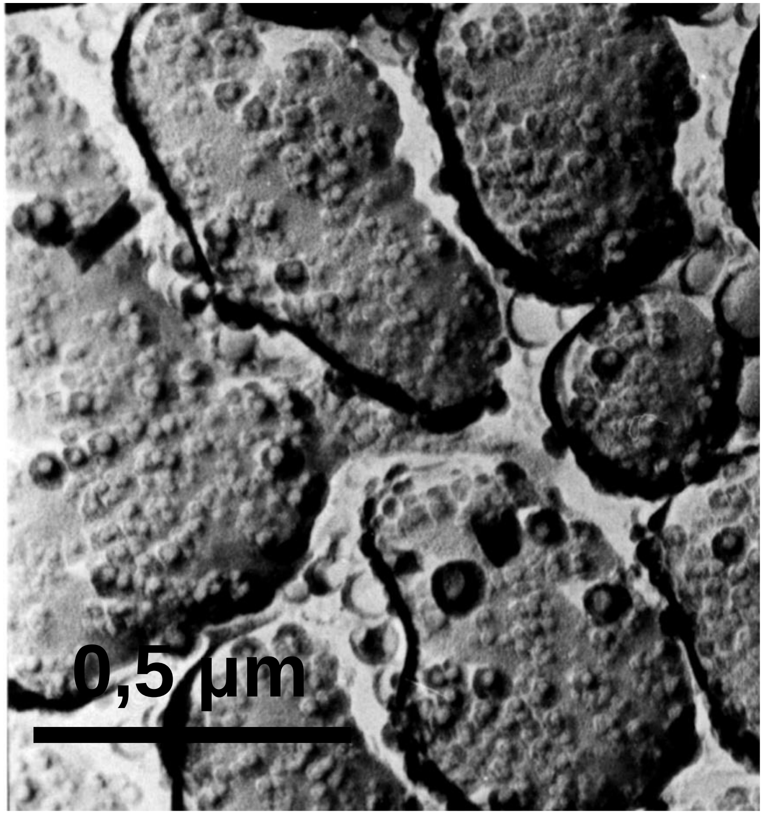
3 the **C phase incorporates into the grain boundaries** at impinging of crystals and limit the grain growth, **texture evolution is limited**

4 when the encapsulation of In crystals by the C layer is completed, the condensation of **In** results in **repeated nucleation**

- The whole growth process is continued

Crystals developed by repeated nucleation on the surface of In crystals encapsulated by a-C layer



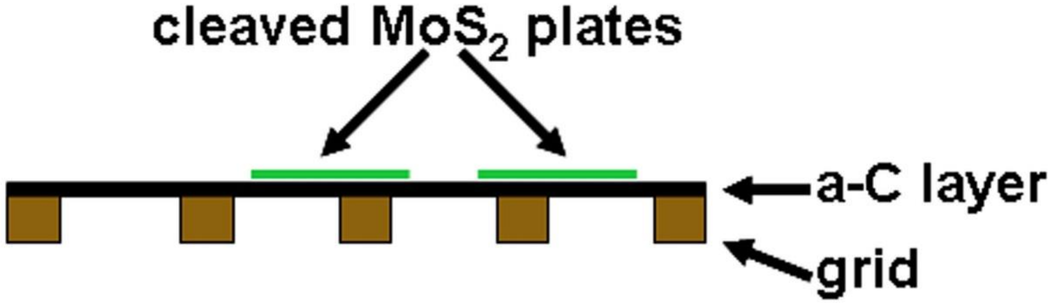


Deposition of In on air cleaved {0001} surface of MoS_2 crystals

a-C or SiO_2 layer substrates were prepared on TEM micrograms

The metall microgrid

Substrate for polycrystalline film growth and texture evolution onamorphous substrates



Substrate for epitaxial growth experiements

Epitaxial growth of In film on air cleaved 0001 MoS_2 surface

effect of substrate surface contamination

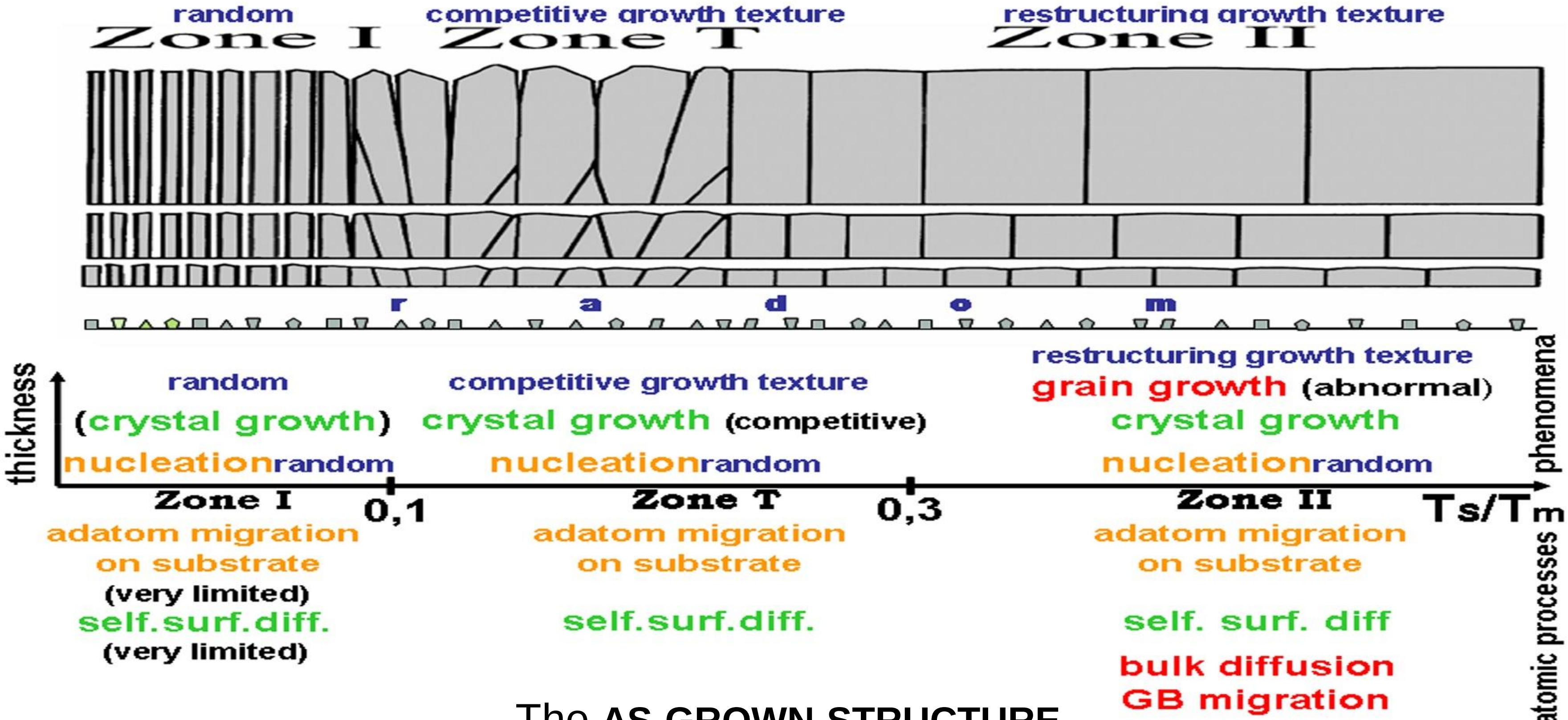
T_S = 100°C, 0,86T_m

2 . 10^{-6}Pa

0,3 nm/s

0,2 µm

The STRUCTURE ZONE MODEL of elemental thin films growing on amorphous substrate



The **AS-GROWN STRUCTURE**

fiber-like, porous, random
cross section:
honogenous

columnar, texture
cross section:
grain size&texture varies along thicknes
V-shaped columns

columnar, texture
cross section:
grain size&texture homogenous
GB-s: perpendicular to the film plane

I.Petrov, P.B.Barna, L.Hultman, J.E.Greene: *J.Vac.Sci.Technol. A 21(5) (2003) S117*

P.B.Barna, G. Radnóczi, *Structure formation during deposition of polycrastalline metallic fiulms, in K. Barmak&K. Coffey (Ed), Metallic films for electronic, optical and magnetic properties, Woodhead Publishing Series in Electronic and Optical Materials, Nr. 40 (2014)67-120*

We are grateful and indepted to late

Professor J. F. Pócza,

who was our mentor, who initiated and supervised the systematic study of the structure evolution in thin films by proposing the application of in situ UHV transmission electron microscopy experiments and by introducing a new synthetic view for the evaluation and interpretation of the results in their complexity.

Publications discussing results of in situ UHV transmission electron microscopic thin film experiments

Department of Thin Films, Research Institute for Technical Physics and Materials Science, Centre for Energy Research, Hungarian Academy of Sciences,
1525 Budapest, P.O.Box 49, Hungary

E.F.Pócza, Á.Barna, P.Barna: Nucleation and growth processes in vacuum deposited Ge films,
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