INTERNATIONAL ATOMIC ENERGY AGENCY
UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION

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EXPERIMENTAL WORKSHOP ON HIGH TEMPERATURE SUPERCONDUCTORS & RELATED MATERIALS (BASIC ACTIVITIES)

12 - 30 MARCH 1990

THIN FILMS

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Thin Films

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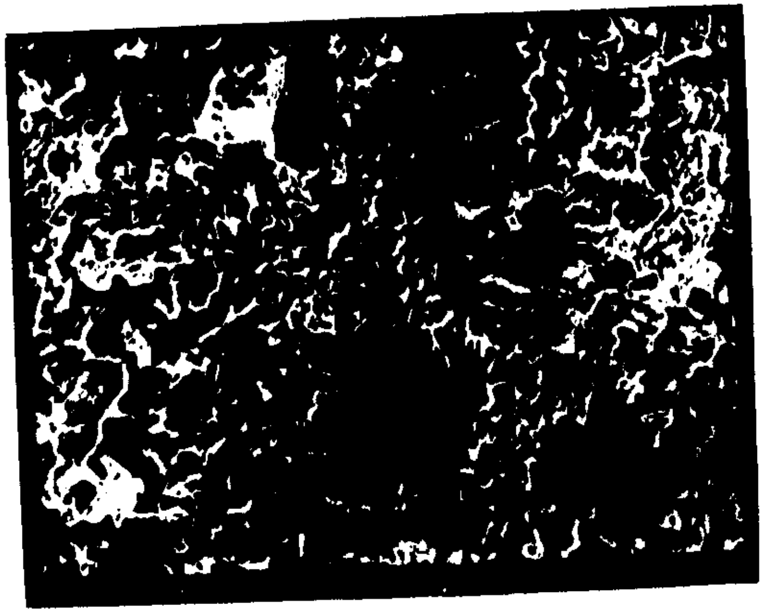


General problems:

- strong anisotropy of electrical parameters
- grain boundaries (weak links)

Dimos - experiment

→ bulk material: poor critical current density
($\approx 10^3$ A/cm² at 77K and B = 0T)

textured material: up to $2 \cdot 10^4$ A/cm²

single crystals or epitaxial thin films

↑

technical choice

D. Dimos, P. Chaudhari, J. Mannhart, and F.K. LeGoues
Phys. Rev. Lett. 61, 219 (1988)

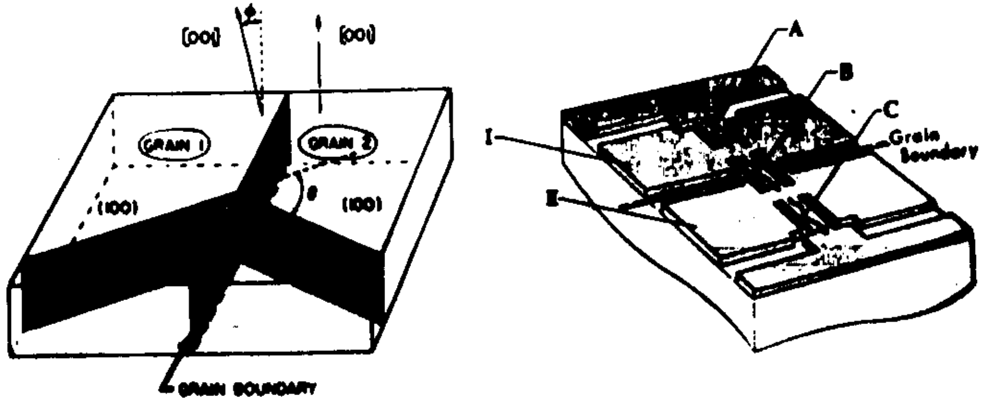
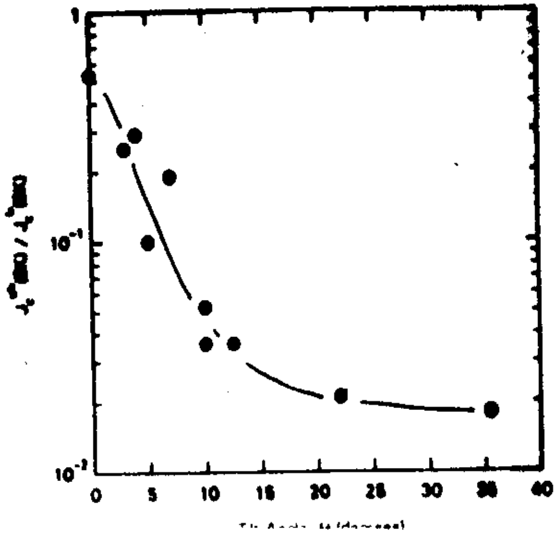
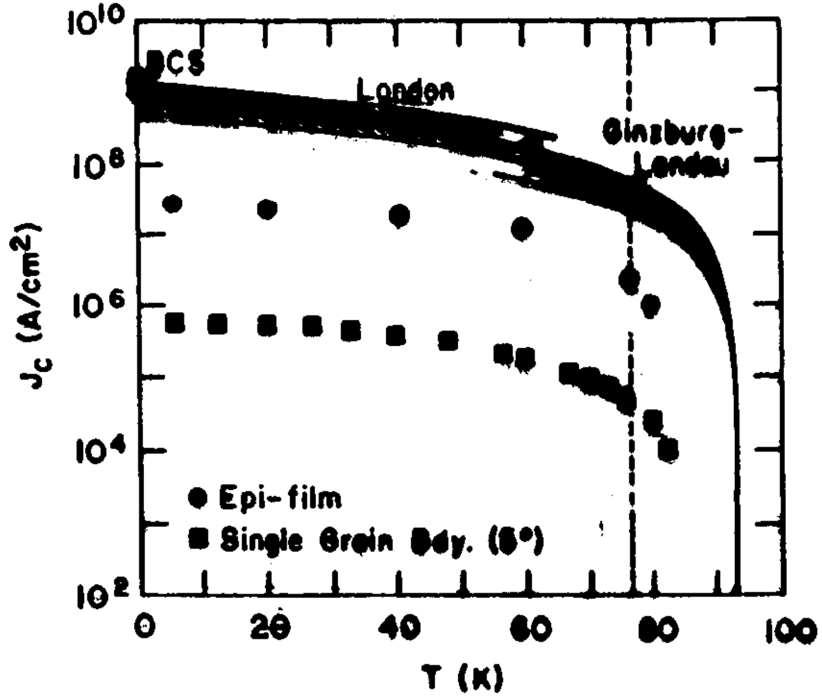


TABLE I. Critical current density (10^3 A/cm²) at 4.2–5 K.

θ (φ)	Grain 1	Grain 2	Grain Boundary
0° (3°)	7140	8000	4000
3° (7°)	3900	5300	1400
4° (6°)	270	220	73
5° (9°)	6800	5700	560
7° (6°)	140	180	40
10° (6°)	7000	8000	410
10° (6°)	7000	6100	240
12.5° (2°)	3000	3400	160
22° (2°)	800	200	11
35.5° (4°)	1350	1400	25





BCS
London
Ginzburg-Landau
● Epi-film
■ Single Grain Bdy. (5°)
J_c (A/cm^2)
T (K)

...ation Methods:

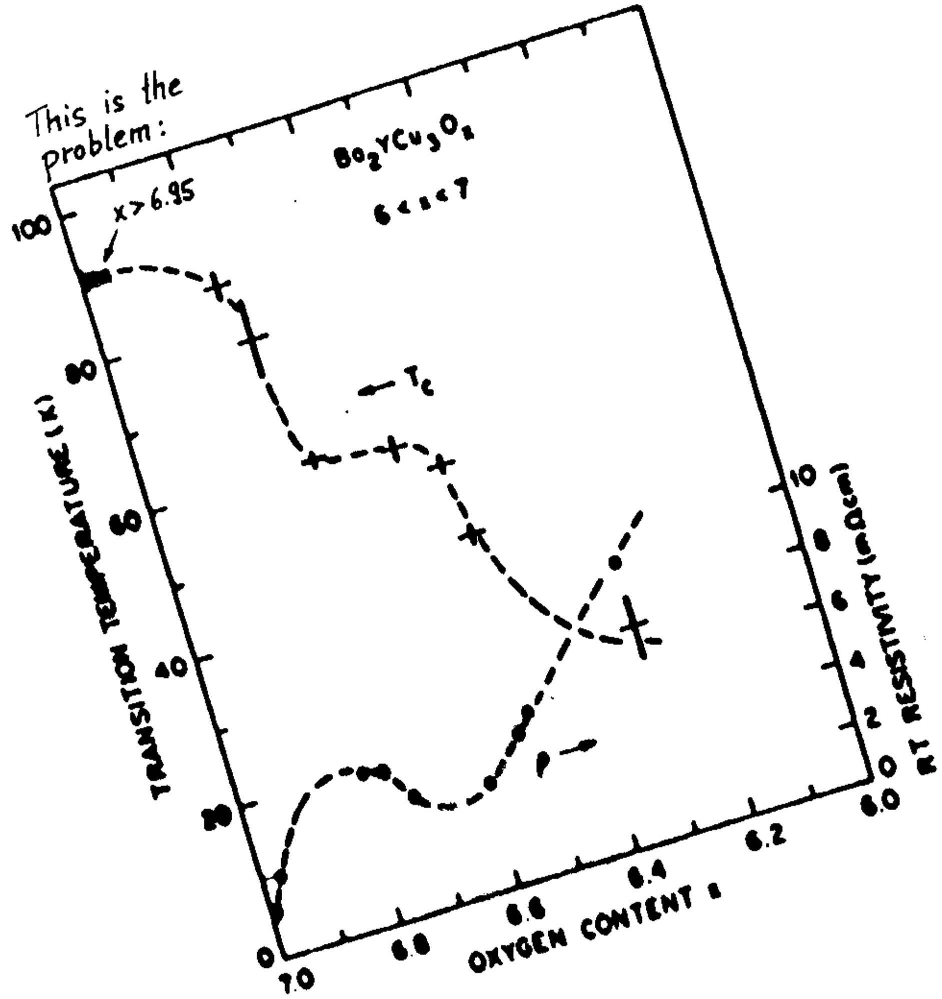
- rf/dc- sputtering
- electron beam evaporation
  Molecular beam epitaxy (MBE)
- laser ablation
- chemical vapour deposition (CVD)
- and other methods ...

In principle not possible to grow $YBa_2Cu_3O_{6+x}$, but only $YBa_2Cu_3O_{7.0}$,

nearly all thin film ...ation methods

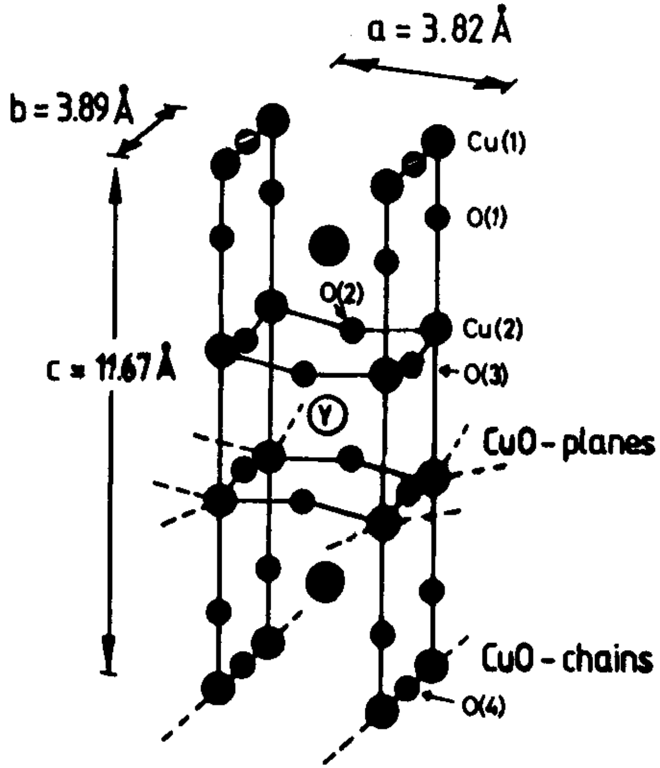
tetragonal phase, ...ial growth on ...trates with ...

This is the problem:



3-step process:

- deposition of the elements
  (amorphous film)
- crystallization to tetragonal structure
  (ex situ, T>900°C in 1 bar O_2)
- phase transformation
  tetragonal → orthorhombic
  (slow cooling in 1 bar O_2)

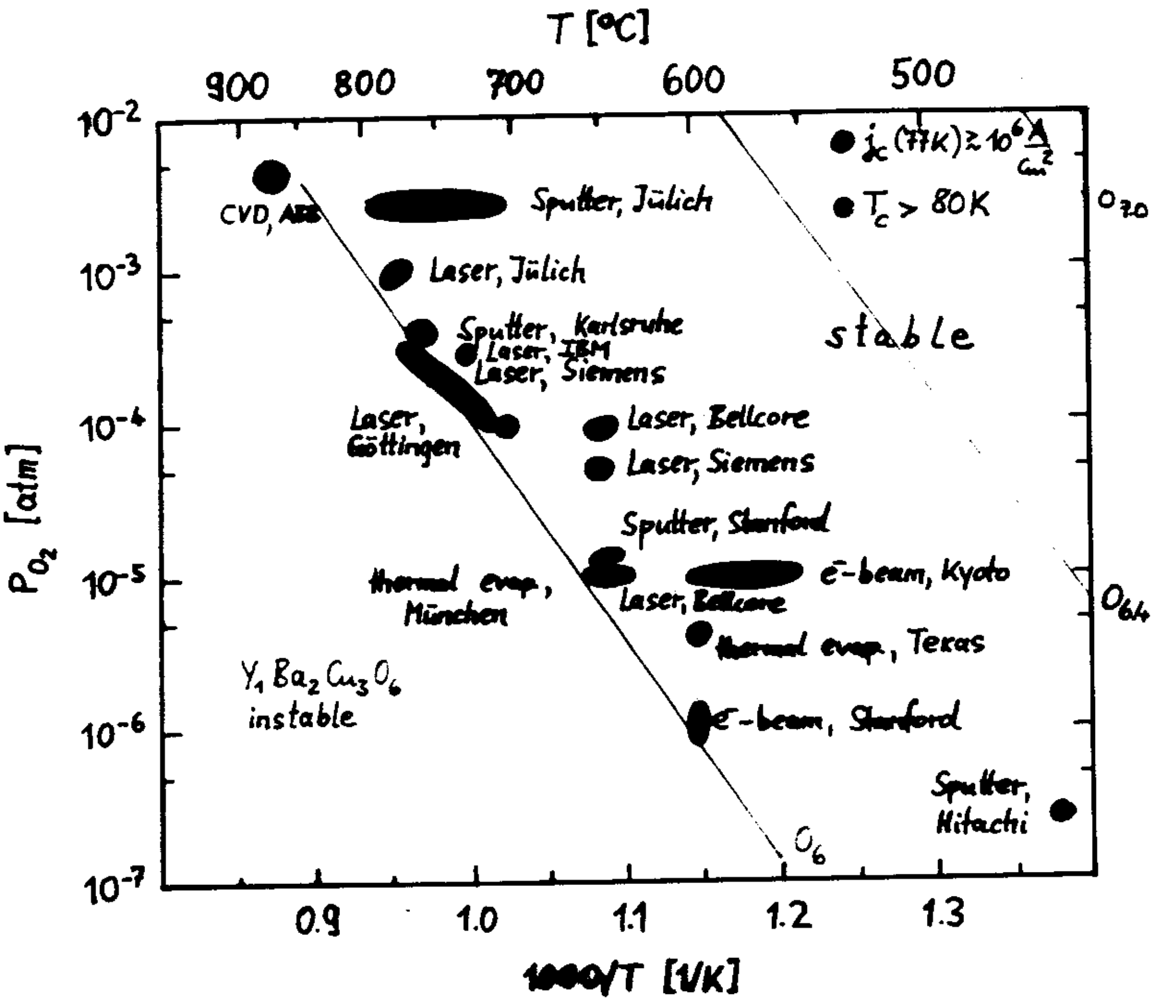


$Y_1Ba_2Cu_3O_{7-\delta}$

2-step process: (better way)

- deposition at 600°C - 750°C in
  oxygen partial pressure
  (epitaxial growth of tetragonal phase)
- phase transformation
  tetragonal → orthorhombic
  (slow cooling in 1 bar O_2)

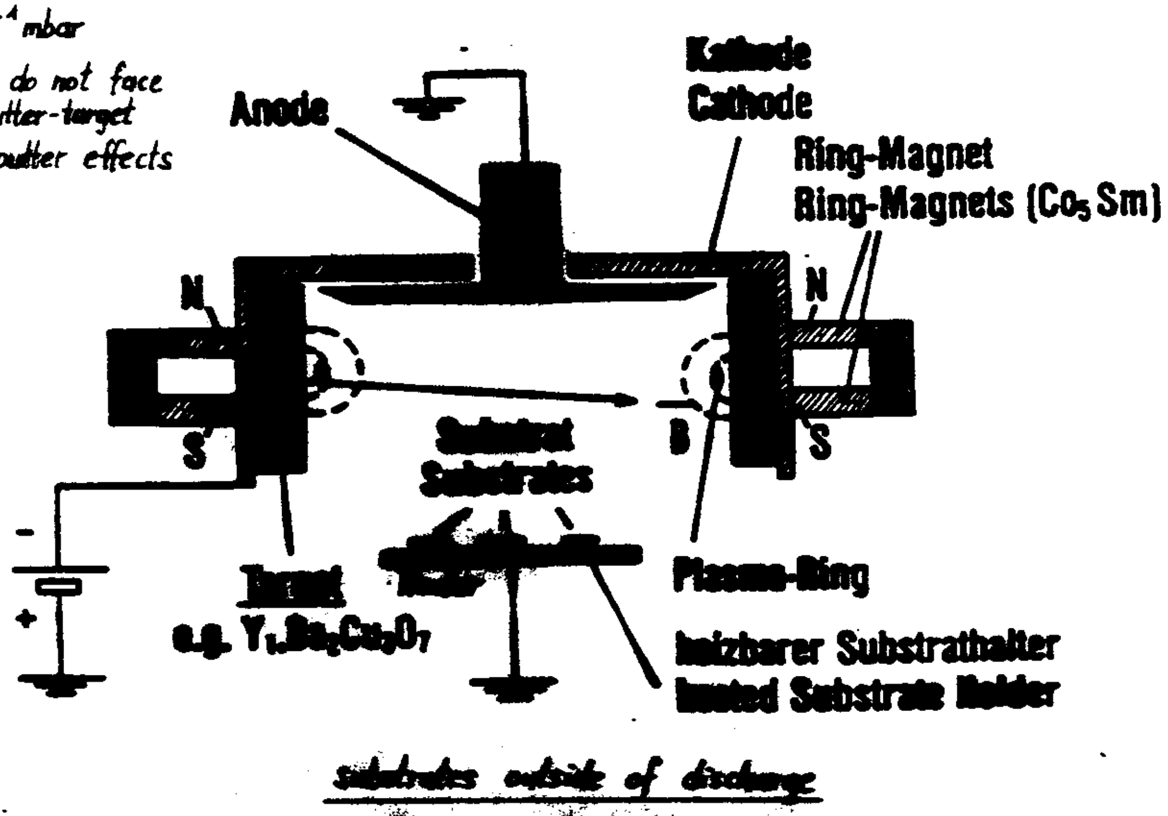
All methods work with the 2-step process.



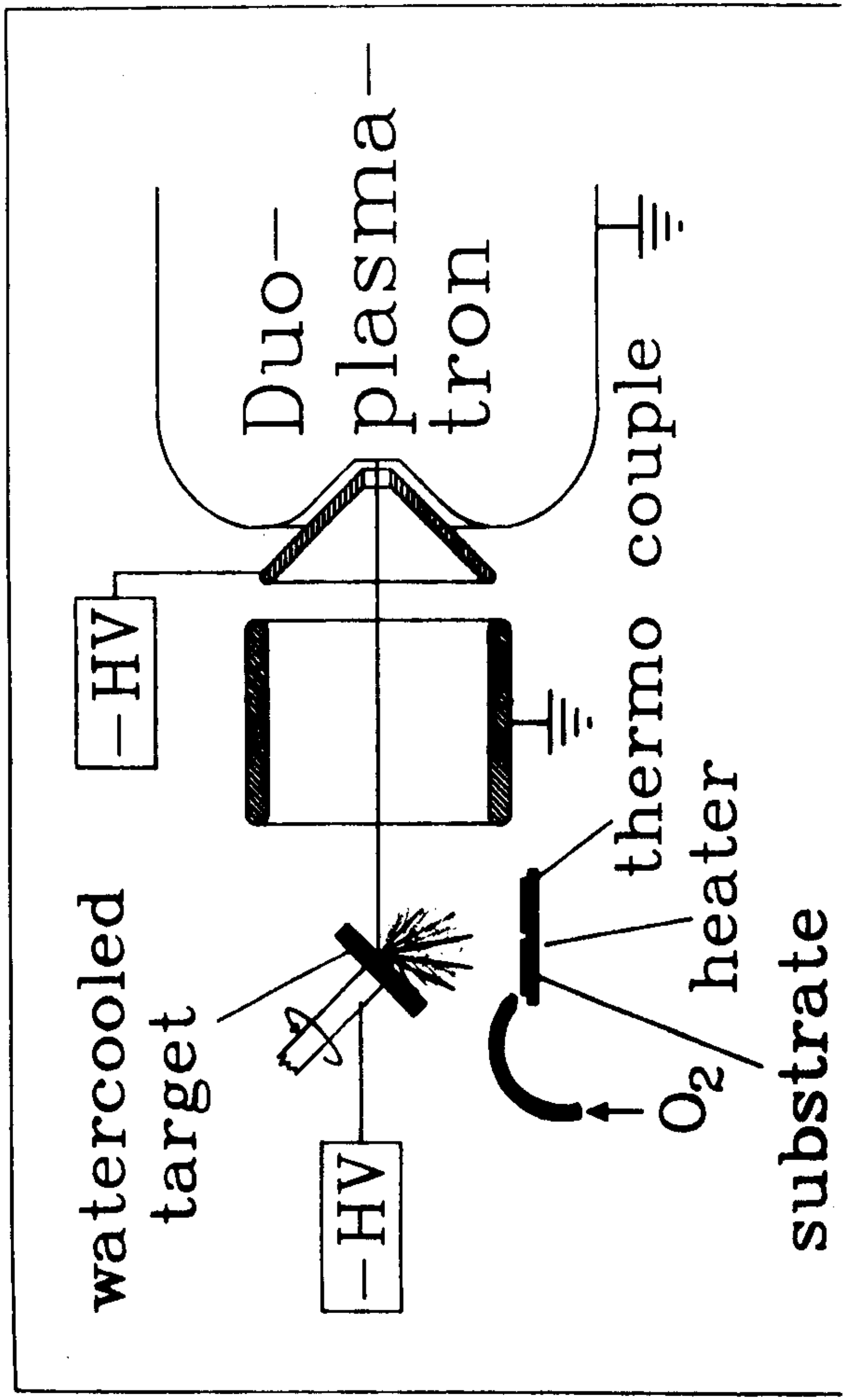
DC-Sputtering

for example: J. Geerk, G. Linker, O. Meyer
Kernforschungszentrum Karlsruhe

Composite target
DC-voltage 120 - 480V
$P_{O_2} \lessapprox 5 \cdot 10^{-4}$ mbar
substrates do not face the sputter-target
→ no re-sputter effects

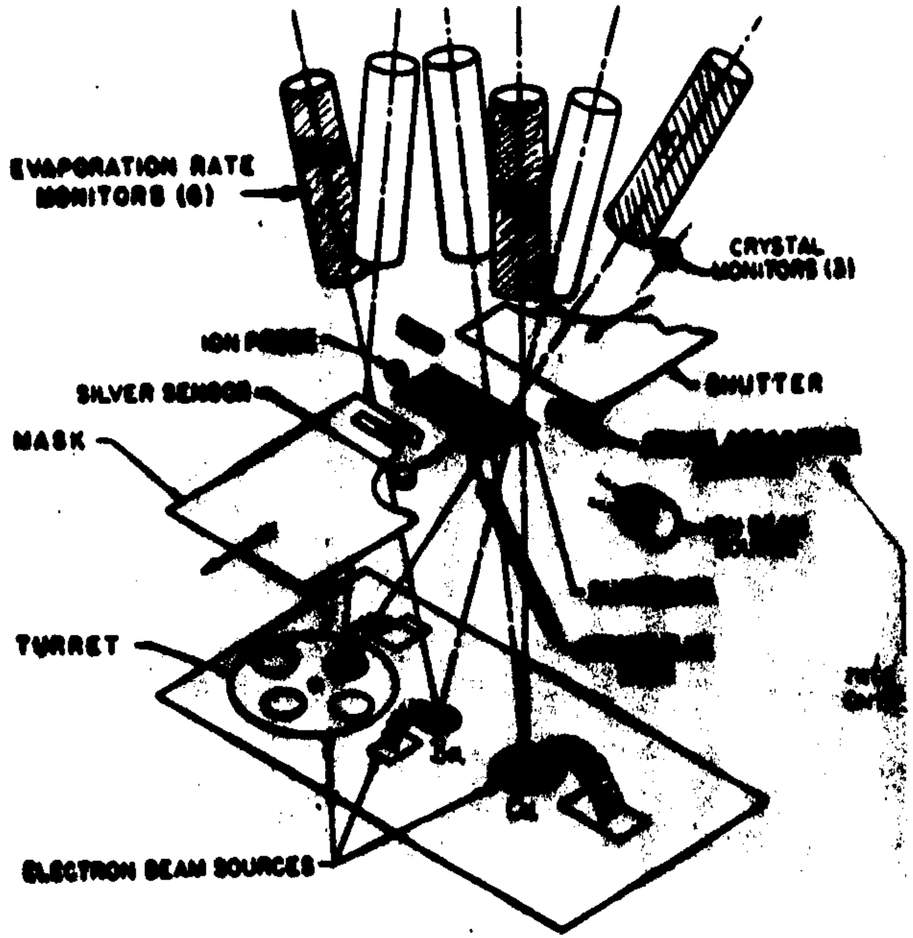


substrates outside of discharge



Electron Beam Coevaporation

for ex.: N. Missert, R. Hammond, J.E. Mooij, V. Matijasevic, P. Rosenthal, T.H. Geballe, A. Kapitulnik, M.R. Beasley
Stanford University



$p_{O_2} \leq 2 \cdot 10^{-4}$ mbar

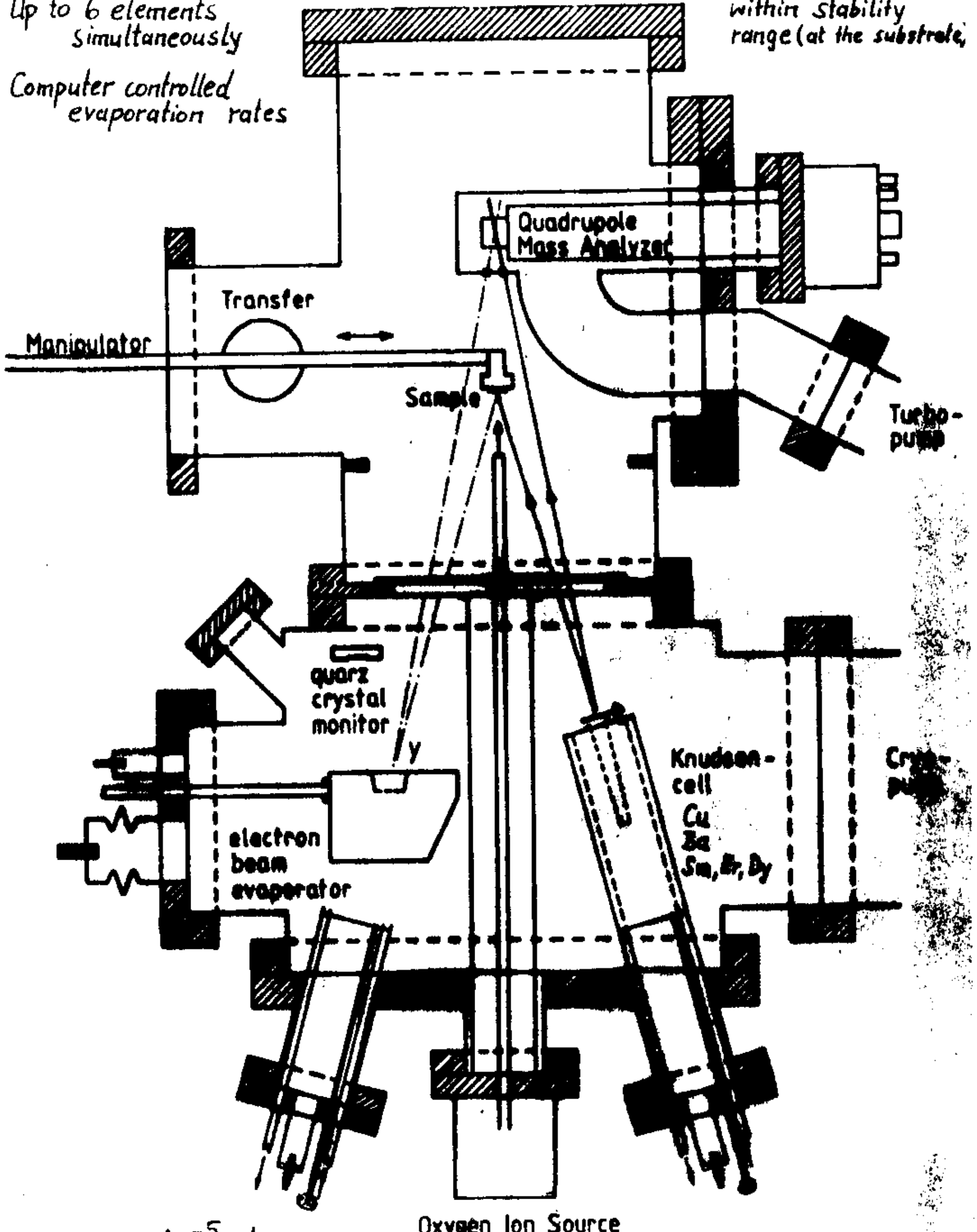
MBE - CHAMBER

Base pressure ≤ 10^{-10} mbar

Up to 6 elements simultaneously

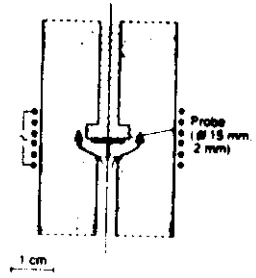
Computer controlled evaporation rates

oxygen pressure during preparation within stability range (at the substrate)

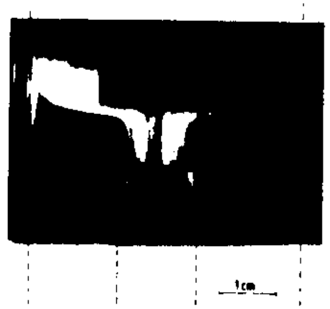


$P_{O_2} \leq 10^{-5}$ mbar

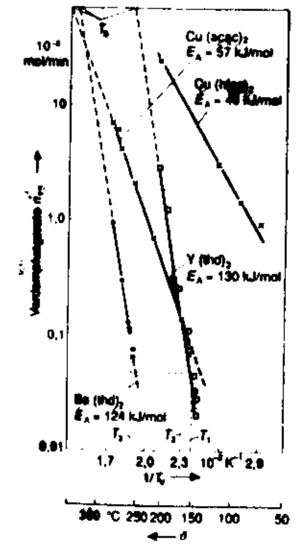
Chemische Abscheidung von $YBa_2Cu_3O_{7-\delta}$ aus der Gasphase



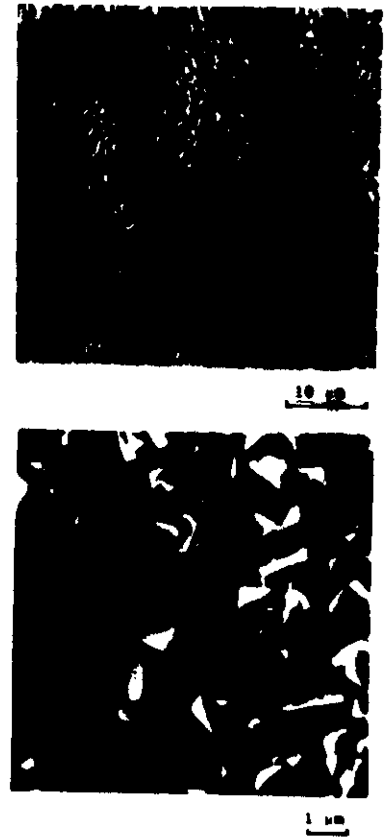
[redacted]



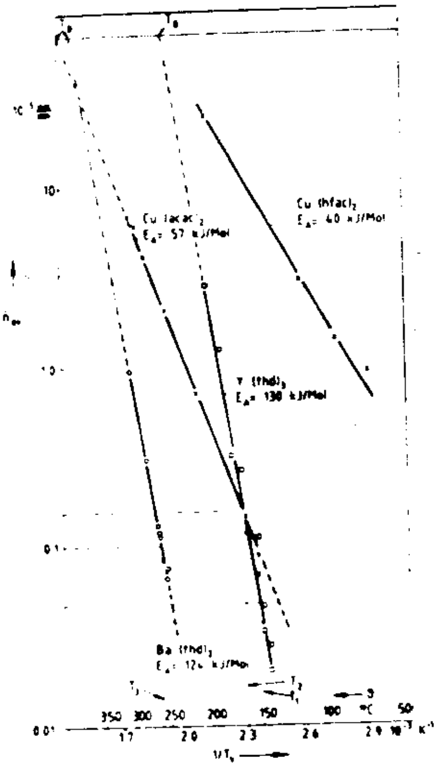
Gasfluß im CVD-Reaktor (70 l/h)



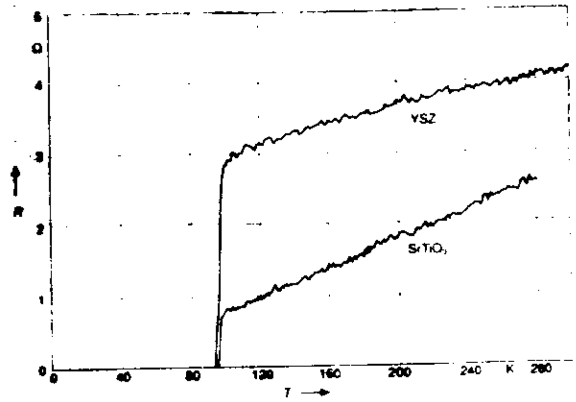
Verdampfungsraten der für die Abscheidung verwendeten Ausgangsmaterialien

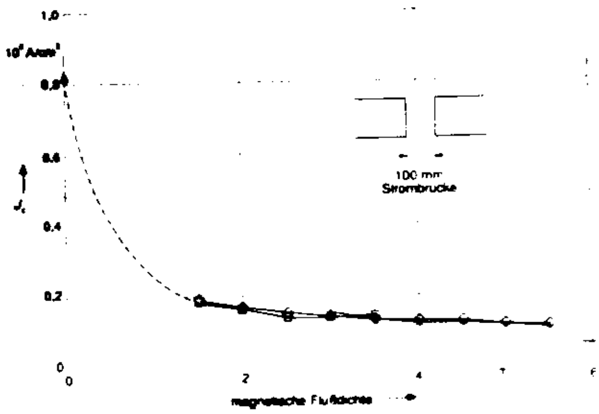


SEM-pictures of $YBa_2Cu_3O_{7-\delta}$ on YSZ-substrates

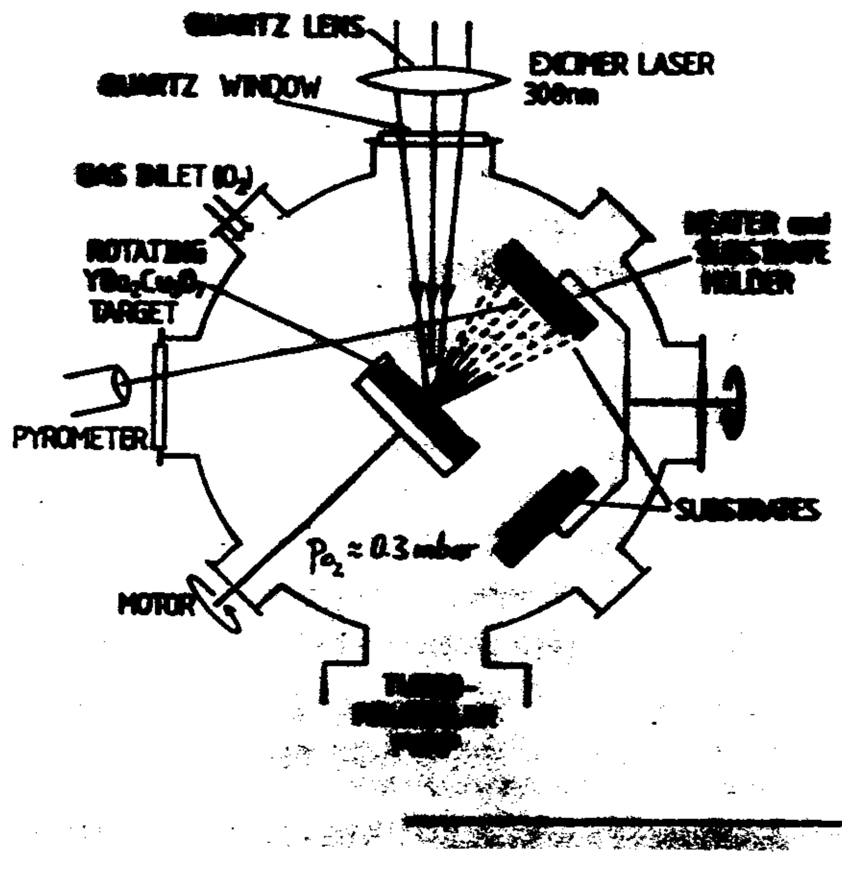


Molar evaporation rates $\dot{n}_{ev}$ of Cu(acac)$_2$, Cu(hfac)$_2$, Ba(thd)$_2$ and Y(thd)3 versus the reciprocal, absolute evaporation temperature T_v. ($\dot{V}_{Ar}$ = 25 l/h (STP), P_{tot} = 500 Pa, T_1,T_2 and T_3 indicates the evaporation temperatures for Y(thd)$_3$, Ba(thd)$_2$ and Cu(acac)$_2$ used for the deposition experiments).





for example: B. Roas Universität Erlangen
L. Schultz Siemens AG, Erlangen
Appl. Phys. Lett. 53, 1557 (1988)



LASER Siemens XP 2020 XeCl

- wavelength: 308nm
- pulse: 1 - 2 Joule/30 ns
- repetition rate 5Hz

TARGET

- sintered $YBa_2Cu_3O_{7-x}$ - pellet
- energy density: 4 Joule/cm^2

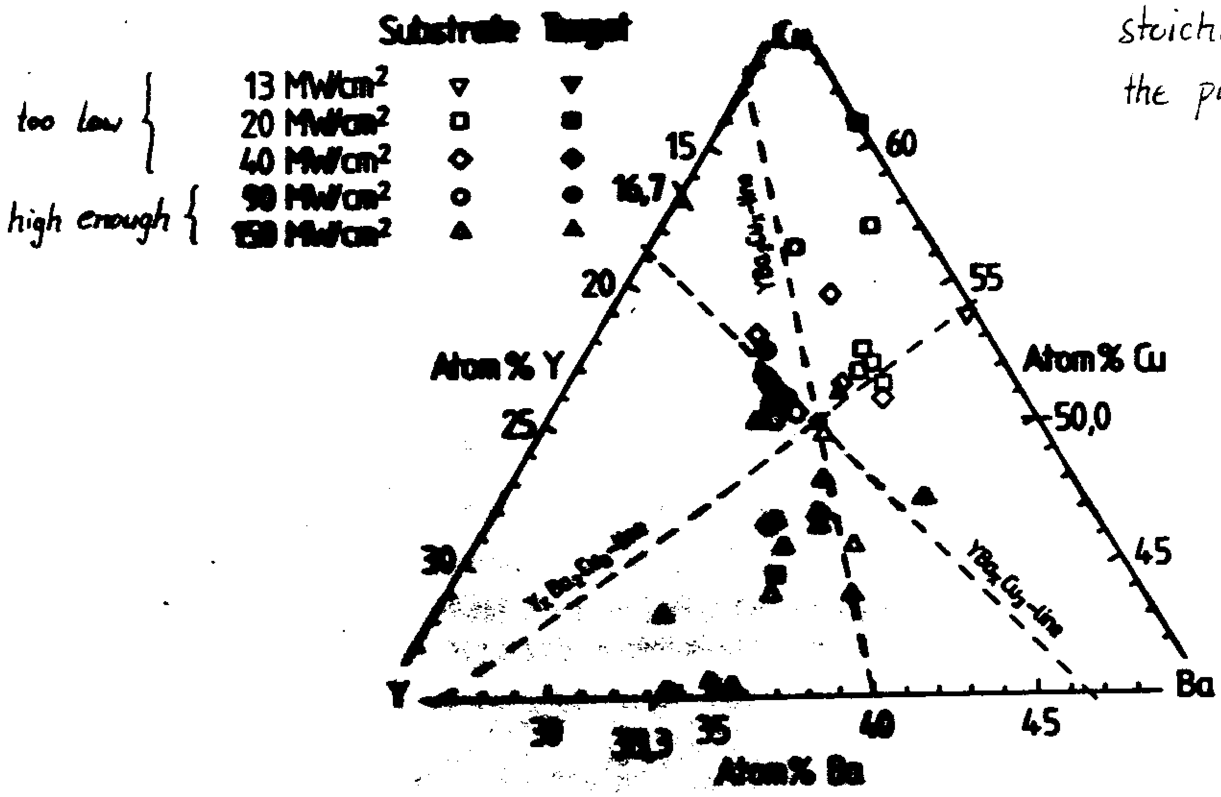
FILM GROWTH CONDITIONS

- substrate temperature: 700°C - 800°C
- oxygen pressure: 0.2 mbar - 0.5 mbar
- single crystalline substrates ($SrTiO_3$, MgO, $LaGaO_3$)
- cooling in 1 bar O_2

advantages:
- short preparation times
- high oxygen pressure possib.
- conservation of stoichiometr.

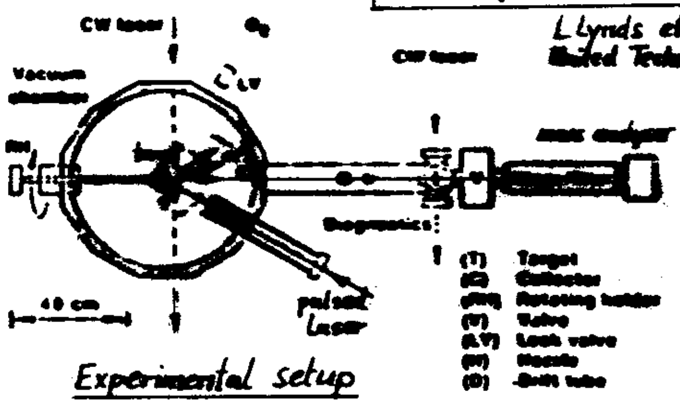
disadvantages:
- surface roughness
- particles on film surface
- small range of homogeneous growth rates

Dependence of stoichiometry on the pulse power:

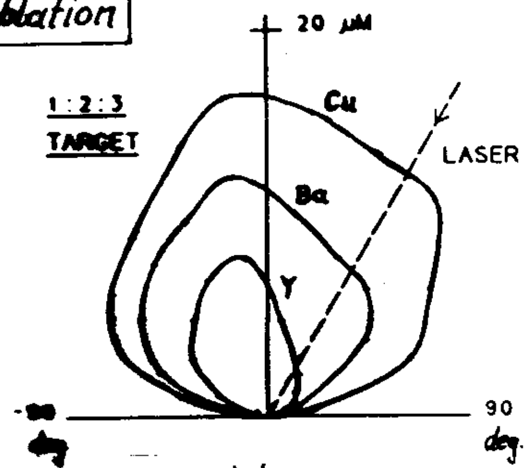


The Physics of Pulsed Laser Ablation

L. Lynds et al.
United Technologies Research Centre

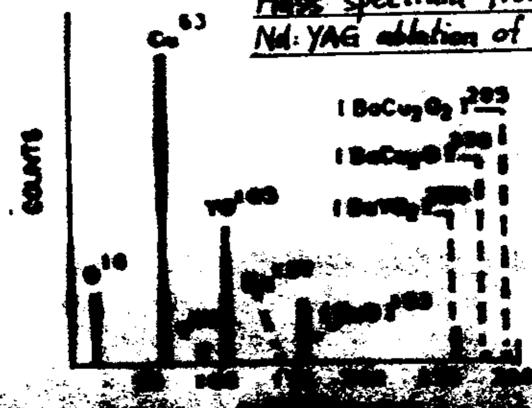


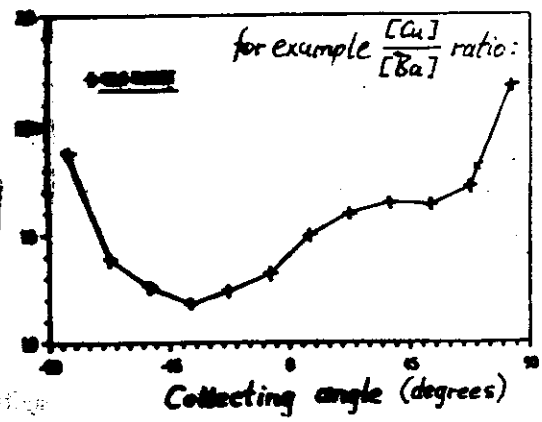
Experimental setup

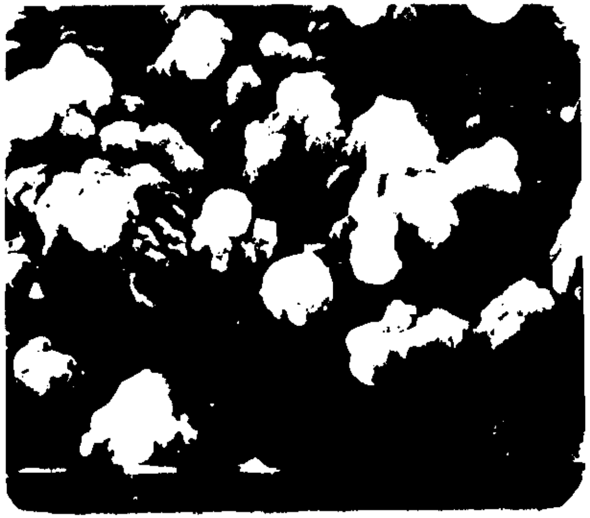


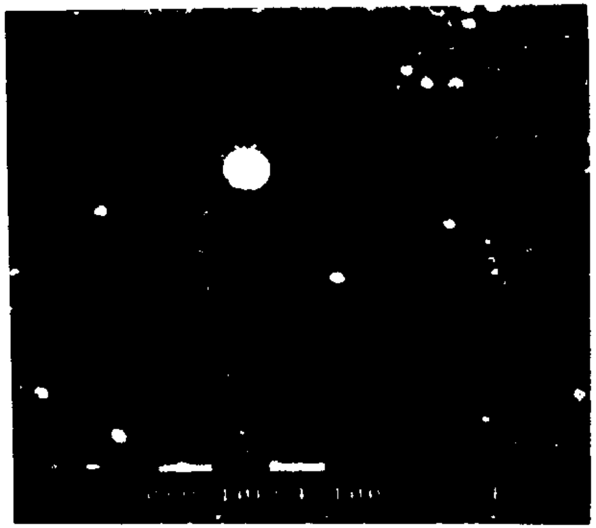
Elemental mass distributions

Mass spectrum from Nd:YAG ablation of 123-target

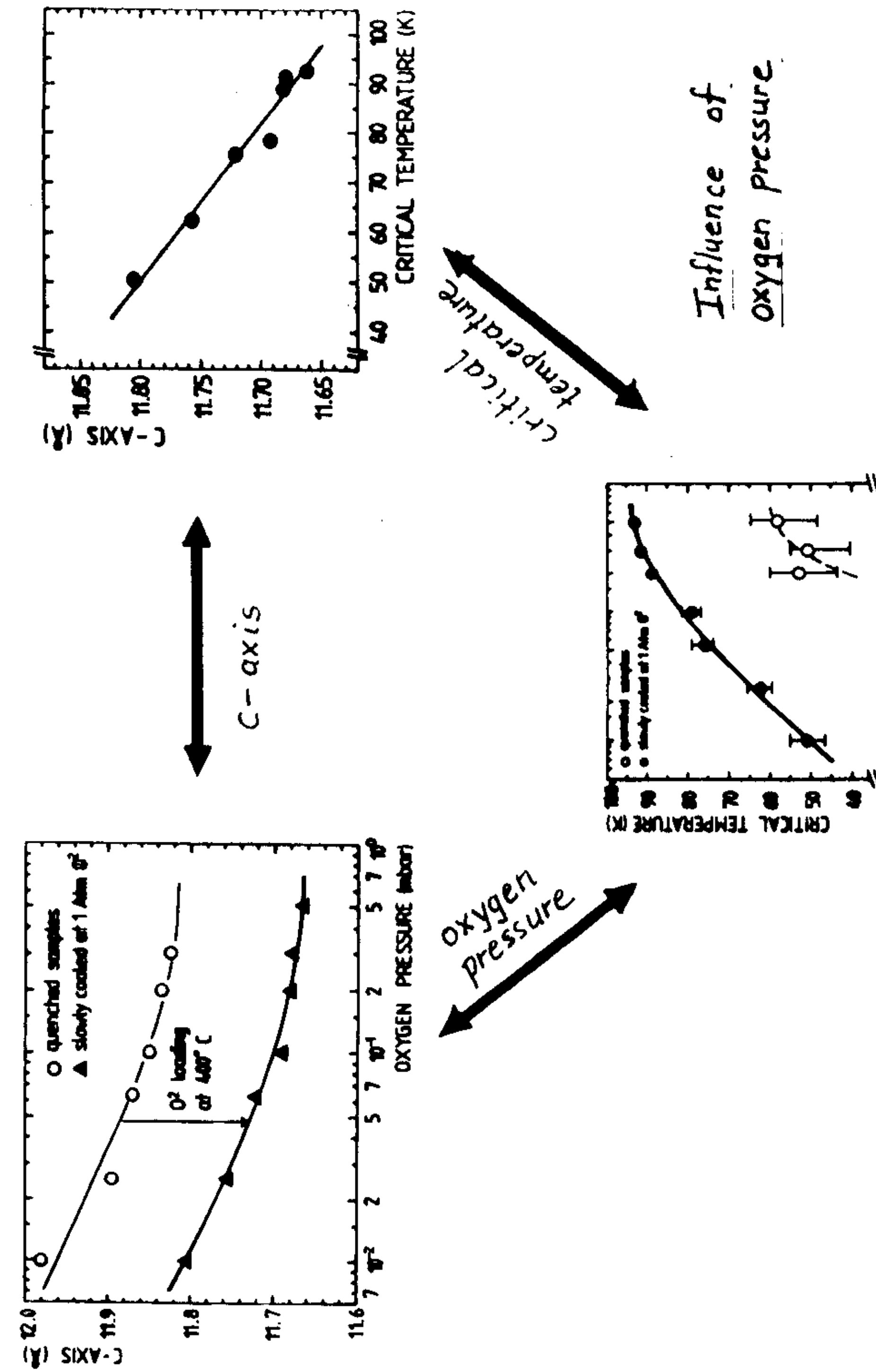




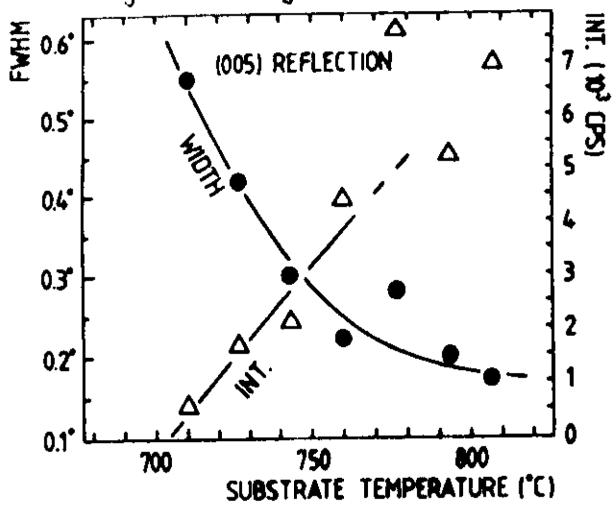




Morphologie der Filme in Abhängigkeit der Laserwellenlänge (oben: 1064 nm, unten: 266 nm).



Crystallinity:



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Influence of substrate temperature

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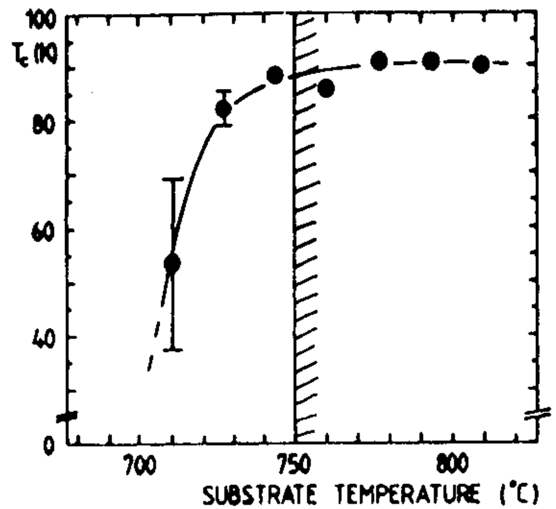
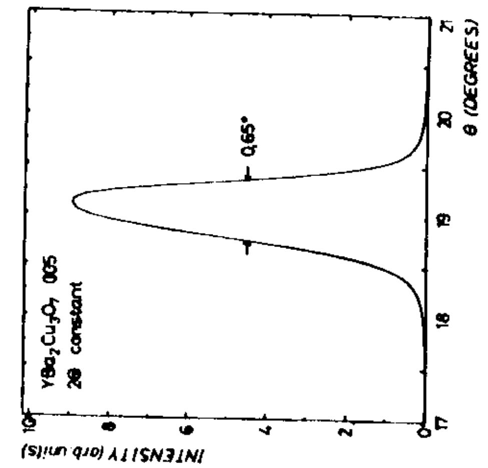


! substrate temperature too high:

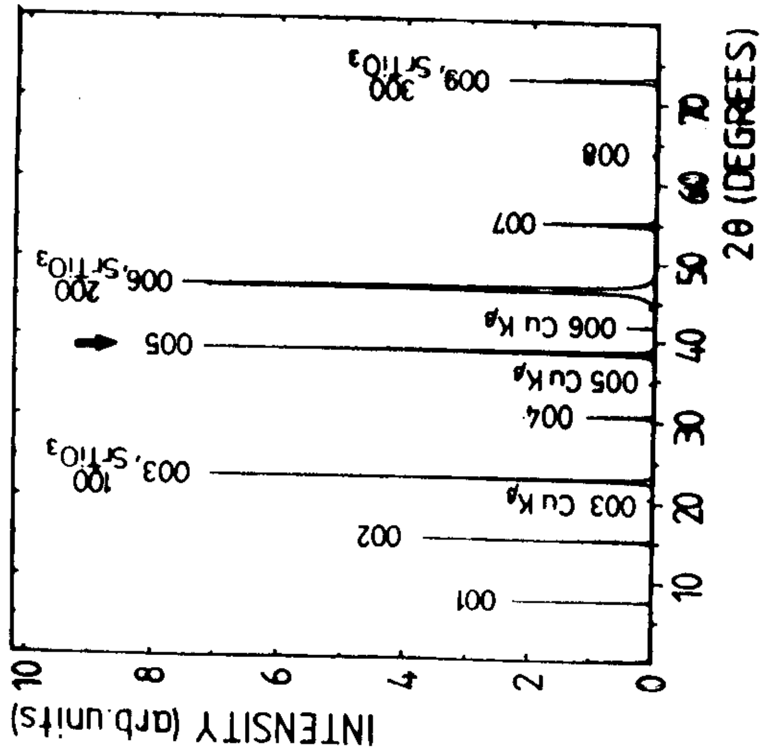


Rocking curve of the (005) peak.

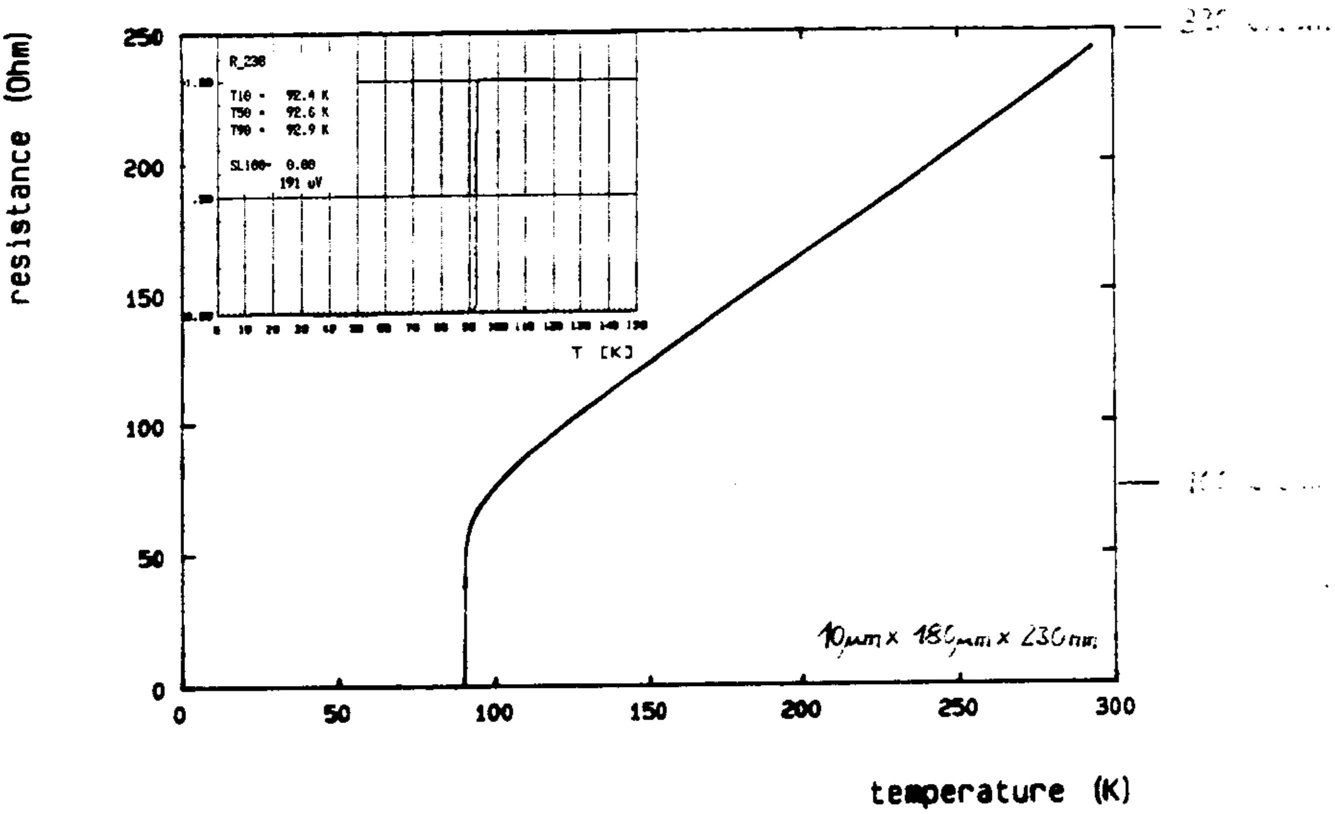
good superconductor:

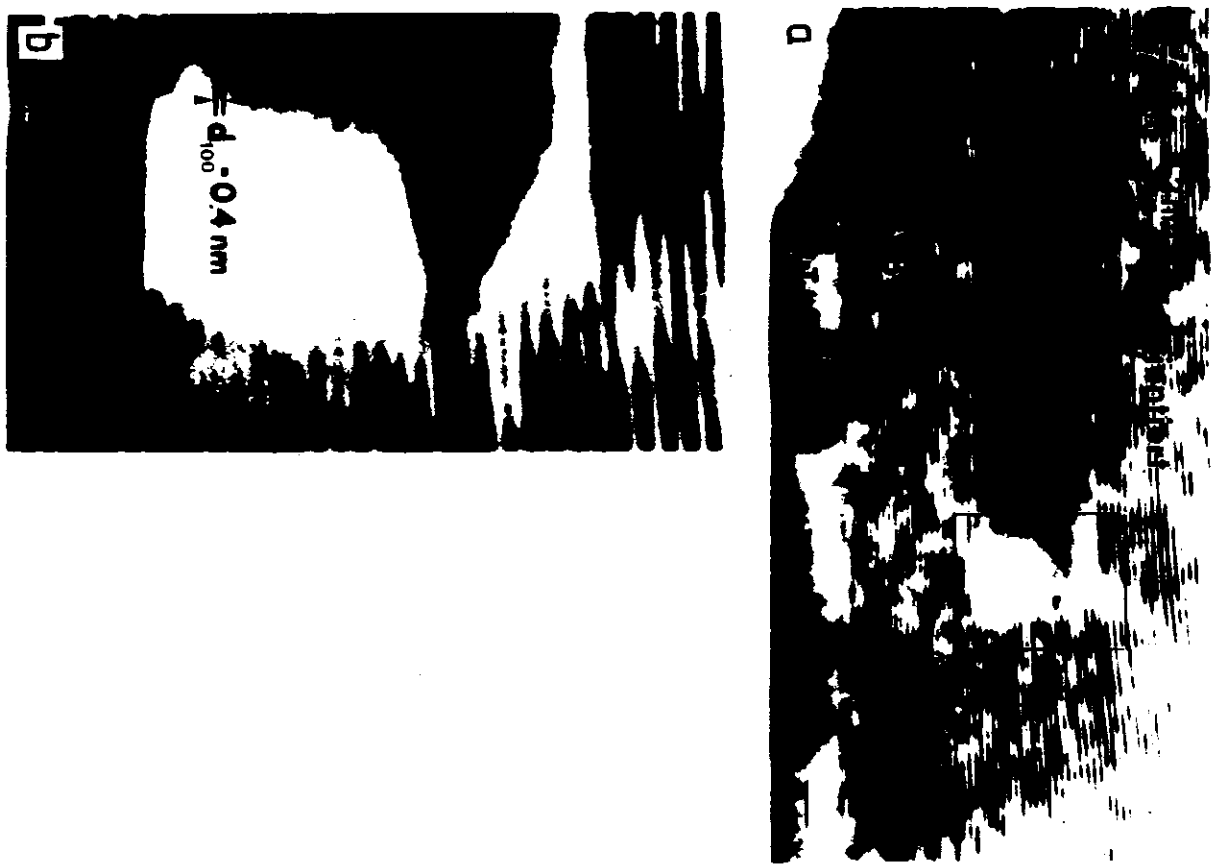
$2\Theta = 38.55^\circ$

$\hat{=}\ c = 11.67\ \text{Å}$

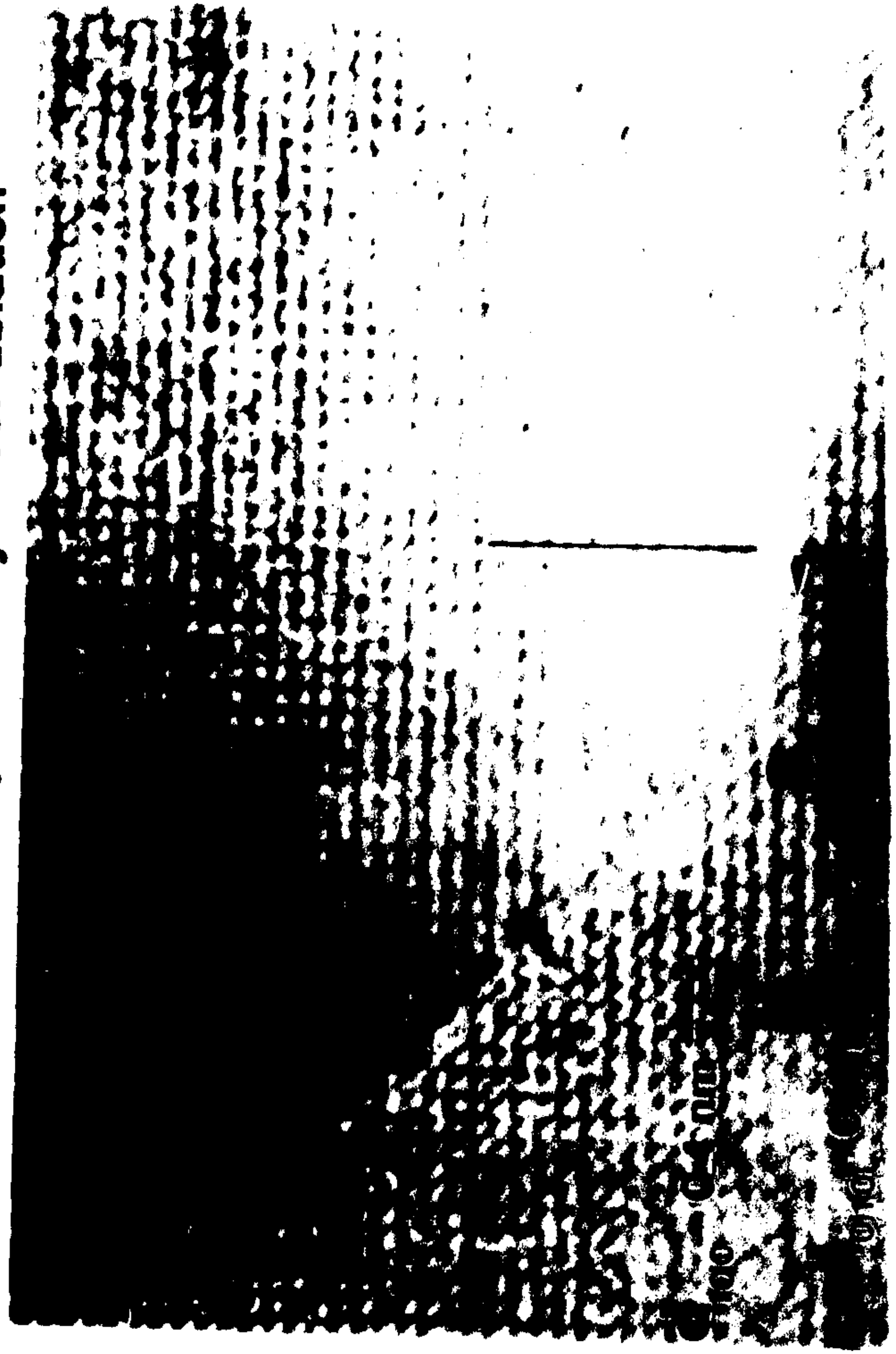


X-ray diffraction pattern of an $YBa_2Cu_3O_{7-x}$ thin film epitaxially grown on a <100> $SrTiO_3$ substrate.


resistance (Ohm)
temperature (K)
0
50
100
150
200
250
300
R_238
T10 = 92.4 K
T50 = 92.6 K
T90 = 92.9 K
SL100= 0.00
191 uV
T [K]
10μm x 180μm x 230nm


a
b
d_{100}=0.4 nm

$YBa_2Cu_3O_x$ thin film deposited by laser ablation



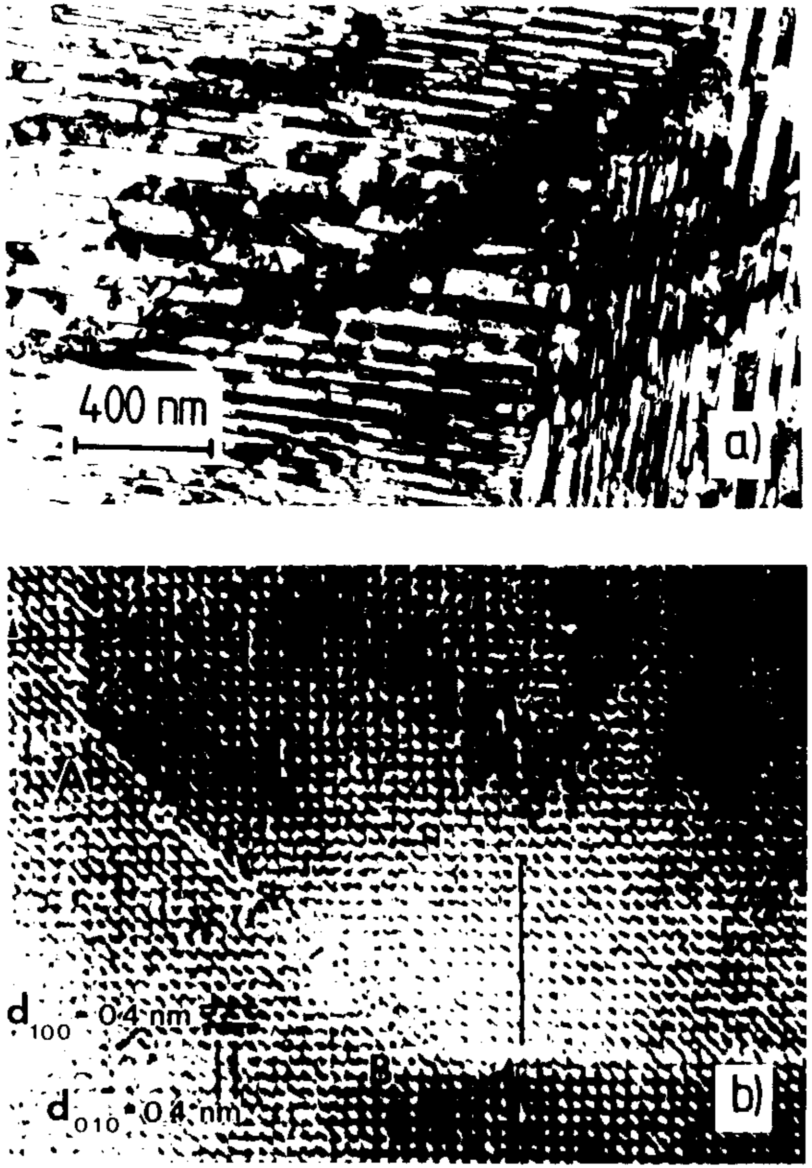


Fig. 4:

a) Twin boundaries along the [110] direction in a laser deposited $YBa_2Cu_3O_x$ film. The distance of two boundaries varies from 40 to 80 nm.

b) High resolution transmission electron micrograph of an epitaxial film in the [001] pole. A stacking fault and a shift of the lattice by half of an unit cell can be seen.

Scanning Tunneling Microscopy of laser-evaporated YBaCuO thin films in air

(in collaboration with Prof.Dr. Kirschner. Institut für Atom- und Festkörperphysik der Freien Universität Berlin)