

# Vybrané partie z elektronové mikroskopie

## část 2 Elektronové a iontové zdroje

Jaroslav Chmelík, Bohuslav Sed'a

# Contents

- Možné způsoby použití
- Charakteristiky sondy a zdroje
- Elektronové zdroje
- Iontové zdroje
- Další informace

# Use cases

- TEM - paralelní zpracování informace z plochy odpovídající zornému poli ~ 100 nm - 100 um, sonda odpovídá velikosti zorného pole
- STEM, SEM, FIB (sekvenční zpracování informace, sonda odpovídá velikosti 1 obrazového bodu)
- FIB - nanoobrábění

zvětšení		Zorné pole Odp. 100 mm	Rozlišení 2000x2000 bodů 1000 čar
100	struktury mikrosvěta zobrazitelné běžnými mikoskopy, velká hloubka ostrosti, informace o složení	1 mm	1 μm
1,000	eukaryotické buňky (všechny živočišné buňky)	100 μm	100 nm
10,000	prokaryotické buňky (bakterie)	10 μm	10 nm
100,000	viry	1 μm	1 nm
1,000,000	viry, struktura krystalové mřížky	100 nm	1 Å

# Charakteristiky sondy

velikost, proud, energie, aperturní úhel (DOF), dU, stabilita proudu, stabilita polohy, stabilita nastavení

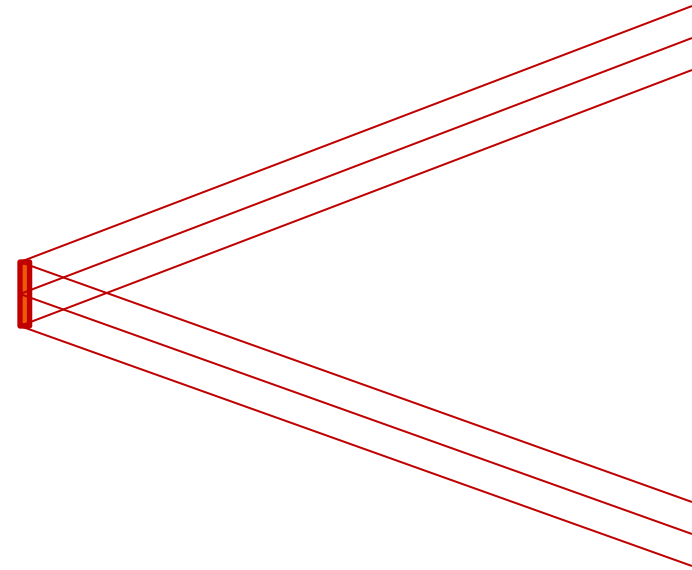
	TEM	STEM, SEM, FIB	nano obrábění
velikost sondy	100 nm - 1 mm, ale vysoká úhlová koherence	0.05 nm (STEM) 0.5 nm (SEM) - 100 nm 5 nm (FIB)	5nm - 1.5 $\mu$ m
počet částic	co vzorek snese (nabíjení vzorku, tepelné poškození)	co vzorek snese (nabíjení vzorku)	co vzorek snese (nabíjení vzorku, tepelné poškození)
energie částic	10 keV - 300 keV kontrast, rozlišovací schopnost, tloušťka vzorku	0.2 keV - 30 keV kontrast, nabíjení vzorků, rozlišovací schopnost, X-ray analýza	500eV-30keV
energieová šířka	čím menší tím lepší, chromatická vada čoček (STEM, SEM, FIB), fázový kontrast (TEM), EELS spektroskopie, typická hodnota 1 eV pro elektrony, 5 - 50 eV pro ionty		
stabilita emise	krátkodobá - nízká dlouhodobá - závisí od aplikace	krátkodobá - vysoká dlouhodobá - závisí od aplikace	krátkodobá - vysoká

# Charakteristiky zdroje

- emisní proud -  $I$
- úhel emise -  $\Omega$
- velikost zdroje -  $S$
- energie emitovaných částic -  $\varphi$
- energieová šířka emise
- stabilita emisního proudu
- plošná proudová hustota emise

úhlová proudová hustota emise  
(angular intensity) [mA/sr]

$$I' = \frac{I}{\Delta\Omega} = \frac{I}{\pi\alpha^2}$$



směrová proudová hustota  $B$   
(brightness) [A/sr/m<sup>2</sup>]

$$\beta = \frac{I}{\Delta S \Delta\Omega} = \frac{4I}{(\pi d \alpha)^2}$$

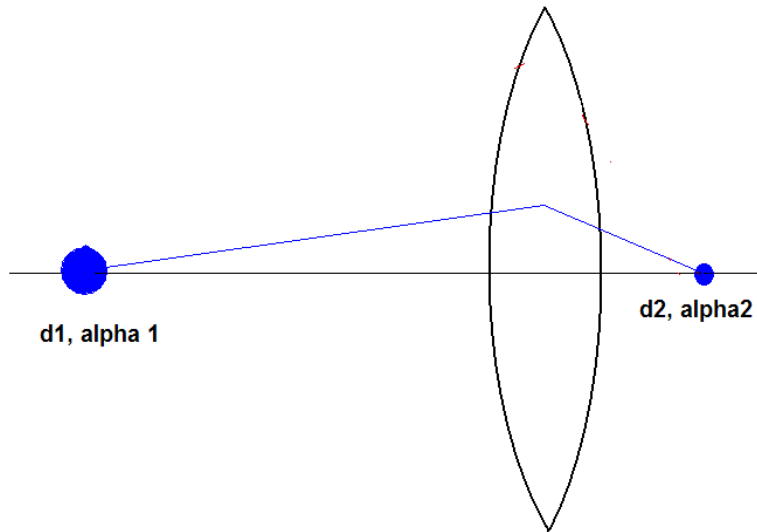
reduced brightness  $B_r$  [A/sr/m<sup>2</sup>/V]

$$\beta_r = \frac{I}{\Delta S \Delta\Omega \varphi} = \frac{4I}{(\pi d \alpha)^2 \varphi}$$

# Brightness

Unipotencial lens:

- direct magnification = 1/ angular magnification
- brightness does not change



$$M' = M^{-1}$$

$$\beta_1 = \frac{4I}{(\pi d_1 \alpha_1)^2}$$

$$d_2 = d_1 \cdot M$$

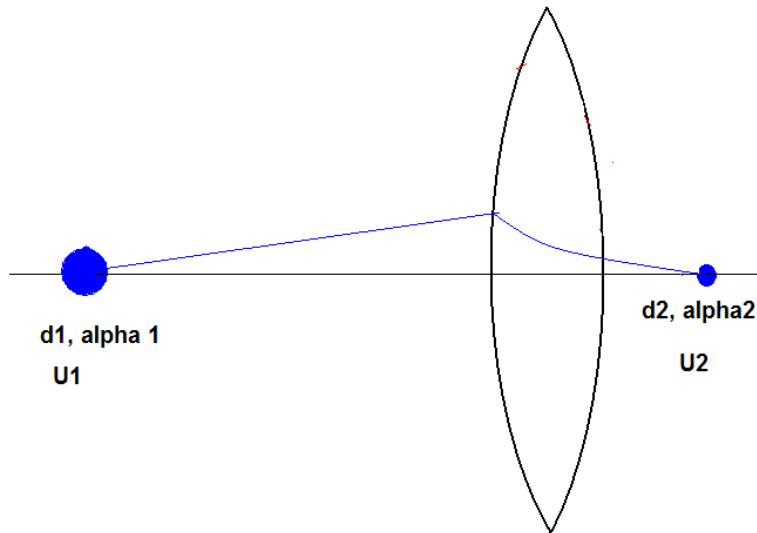
$$\alpha_2 = \alpha_1 \cdot M' = \alpha_1 \cdot M^{-1}$$

$$\beta_2 = \frac{4I}{(\pi d_2 \alpha_2)^2} = \frac{4I}{(\pi d_1 M \alpha_1 M^{-1})^2} = \beta_1$$

# Brightness

Accelerating and decelerating lenses:

- direct magnification  $\neq 1$  / angular magnification
- brightness does change
- **reduced brightness doesn't change**



$$M' = \sqrt{\frac{U_1}{U_2}} M^{-1}$$

$$\beta_1 = \frac{4I}{(\pi d_1 \alpha_1)^2}$$

$$d_2 = d_1 \cdot M$$

$$\alpha_2 = \alpha_1 \cdot M' = \alpha_1 \cdot \sqrt{\frac{U_1}{U_2}} M^{-1}$$

$$\beta_2 = \frac{4I}{(\pi d_2 \alpha_2)^2} = \frac{4I}{(\pi d_1 M \alpha_1 M^{-1})^2} \frac{U_1}{U_2} = \beta_1 \frac{U_2}{U_1}$$

# Proud sondy → SNR

- SEM
- obrázek 1000x1000 bodů
- dwell time 50 ns -> frame time = 50 ms -> refresh rate 20 obr/s
- 1 dopadající elektron = 1 detekovaný signálový elektron
- shot noise - Poisson distribution  $SNR = \sqrt{N}$

SNR	pocet dop. elektr.	proud sondy [pA]
3	9	32
6	36	115
12	144	460



# Brightness zdroje

- SEM
- obrázek 1000x1000 bodů
- dwell time 50 ns -> frame time = 50 ms -> refresh rate 20 obr/s
- 1 dopadající elektron = 1 detekovaný signálový elektron
- shot noise - Poisson distribution  $SNR = \sqrt{N}$
- velikost sondy 1.0 nm
- aperturní úhel 10 mrad
- energie elektronů 10 keV

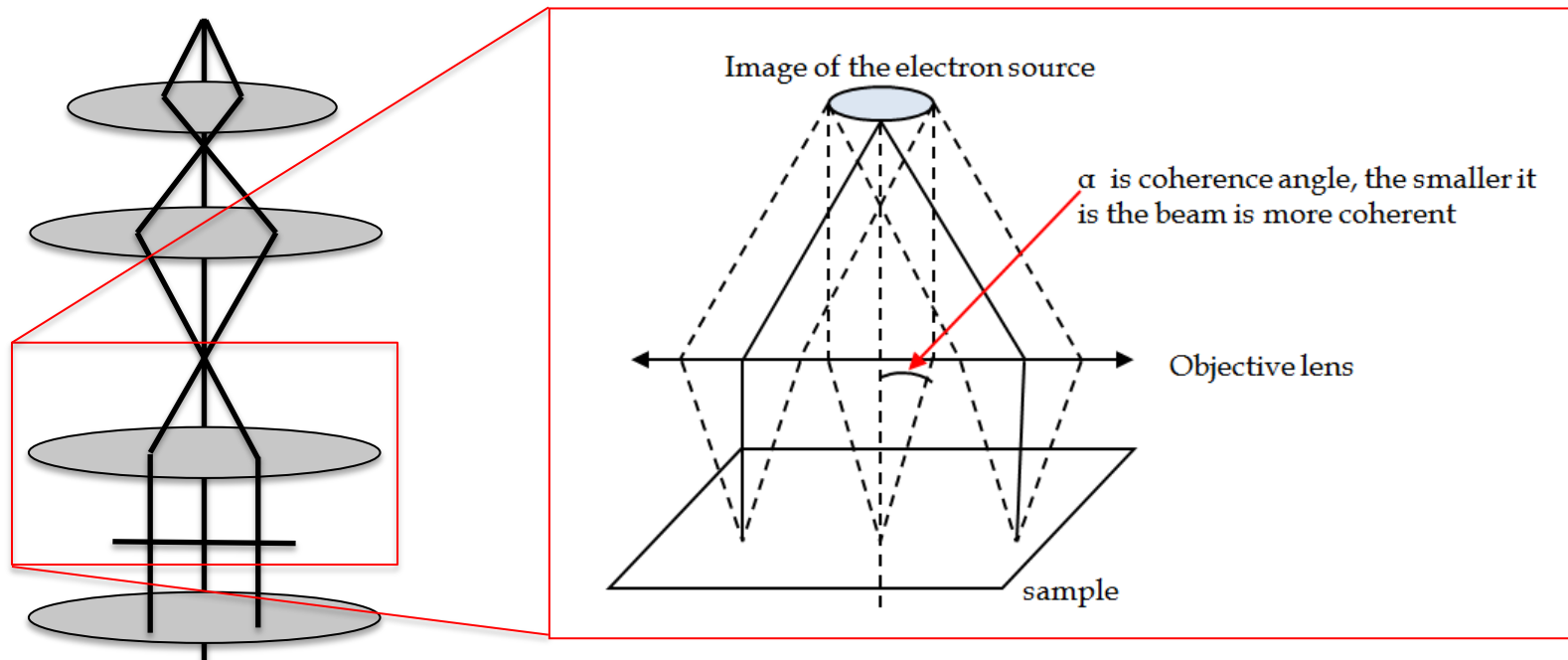
*Brightness určuje velikost proudu, který lze vtěsnat do sondy dané velikosti a aperturního úhlu*

SNR	počet dop. elektronů/pixel	proud sondy [pA]	red. Brightness [A/m <sup>2</sup> *sr*eV]
3	9	32	3*10 <sup>6</sup>
6	36	115	1*10 <sup>7</sup>
12	144	460	4*10 <sup>7</sup>

# Brightness zdroje

- TEM

*Brightness určuje velikost proudu, který lze vtěsnat do paralelního svazku daného průřezu a úhlové koherence*



# Probe size and Source characteristics

Angular intensity

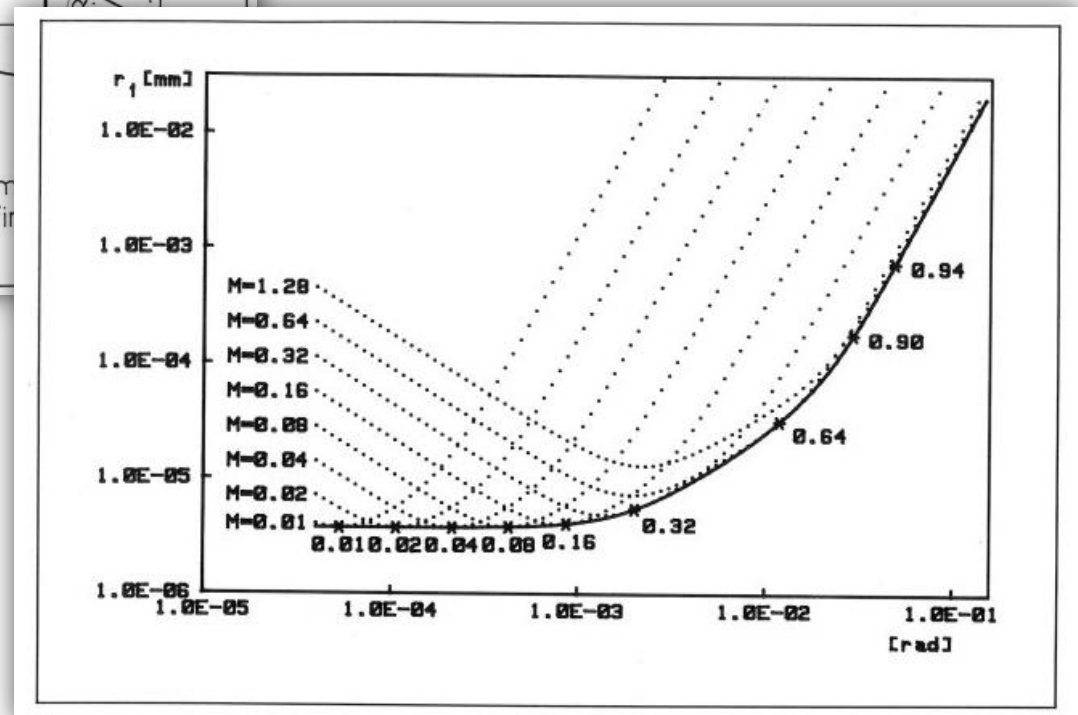
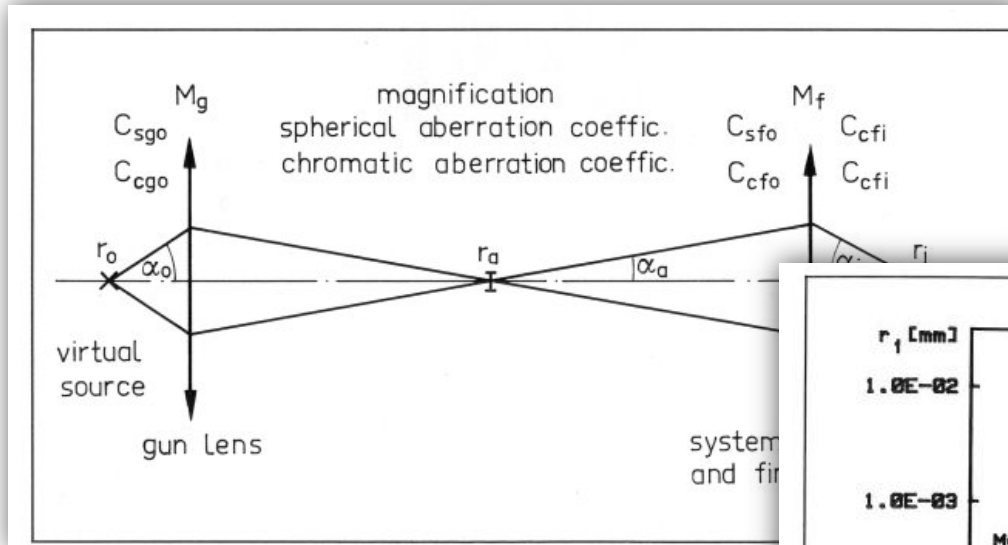
Virtual source size

Energy spread

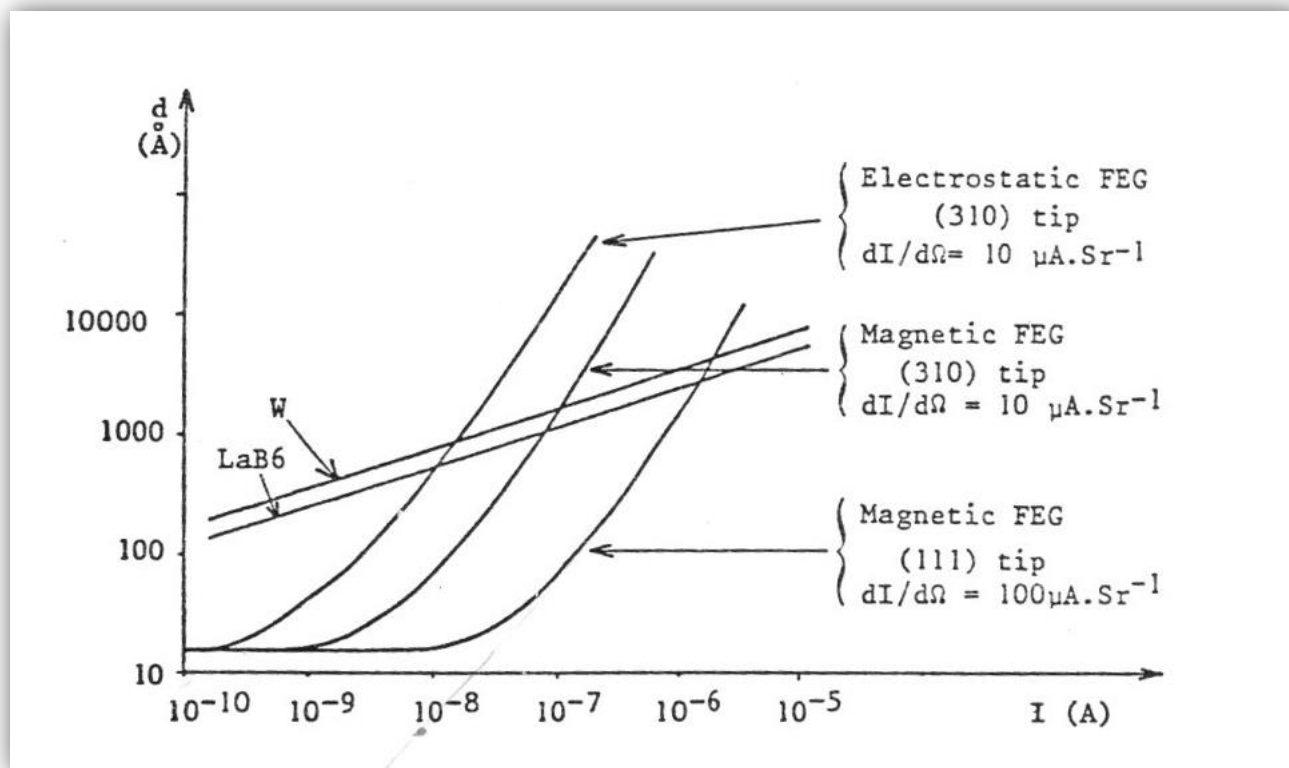
$$d_p^2 = \frac{4I_p}{\pi^2 \beta_r} \alpha^{-2} + (0.6\lambda)^2 \alpha^{-2} + \frac{1}{4} C_s^2 \alpha^6 + \left( C_c \frac{\Delta E}{E} \right)^2 \alpha^2$$

Probe size → Resolution

# Optimální velikost stopy v závislosti na proudu ve stopě



# Optimální velikost stopy v závislosti na proudu ve stopě



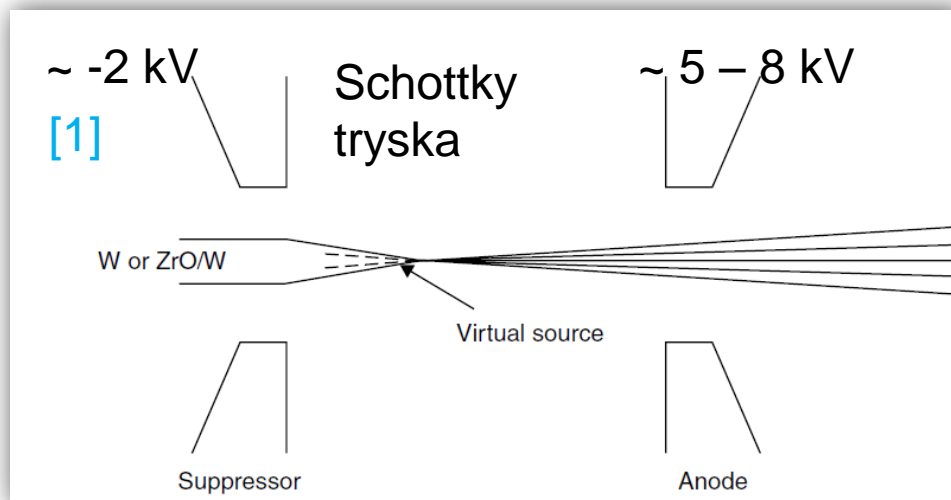
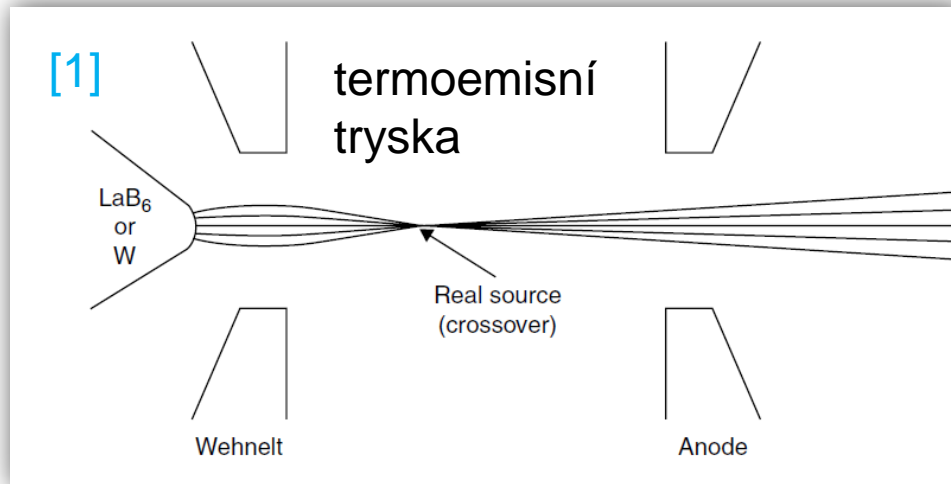
# Elektronové zdroje (dělení)

- termoemisní (thermionic)
  - wolframová vlásenka (hairpin filament, tungsten)
  - LaB6, CeB6 katody
- autoemisní
  - Schottky ZrO/W
  - studený W hrot (cold FEG)

- termoemisní (thermionic)
  - wolframová vlásenka (hairpin filament, tungsten)
  - LaB6, CeB6 katody
- Schottky Emission
- Field Emission
  - cold field emission
  - thermal assisted field emission

point source cathodes

# Real and virtual source



# Porovnání parametrů jednotlivých trysek

Tryska	Termoemisni		Schottky	FEG
Katoda	W	LaB6	W/ZrO	Cold FEG
Výstupní práce [eV]	4.5	2.4	2.7	4.5
Pracovní teplota [K]	2700	1700	1750	300
Velikost křížště [μm]	50	10	~ 0.015	~ 0.005
Energiová šířka [eV]	2-3	1.5	0.6-0.9	0.2-0.3
Vakuum [Pa]	10 <sup>-3</sup>	10 <sup>-4</sup> -10 <sup>-5</sup>	10 <sup>-6</sup> -10 <sup>-7</sup>	10 <sup>-8</sup> -10 <sup>-9</sup>
Maximální proud [μA]	1- 3	1-3	0.3	0.1
Životnost katody [h]	40-100	500-1000	>2000	>2000
Brightness [A/m <sup>2</sup> sr]@100 kV	(1-3)*10 <sup>9</sup>	(3-10)*10 <sup>9</sup>	(0.2-1)*10 <sup>13</sup>	(0.5-5)*10 <sup>13</sup>
Red. brightness [A/m <sup>2</sup> sr·eV]	(1-3)*10 <sup>4</sup>	(3-10)*10 <sup>5</sup>	(0.2-1)*10 <sup>8</sup>	(0.5-5)*10 <sup>8</sup>
Směrová proudová hustota [mA/sr]			0.1 - 1	0.3 - 4



- termoemisní W : levné, nízké nároky na vakuum, stabilní, robustní, dobrá alternativa pokud brightness zdroje je dostatečná pro danou aplikaci a výměna po cca 100 hodinách nepředstavuje problém v použití
- termoemisní LaB6 : vyšší nároky na vakuum (IGP), provozně dražší, delší doba života, stabilní, robustní, o něco vyšší jas, hodně používána v TEM pro běžnou práci (jas LaB6 dostatečný při 100 kV)
- Schottky ZrO/W : vysoké nároky na vakuum (2xIGP), provozně dražší, velmi vysoký jas, malá energiová šířka emise, nízký šum, hodně používána v SEM
- CFE : extrémně vysoké nároky na vakuum, nejvyšší jas, nejnižší energiová šířka zdroje, šum vyšší než ZrO/W a zhoršující se s vakuem, pro některé aplikace malý maximální dosažitelný proud, použití nachází ve špičkových TEM

# Termoemisní elektronová tryska - fyzikální princip

distribuční funkce

termoemisní elektrony

$$\Phi_s = 4.5 \text{ eV (W)}$$

$$\Phi_s = 2.7 \text{ eV (LaB6)}$$

$$k^*T / \Phi_s \sim 0.05$$

Richardson law:

$$j_c = A T_c^2 \exp(-\phi / kT_c)$$

$E_F$  (W)

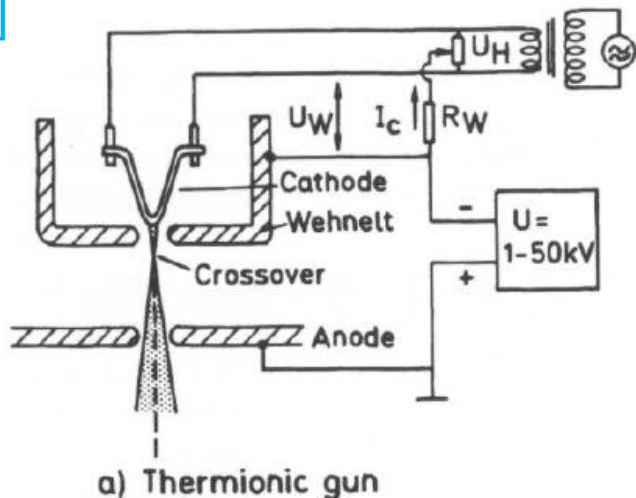
vodivostní  
elektrony (0 K)

kov

vakuum

# Termoemisní elektronová tryska

[2]



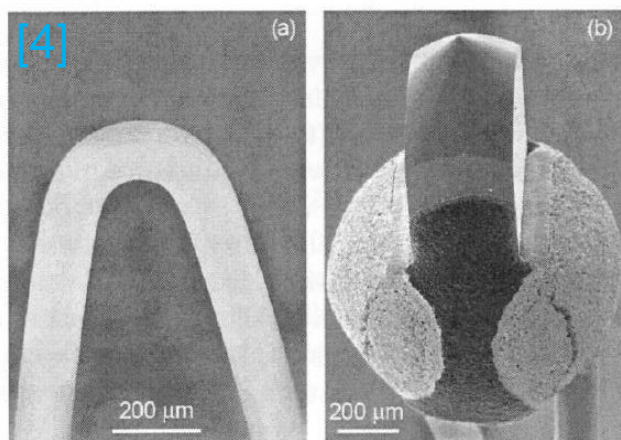
- katoda - wolframové vlákno  $\varnothing$  0.1 - 0.15 mm, zahnuté do tvaru vlásenky nebo nepřímo vyhříváný LaB6 (CeB6) krystal, záporný potenciál odpovídající požadované energii svazku snižené o úbytek napětí pro wehnelt

- žhavicí proudový zdroj,  $T \sim 2700$  K (W), 1900 K (LaB6)

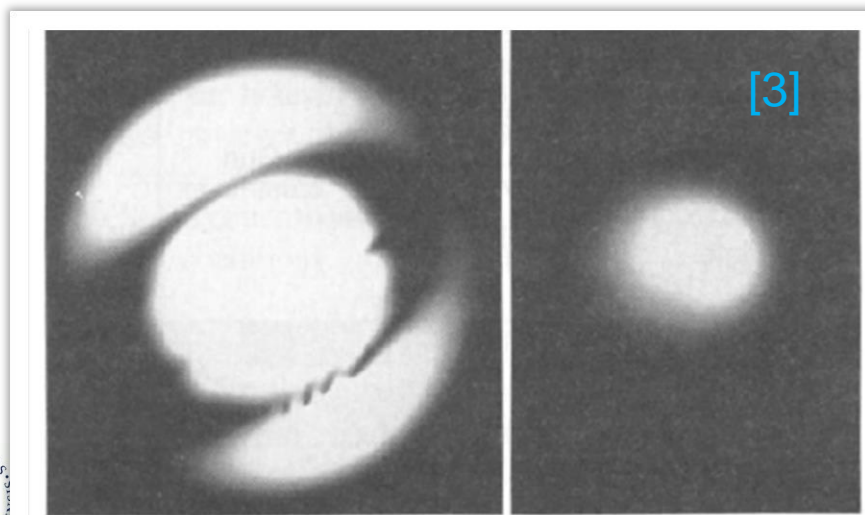
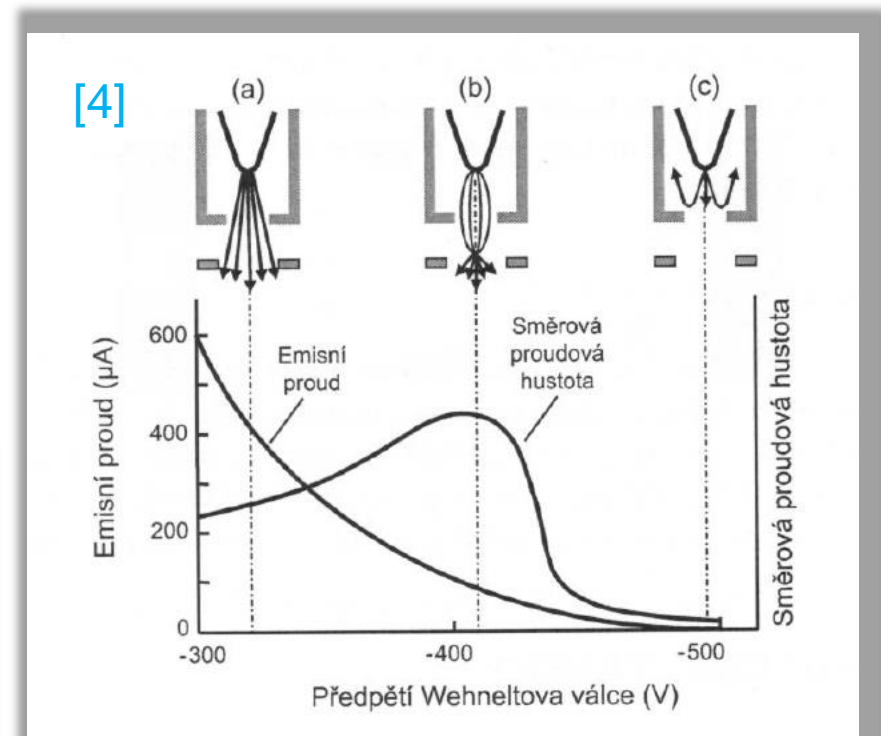
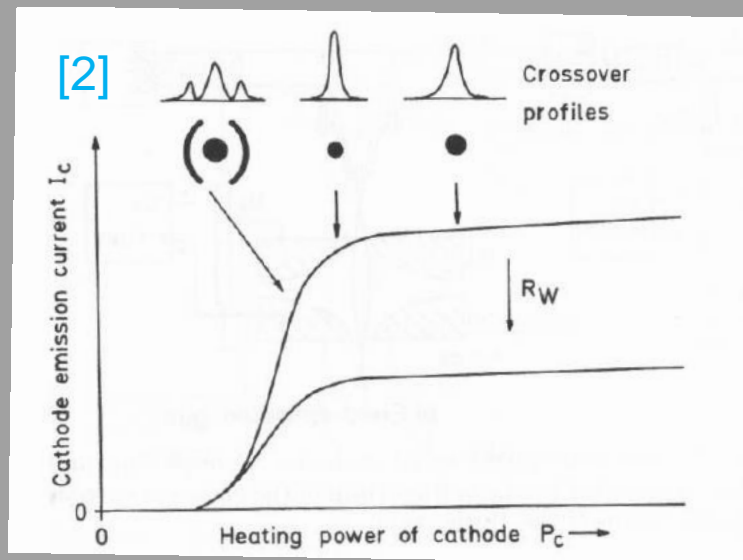
- stínící elektroda (wehnelt), negativní předpětí 100 - 1000 V oproti katodě -  $R_w \cdot I_e$  (autobias)

- anoda - na zemním potenciálu

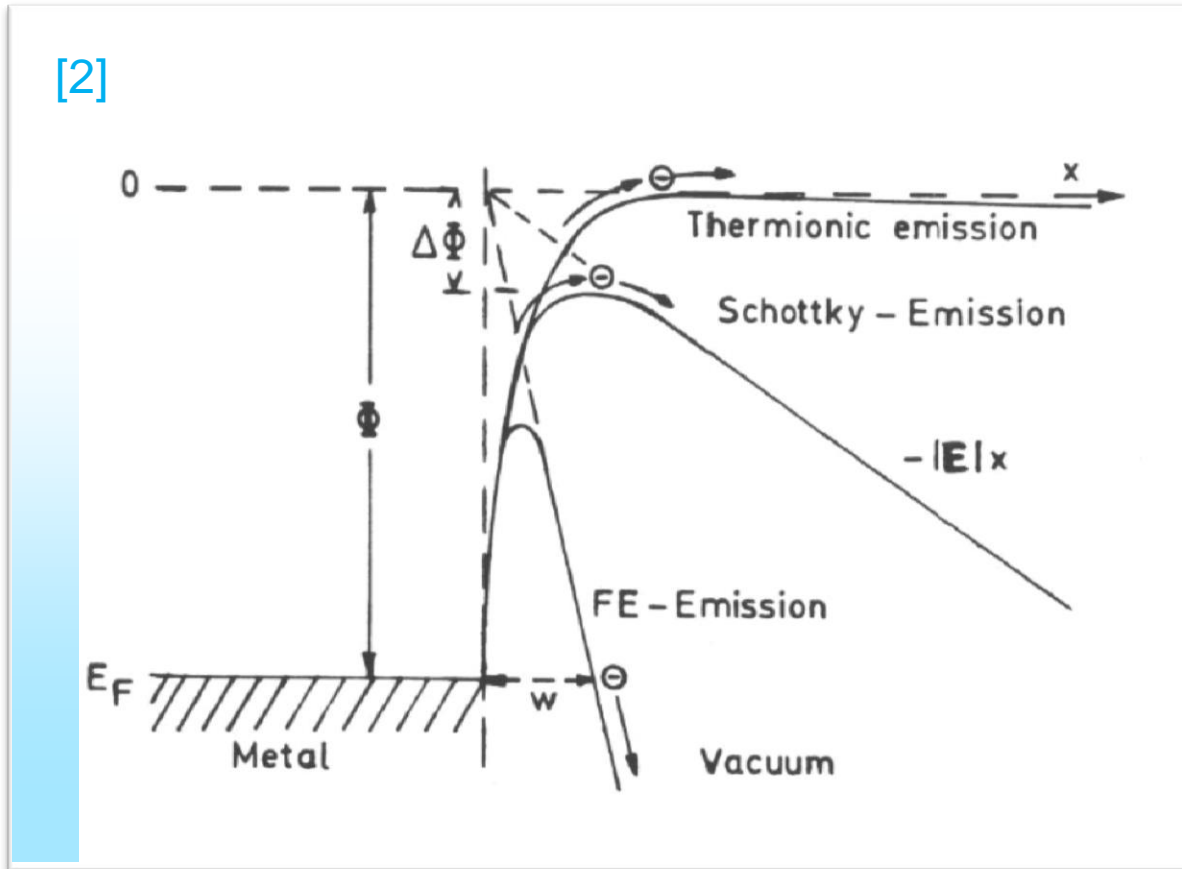
[4]



# Termoemisní elektronová tryska - použití



# Fyzikální principy elektronové emise



- vliv vnějšího pole na snížení výšky potenciálové bariéry  
 $10^6 \text{ V/cm} \sim 0.4 \text{ eV}$

- vliv vnějšího pole na zúžení potenciálové bariéry  
 $10^7 \text{ V/cm} \sim \text{nm}$

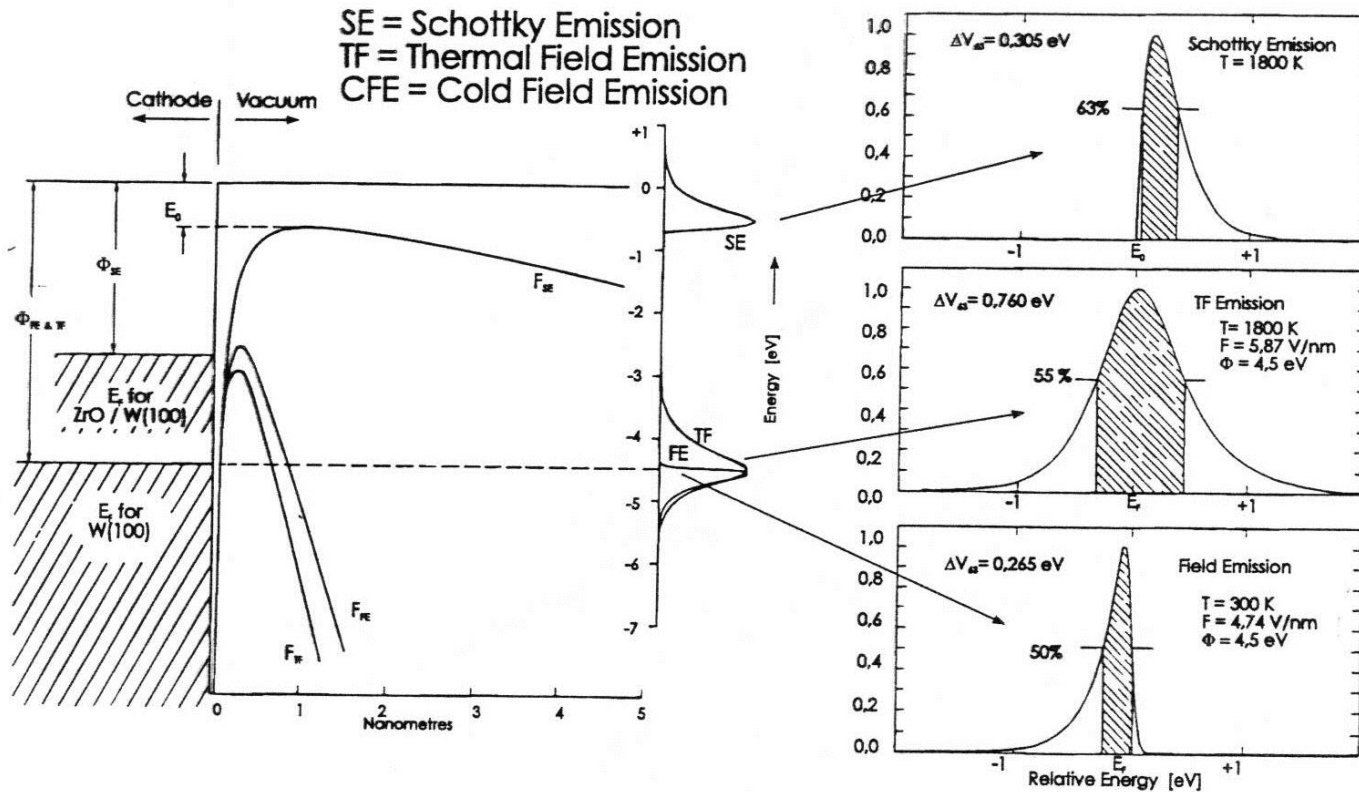
- výstupní práce

- materiál
- krystalová orientace
- aktivace

- teplota katody

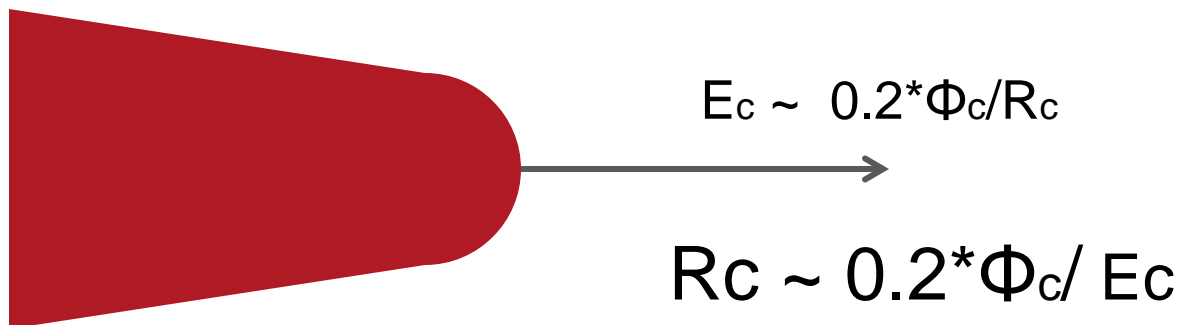
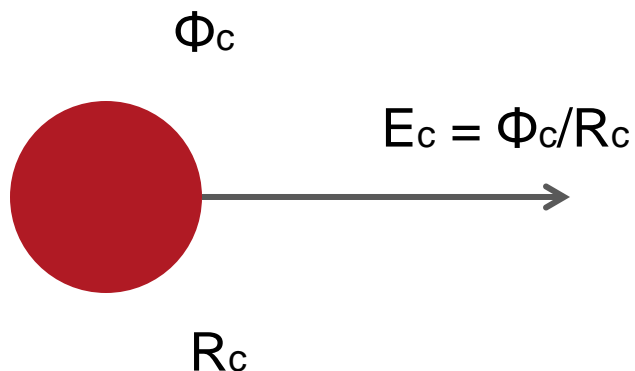
# Energy spread of emitted electrons

Energy level diagram and resulting energy distributions for the three emission regimes:



After: D.W. Tuggle et al., J. Microscopy 140 (1985) 293

# Jak získat vysokou intenzitu pole?



$$R_c \sim 0.2 \cdot 5 \cdot 10^3 / 10^8 = 10^{-5} \text{ m}$$

# Autoemisní elektronová tryska - funkce a jejich implementace

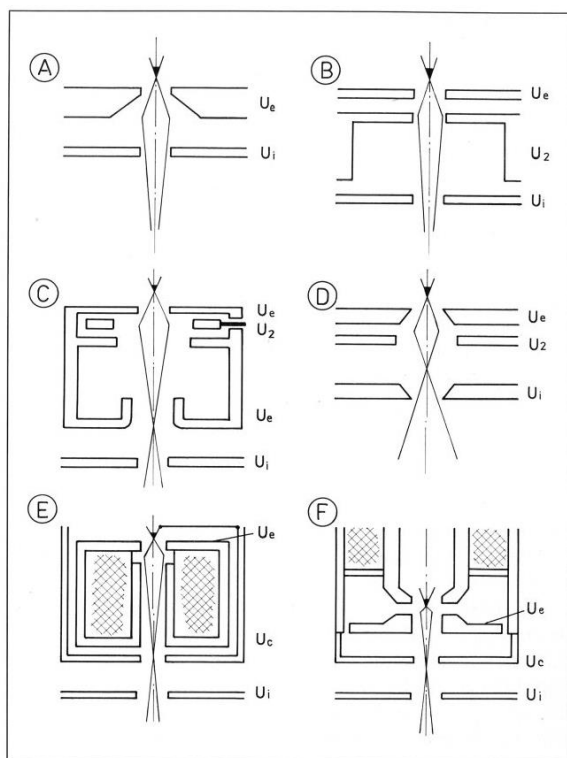


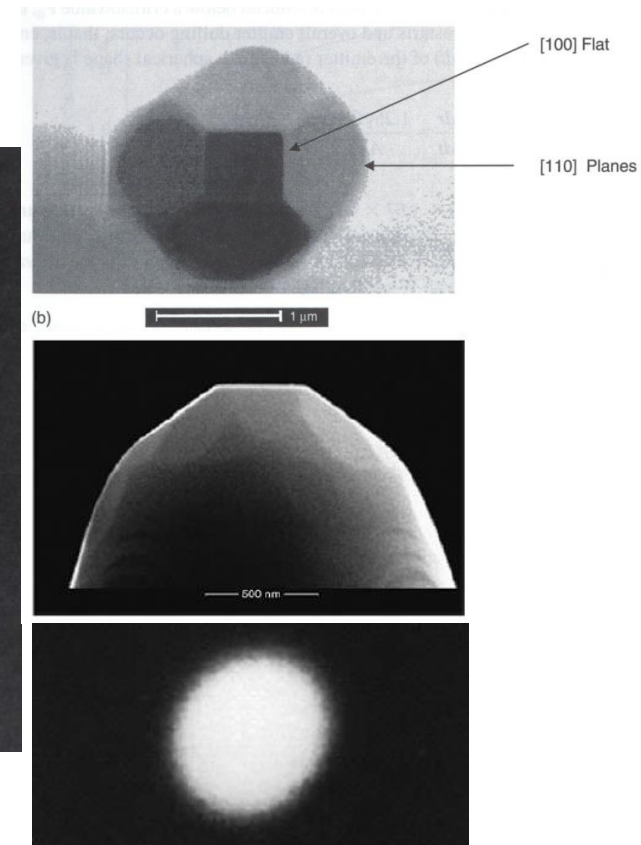
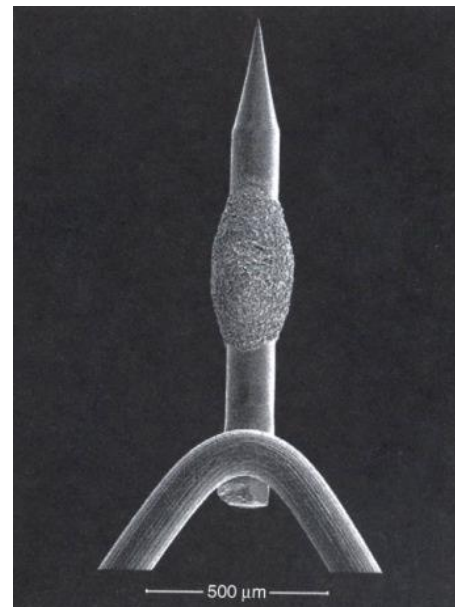
Fig. 4. The main types of FEG (schematic) in common use.  
A – Triode gun [7], [8], [9], [10].  
B – Tetrode gun [11].  
C – Pentode with einzel lens, crossover in the accelerator [12].  
D – Tetrode with crossover in the accelerator [13], [14].  
E – Tetrode gun with magnetic pre-accelerator lens [5], [6], [15].  
F – Present tetrode gun with magnetic pre-accelerator lens.

- extrakce elektronů z hrotu
- urychlení na požadovanou energii
- fokusace elektronů
- centrovací prvky  
(mechanické/elektrostatické)
- energiová filtrace

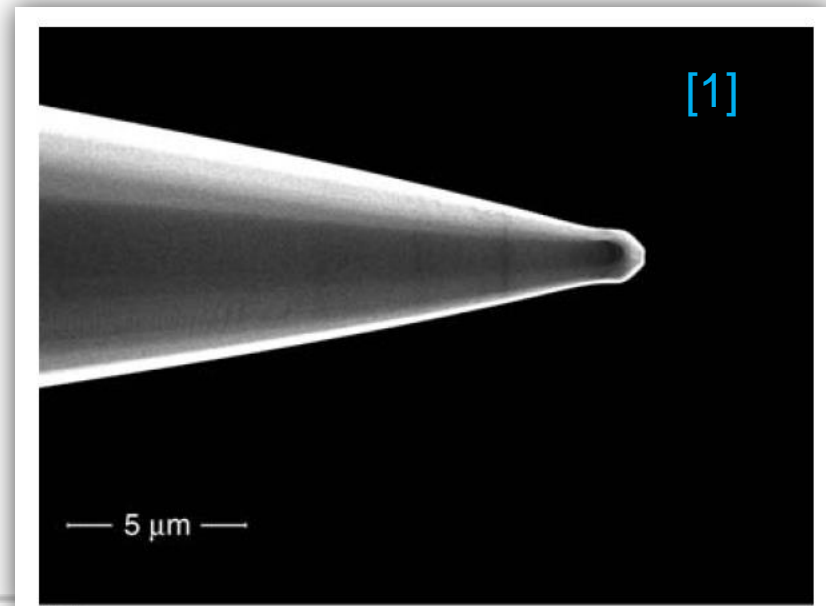
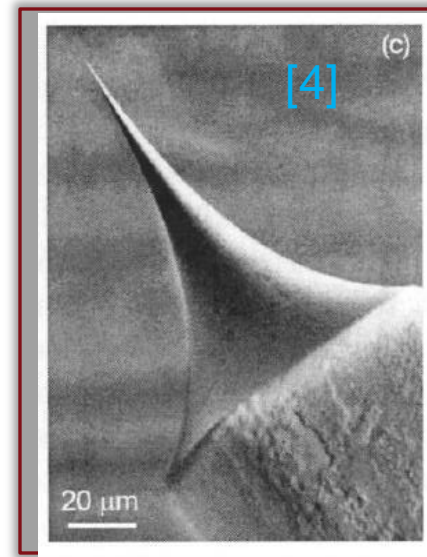
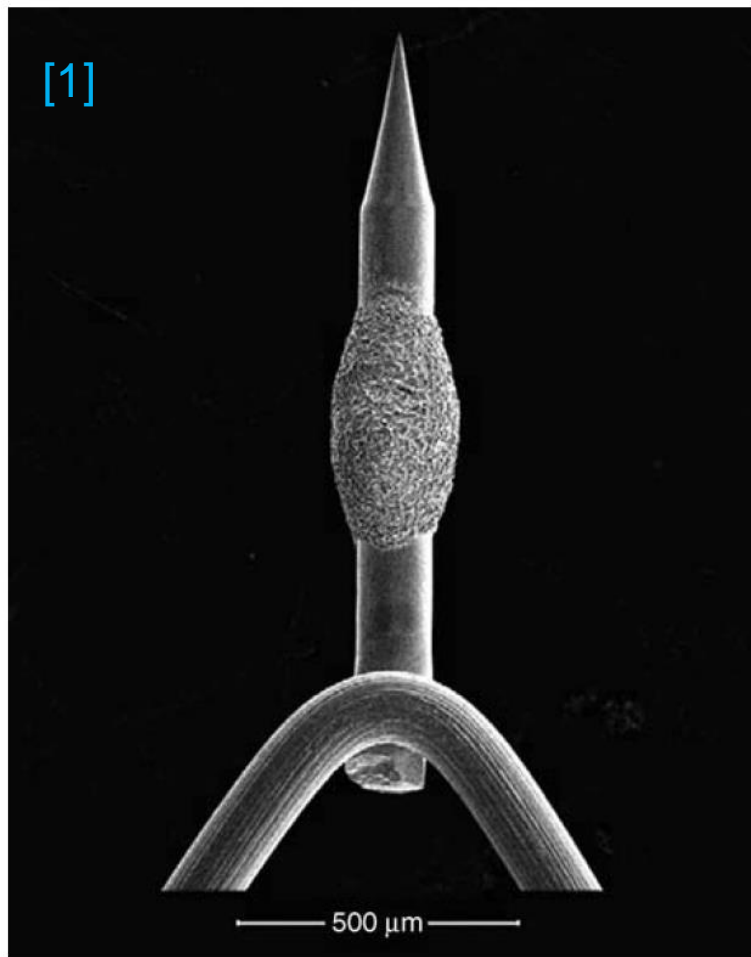


# ZrO/W Schottky emitter

- today most widespread electron source
- point cathode
- tip radius 0.3 - 1  $\mu\text{m}$
- field assisted thermal emission
- virtual crossover  $\sim 20\text{ nm}$
- single crystalline W rod  $\langle 100 \rangle$
- ZrO<sub>2</sub> reservoir
- temperature 1750-1800 K
- tip shape and field strength
- lowering work function to  $\sim 2.7\text{ eV}$ 
  - material
  - crystal orientation
  - ZrO
  - E field



# ZrO<sub>2</sub>/W Schottky emitter



# Extraction section

## 3 electrode setup

- part of uniform emission from central low-work-function (100) crystal plane is transmitted through extractor aperture
- the rest of central emission including thermal emission from other facets is collected on extractor electrode

## Tip

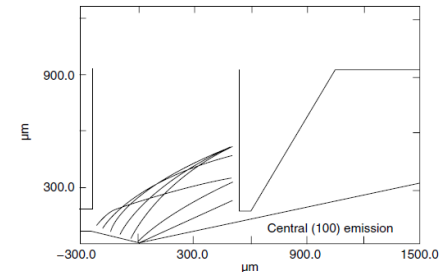
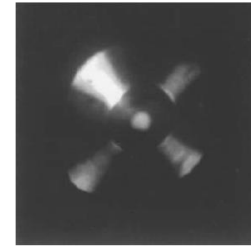
- acceleration voltage applied to tip electrode (cathode)

## Suppressor

- reduce total emission current by reducing extraneous thermal emission from tip electrode cylindrical shaft

## Extractor

- Extractor voltage sets angular intensity
- $V_{ext} = f(\text{angular intensity, central plane WF, emitter radius, emitter T, electrode setup, ...})$
- initial acceleration



$I_{total}$

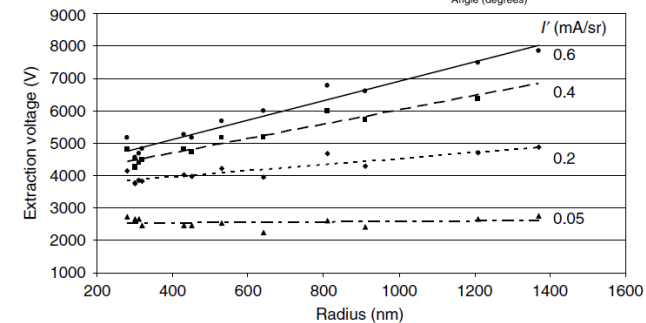
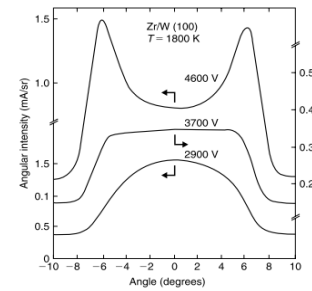
$\sim 100 \mu A$

$I_{facet}$

$\sim 10\%$

$I_{probe}$

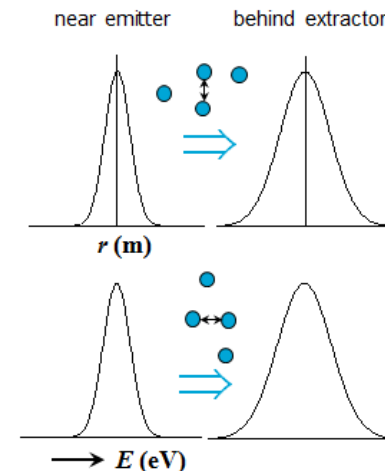
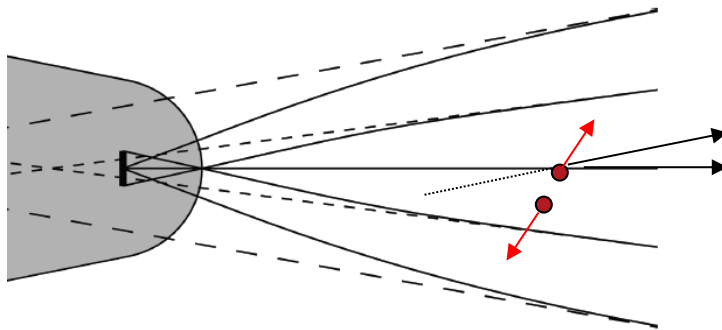
$\sim 1-1e-6\%$



# Coulomb interactions $\rightarrow$ dU, Brightness

- Right after emission  $B = 1.44 \frac{ej}{\pi k_B T} \sim 10^8 \frac{A}{m^2 srV}$   $\Delta E_{FW50} = 0.3 \text{ eV}$
- Both change with  $T$ , field, work function

- Longitudinal (Boersch effect)  $\rightarrow$  energy spread increase
- Lateral (Loeffler effect)  $\rightarrow$  crossover enlargement, trajectory displacement, radial broadening  $\rightarrow$  reduced brightness reduction
- Reduced brightness is highest at tip surface
- Energy spread is lowest at tip surface
- Using smaller gun aperture lowers Coulomb interactions



# Vzájemná interakce nabitých částic

Závislosti na  $m$ ,  $l$ ,  $V$ ,  $\alpha$

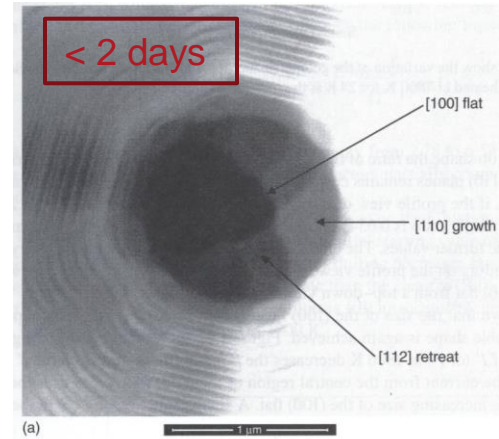
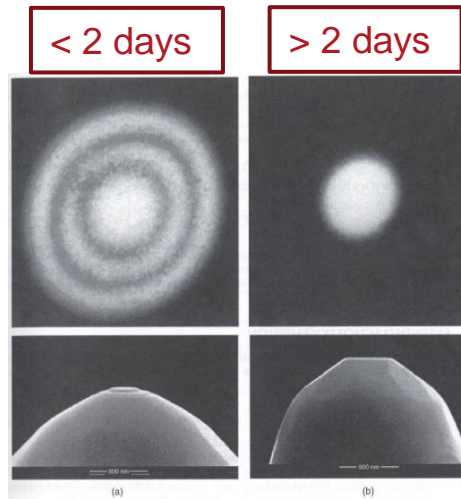
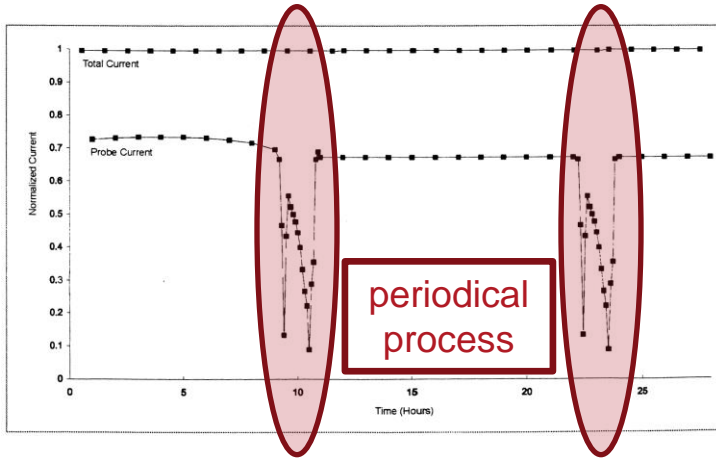
$$\frac{\partial E}{E} \sim \frac{m^{1/2} l}{\alpha V^{3/2}} \quad (\text{energiové rozšíření - průchod svazku křížištěm})$$

$$\frac{\partial E}{E} \sim \frac{m l^2 L}{V^2} \quad (\text{energiové rozšíření - rovnoběžný svazek bez křížiště})$$

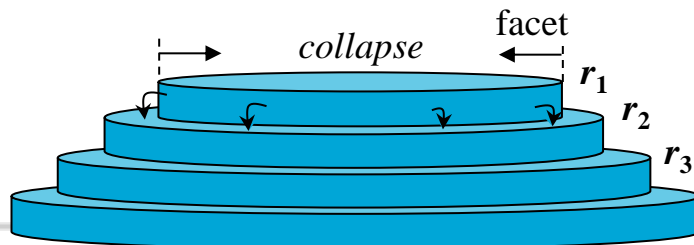
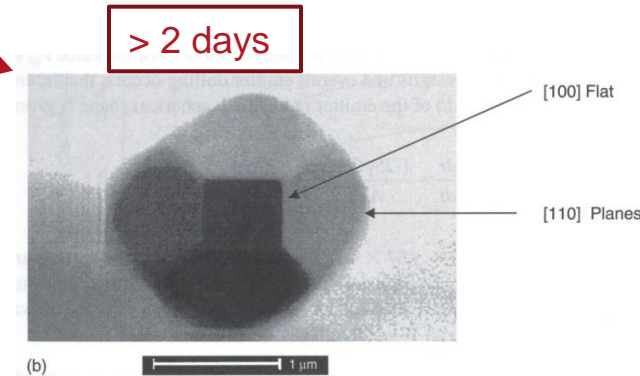
$$\Delta r \sim \frac{m l^2 L}{\alpha^3 V^3} \quad (\text{„trajectory displacement“})$$

$$\Delta \alpha \sim \frac{m l^2}{\alpha^3 V^3} \quad (\text{„angular” spread})$$

# Ring collapse

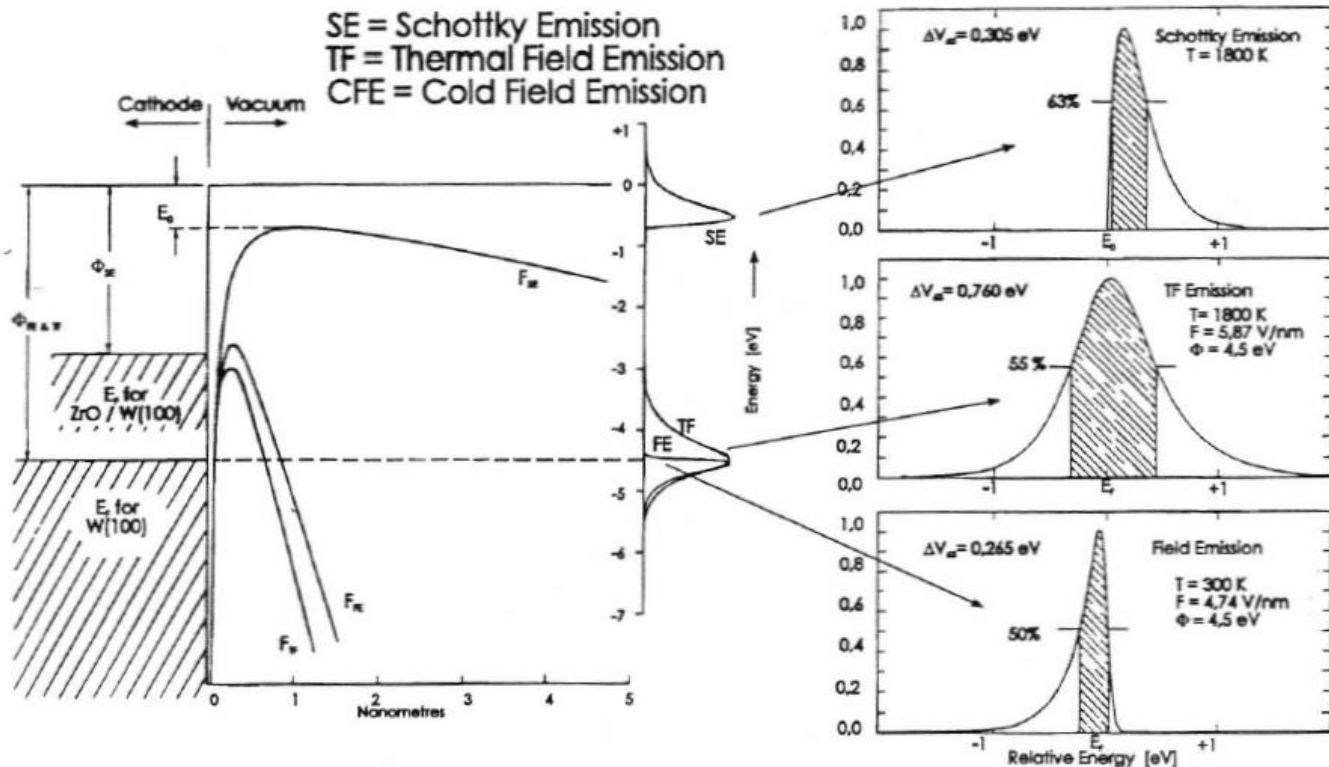


- angular intensity  $I' \geq X \text{ mA/sr}$  is necessary to maintain **stabilized endform**  
 → stabilized angular intensity
- lower angular intensity →
  - lower Energy spread → lower d50 at low beam energies
  - lower Brightness → higher d50 at high beam currents



# Total energy distribution

Energy level diagram and resulting energy distributions for the three emission regimes:



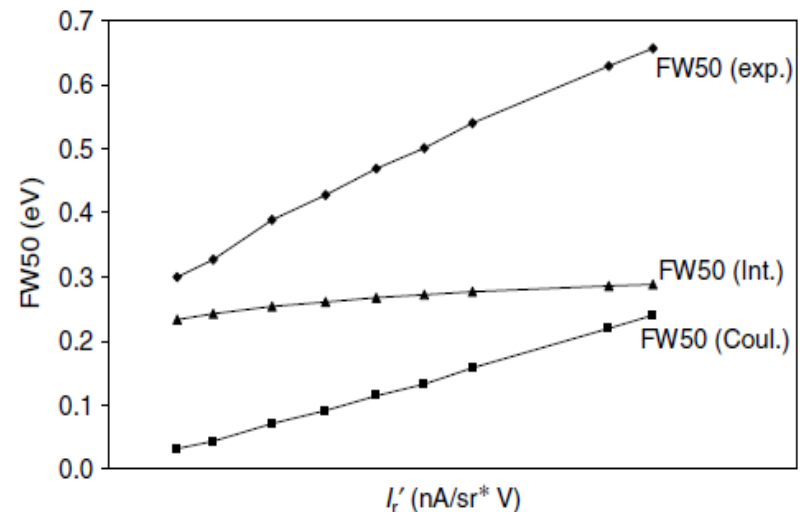
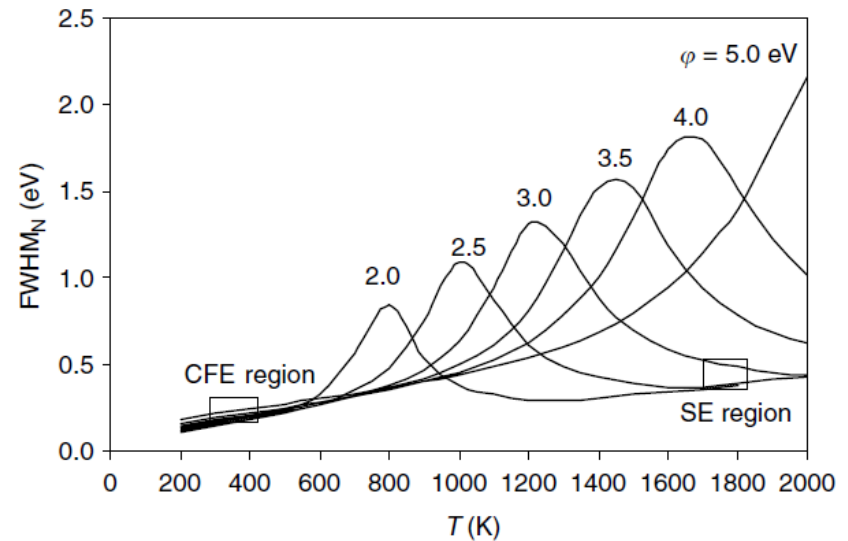
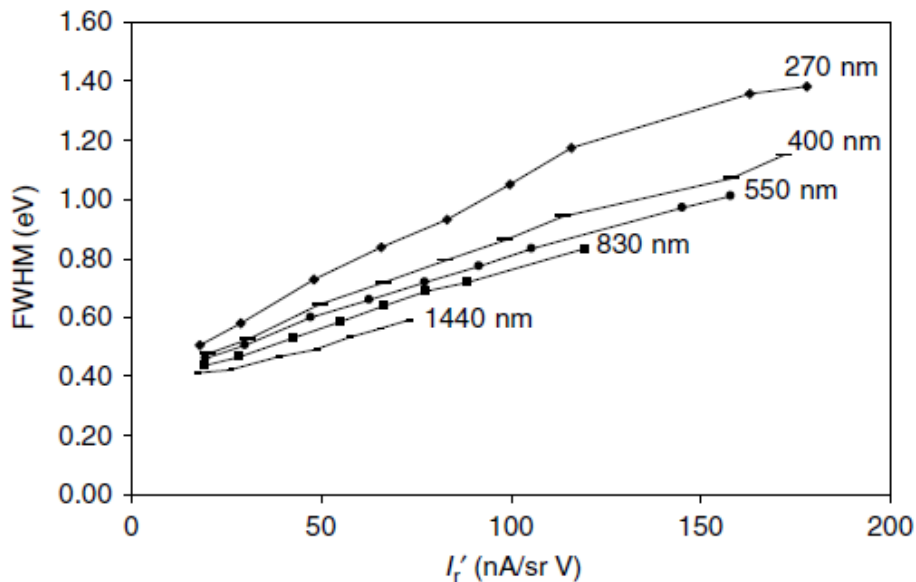
Alter: D.W. Tuggle et al., J. Microscopy 140 (1985) 293



# Total energy distribution

Energy spread depends on

- **Work Function (WF)**
- Tip temperature
- Reduced angular intensity  $I'/V_{ext}$
- Tip radius
- Coulomb interactions





# Current fluctuations

## Short-term ( $< 0.01$ Hz)

- local E field (random motion of atoms on the surface,  $i^+$  and  $n_0$  bombardment)
- local WF fluctuations (adsorption/desorption of gases, local  $ZrO_x$  concentration fluctuations)

## Long-term ( $> 0.1$ Hz)

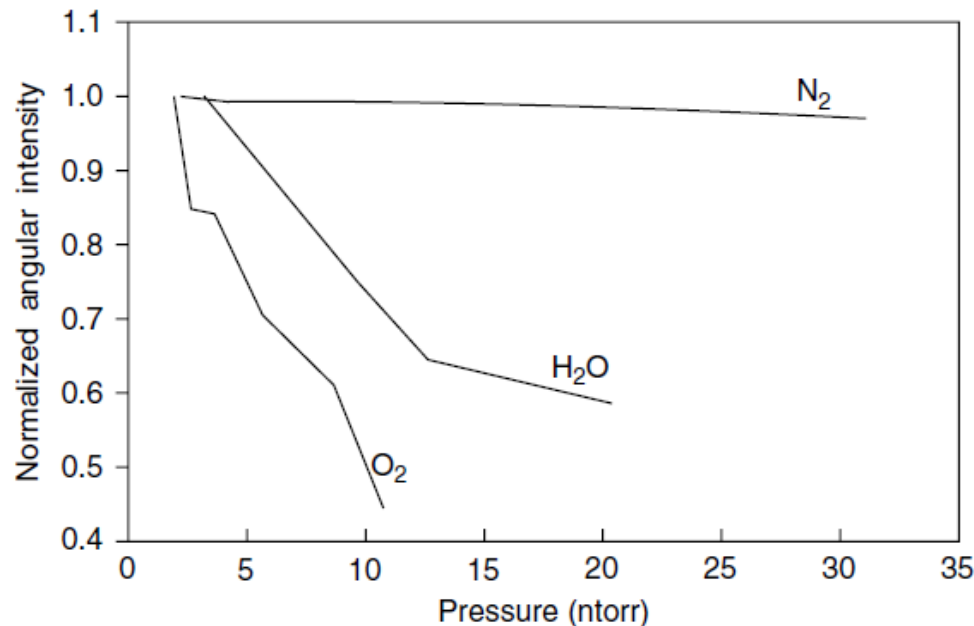
- macroscopic diffusion of W atoms, tip end-form variations (e.g. ring collapse)
- macroscopic E field and WF variations due to adsorption and desorption of gases

## Current fluctuations impacted by

- emitter size
- emitter temperature
- vacuum level
- angular intensity

# Residual gases

- Vacuum level is primary factor influencing emission characteristics
- Bake out → ideally  $1\text{e-}8$  Pa
- Electron stimulated desorption from oxidized surfaces
- Oxygen poisoning is reversible within operation range

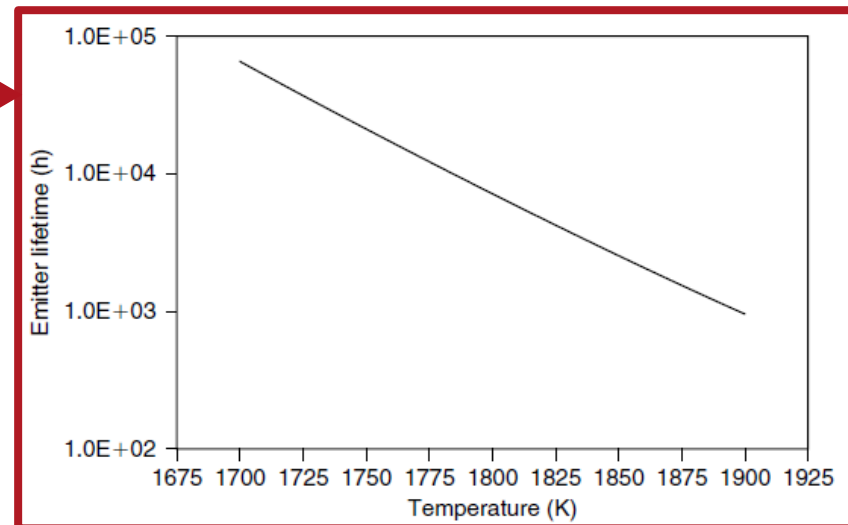


# Lifetime

- Tip **dulling**
  - Brightness decreases
  - required Vext gets too high
- high temperature:
  - dangerous for ZrO supply
  - tip grows faster (tip dulling)
- temperature increases during life time - best solution is periodic field adjustments → up to 4 years lifetime feasible

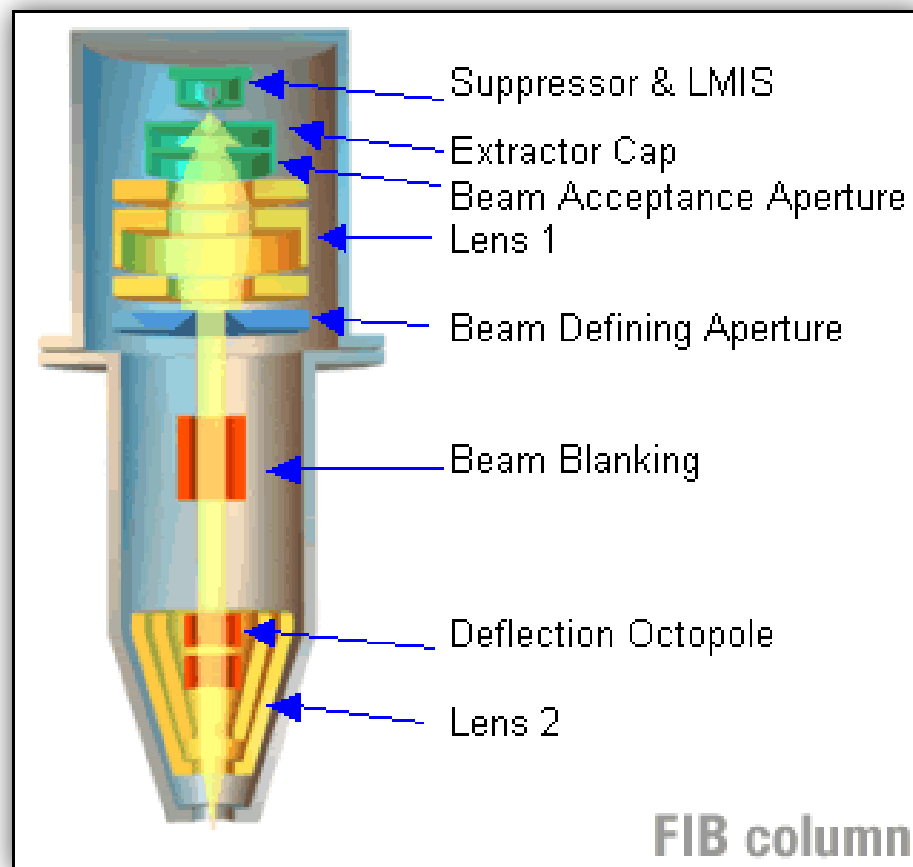
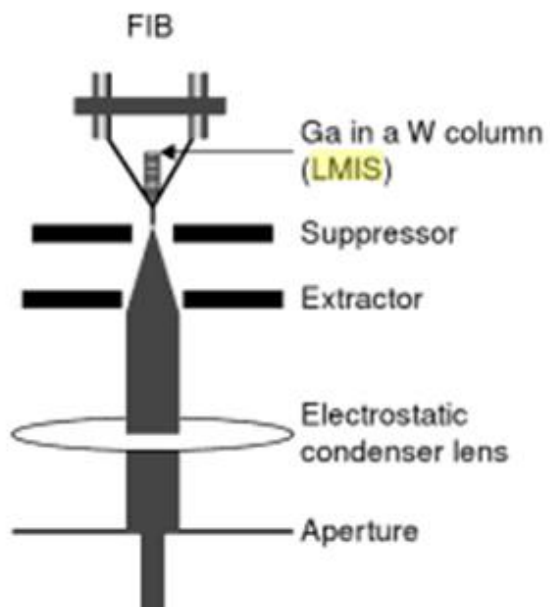
~ 2 years  
up to 4 years feasible

- natural life termination:  
**evaporation** of ZrO<sub>2</sub> reservoir



# Iontové zdroje

- Liquid Metal Ion Sources (LMIS)
- Gas Field Ionization Sources (GFIS)
- Plasma Sources



# Properties of “Perfect” Ion Source

1. Small virtual source size ( $dv$ )
  2. High angular current density ( $I'$ ) on optical axis
  3. Low energy spread ( $dE$ )
  4. Low beam noise (0-100 kHz)
  5. Stable emission; short term (hrs) and long term (weeks)
  6. Limited processing “events” required during operation
  7. Long life time
  8. Operate in modest vacuum
  9. Multiple ion species available
- } Reduced Brightness ( $B_r$ )

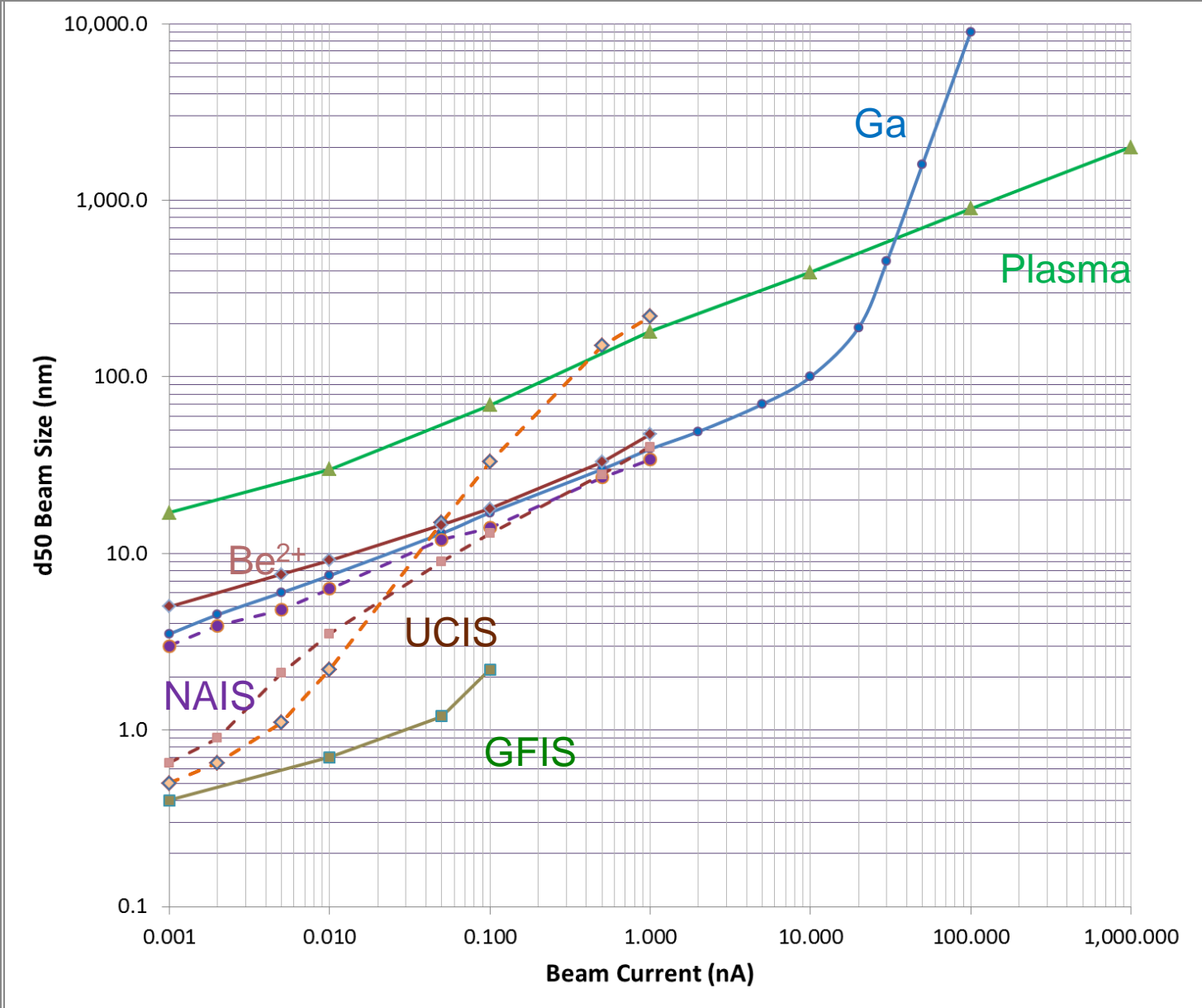
	1 pA Beam Current		1 nA Beam current		Beam current	Ions Available
	$B_r$	$dE_{FWHM}$	$B_r$	$dE_{FWHM}$		
Units	A/m <sup>2</sup> -sr -V	eV	A/m <sup>2</sup> -sr - V	eV	pA	
Ga LMI	10 <sup>6</sup>	5	10 <sup>6</sup>	5	1-60,000	Ga
Other LMIS	10 <sup>3</sup> - 10 <sup>6</sup>	5-20	10 <sup>3</sup> - 10 <sup>6</sup>	5-20	1- 60,000	Many metals
GFIS	5 x 10 <sup>9</sup>	0.4	NA	NA	1-5	He, Ne
NAIS	5 x10 <sup>6</sup> - 10 <sup>7</sup>	<0.5	< 10 <sup>6</sup> ?	< 0.5 ?	1 - 2000	All gases
Li MOTIS	10 <sup>3</sup>	<0.5	NA	NA	5 - 80	Column 1
“2D” UCIS	10 <sup>6</sup> - 5x10 <sup>8</sup>	<0.5	10 <sup>4</sup> - 5x10 <sup>6</sup>	< 1.0 ?	1 - 1000	Column 1
Plasma	10 <sup>4</sup>	5-7	10 <sup>4</sup>	5-7	1 - 10 <sup>6</sup>	All gases

- Single element LMIS
- Alloy LMI
- GFIS
- NAIS and Plasma
- MOT and UCIS

1 IA																	18 VIII A
1 1s	2 II A											13 III A	14 IV A	15 V A	16 VI A	17 VII A	2 He helium 4.003
3 Li lithium 6.941	4 Be beryllium 9.012											5 B boron 10.81	6 C carbon 12.01	7 N nitrogen 14.01	8 O oxygen 16.00	9 F fluorine 19.00	10 Ne neon 20.18
11 Na sodium 22.99	12 Mg magnesium 24.31	3 III B	4 IV B	5 V B	6 VI B	7 VII B	8 VIII B	9 VIII B	10 VIII B	11 I B	12 II B	13 Al aluminum 26.98	14 Si silicon 28.09	15 P phosphorus 30.97	16 S sulfur 32.07	17 Cl chlorine 35.45	18 Ar argon 39.95
19 K potassium 39.10	20 Ca calcium 40.08	21 Sc scandium 44.96	22 Ti titanium 47.87	23 V vanadium 50.94	24 Cr chromium 52.00	25 Mn manganese 54.94	26 Fe iron 55.85	27 Co cobalt 58.93	28 Ni nickel 58.69	29 Cu copper 63.55	30 Zn zinc 65.41	31 Ga gallium 69.72	32 Ge germanium 72.64	33 As arsenic 74.92	34 Se selenium 78.96	35 Br bromine 79.90	36 Kr krypton 83.80
37 Rb rubidium 85.47	38 Sr strontium 87.62	39 Y yttrium 88.91	40 Zr zirconium 91.22	41 Nb niobium 92.91	42 Mo molybdenum 95.94	43 Tc technetium 98	44 Ru ruthenium 101.1	45 Rh rhodium 102.9	46 Pd palladium 106.4	47 Ag silver 107.9	48 Cd cadmium 112.4	49 In indium 114.8	50 Sn tin 118.7	51 Sb antimony 121.8	52 Te tellurium 127.6	53 I iodine 126.9	54 Xe xenon 131.3
55 Cs cesium 132.9	56 Ba barium 137.3	57 La lanthanum 175.0	58 Hf hafnium 178.5	59 Ta tantalum 180.9	60 W tungsten 183.8	61 Re rhenium 186.2	62 Os osmium 190.2	63 Ir iridium 192.2	64 Pt platinum 195.1	65 Au gold 197.0	66 Hg mercury 200.6	67 Tl thallium 204.4	68 Pb lead 207.2	69 Bi bismuth 209.0	70 Po polonium 209	71 At astatine 210	72 Rn radon 222
87 Fr francium 223	88 Ra radium 226	71 Lu lutetium 175.0	72 Hf hafnium 178.5	73 Ta tantalum 180.9	74 W tungsten 183.8	75 Re rhenium 186.2	76 Os osmium 190.2	77 Ir iridium 192.2	78 Pt platinum 195.1	79 Au gold 197.0	80 Hg mercury 200.6	81 Tl thallium 204.4	82 Pb lead 207.2	83 Bi bismuth 209.0	84 Po polonium 209	85 At astatine 210	86 Rn radon 222
† 5f	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d	† 5d
† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d	† 6d
† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f	† 4f
† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f	† 5f
lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)	lanthanides (rare earth metals)
actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides	actinides

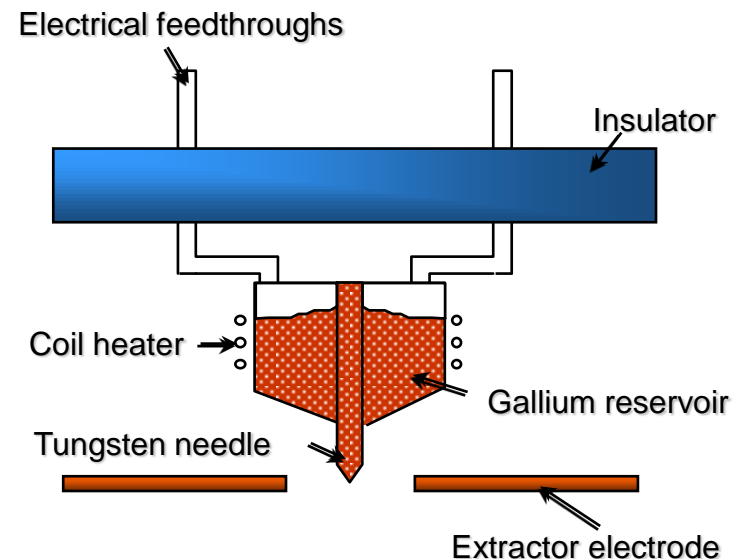
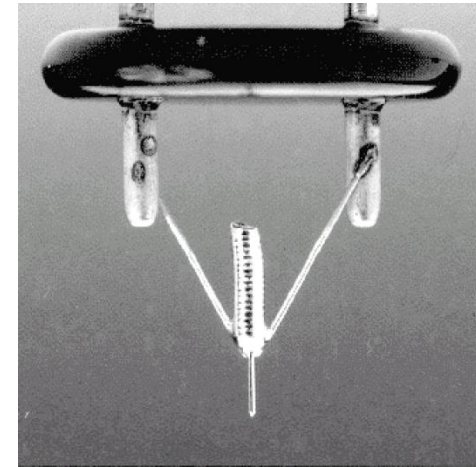


# Column Performance Comparison

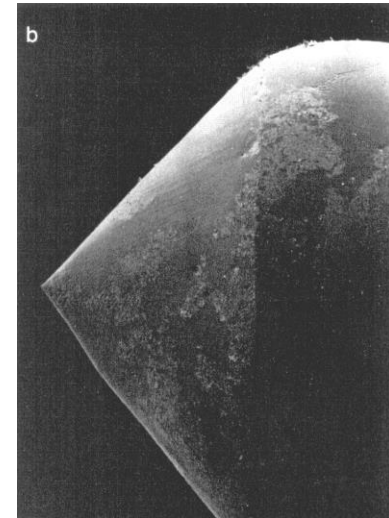
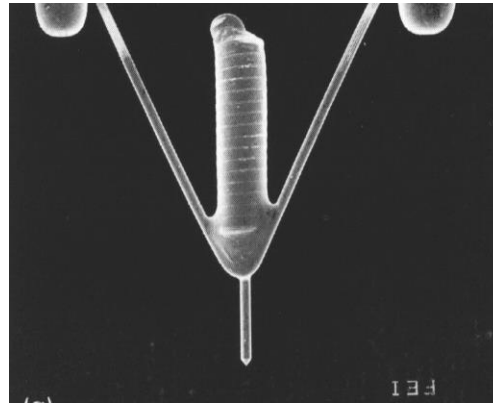
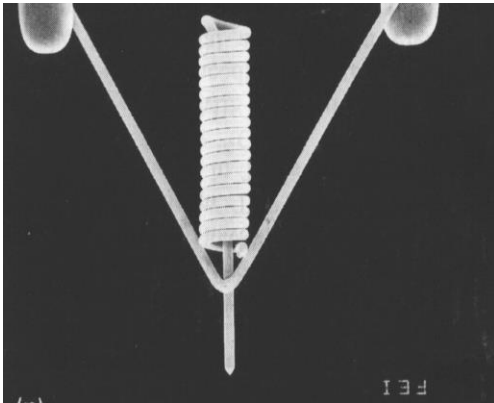


# 1. LMIS

1. Galium je umístěno v zásobníku (spirála). Po ohřátí dojde ke smočení W hrotu (poloměr 2-5  $\mu\text{m}$ ). Za přítomnosti elektrostatického pole ( $10^8 \text{ V/cm}$ ) dojde k vytvoření kužele s vrcholovým poloměrem 2-5 nm **Taylor cone**.
2. Intenzita pole v blízkosti takto vytvořeného hrotu je dostatečně vysoká k vytržení a ionizaci molekul galia.



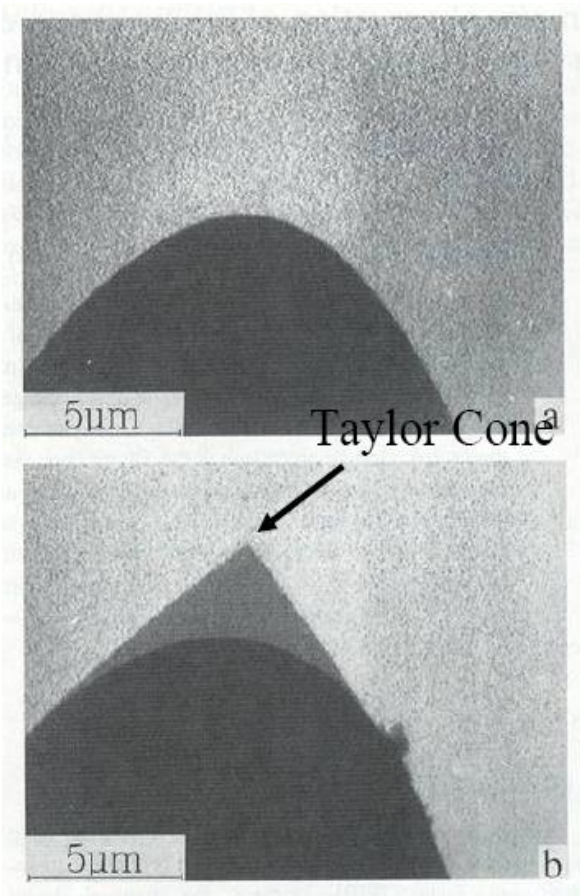
# Liquid Metal Ion Source:



The tungsten is cleaned and wetted with gallium which is held in the spiral by surface tension. The vapour pressure is about  $2 \times 10^{-40}$  mbar.

Frozen-in  
-shape LMIS  
showing  $49^\circ$  half angle. The field emission area is a 2-5nm across giving current densities  $>10^8$  Acm<sup>-2</sup>.

# Taylor cone



LMIS emitter substrate  
with AuGe Taylor cone

*W. Driesel, C. Dietzsch, R. Muhle, J. Vac.  
Sci. Technol. B14, 3367 (1996)*

# Desired Properties for LMIS material

1. Low temperature melting point
2. Low vapor pressure at the melting point
3. Slow to oxidize when in liquid phase
4. Non-reactive in liquid phase
5. Wets materials which can easily be fabricated into substrate emitter

## **Gallium: ideal material**

- ✓ Ga melting point 29.8 C but boiling point is 2204 C
- ✓ Vapor pressure at 30 C is only  $10^{-21}$  mm Hg and at 450 C is  $10^{-11}$  mm Hg!!!
- ✓ Non reactive in the liquid state
- ✓ When molten – super cools so stays liquid at room temperature
- ✓ Wets tungsten easily and is non-reactive with tungsten below 800 C.
- ✓ Moderately heavy mass providing a reasonable sputter rate for milling.

# Liquid metal field ionisation sources

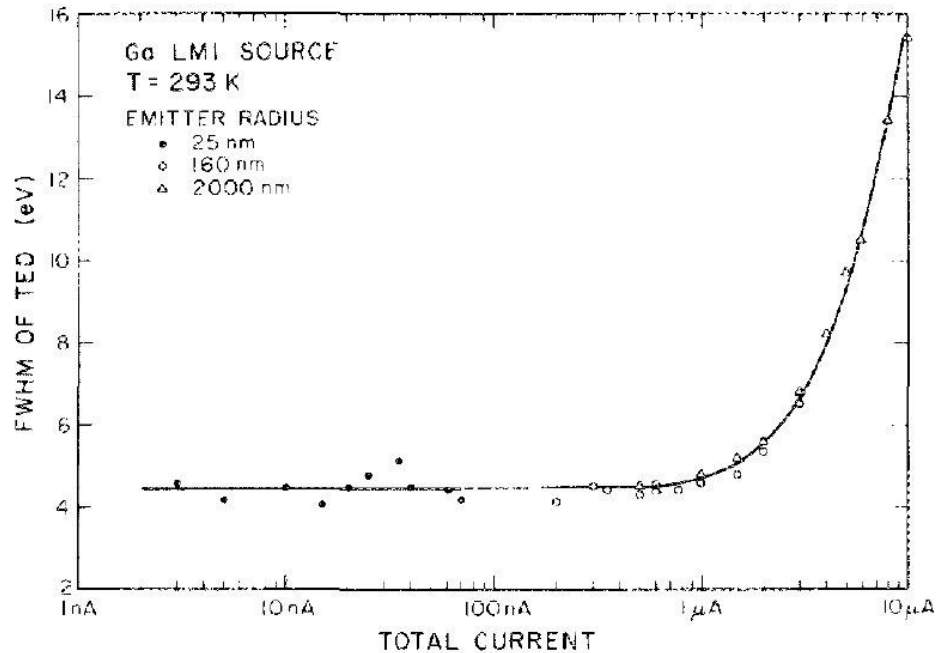
H																	He	
Li	Be											B	C	N	O	F	Ne	
Na	Mg											Al	Si	P	S	Cl	Ar	
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr	
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe	
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn	
Fr	Ra	Ac	Unq	Unp	Unh													
			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu		
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr		

Physical and chemical properties of Gallium  
(Form: Solid; Colour: Silver-colour; Odour: Odourless)

Melting Point, °C	Boiling Point, °C	Density, g/cm <sup>3</sup>
29.78	2403	5.907

Until now, the following LMIS have been produced and studied: Ga, Sn, In, Au, AuSi, AuGe, AuCo, CoGe, CoY, CuGe, CuMg, AlGe, GaIn, AuCoGe, AuCoY, AuSiPr, AuSiBe, AuCoPr, AuCoSi, AuErSi. The most commonly used ion is Gallium since it has the longest liquid range of any metal (from 29.8°C to 2175°C) providing room temperature operation and yields a long lifetime source. Gallium can be focused to a very fine probe size (< 10 nm in diameter). Liquid metal Gallium is high vacuum compatible and Gallium is large ions for physical sputtering. Below the melting point Gallium is a soft, silver white metal that is stable in both air and water.

Current range	
> 25 $\mu\text{A}$	nestabilní emise
2 - 25 $\mu\text{A}$	stabilní emise
0.45 - 2 $\mu\text{A}$	pulsní režim
< 0.45	nestabilní emise, kolaps

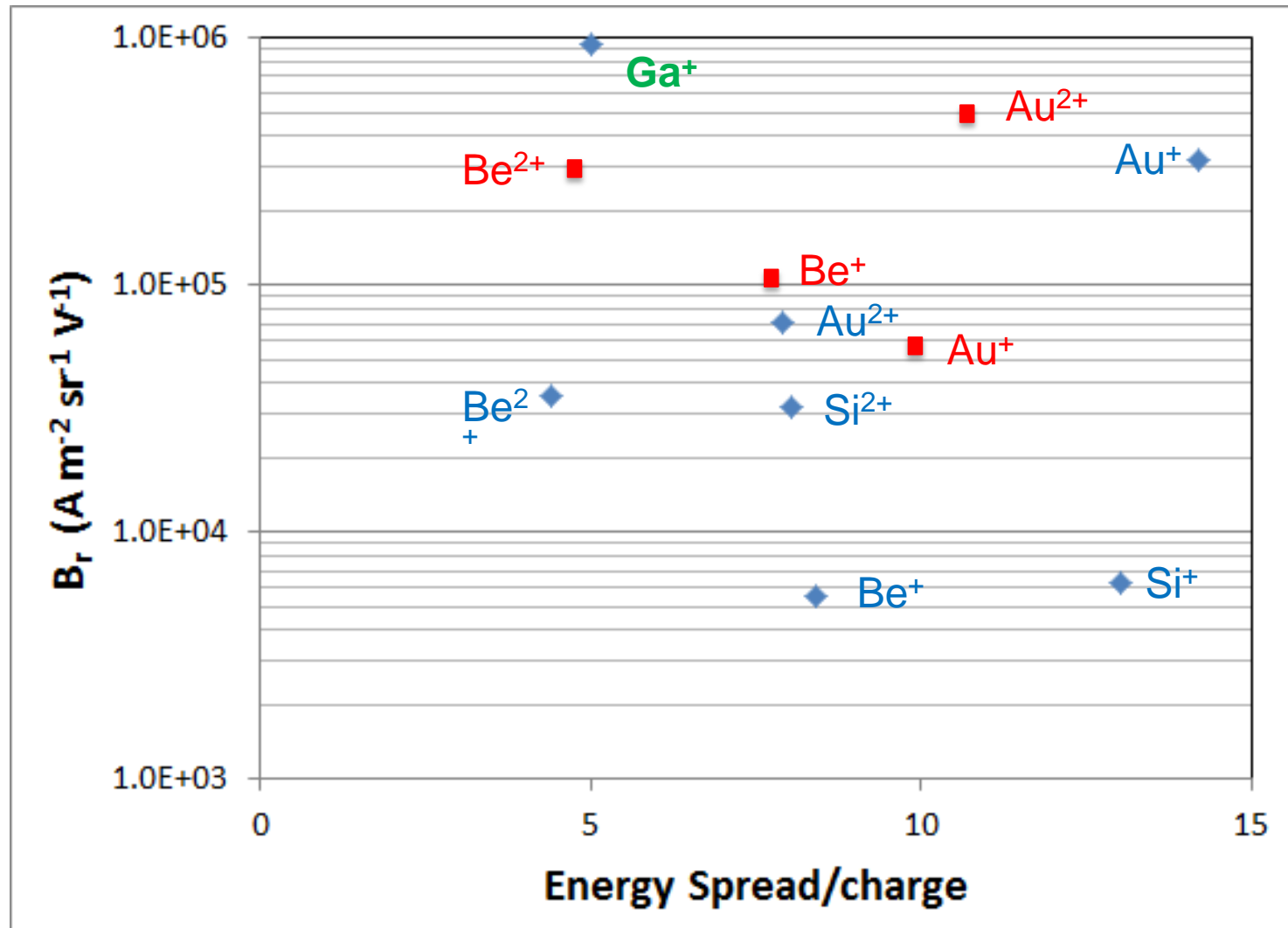


The energy spread of Ga LMIS over the current range 3 nA – 10  $\mu\text{A}$ .

(Bell AE., Rao K., Schwind GA., Swanson LW., J Vac Sci Technol., B6(3), 1988)

- zdroj udržován na spodním okraji režimu stabilní emise ~ 2  $\mu\text{A}$  změnou intenzity extrakčního pole
- ohřev galia pouze na znovuoobnovení Taylorova kužele (cca po několika desítkách hodin provozu)

# $B_r$ versus $dE/e$ – Alloy sources



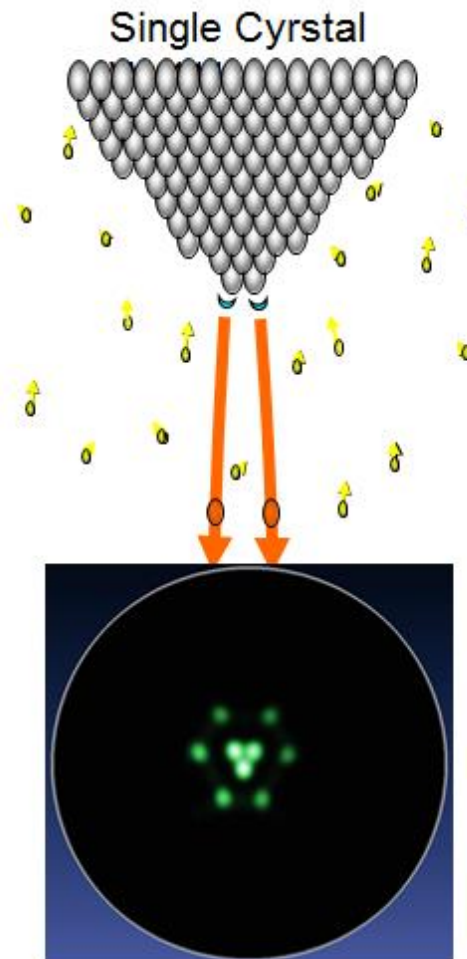
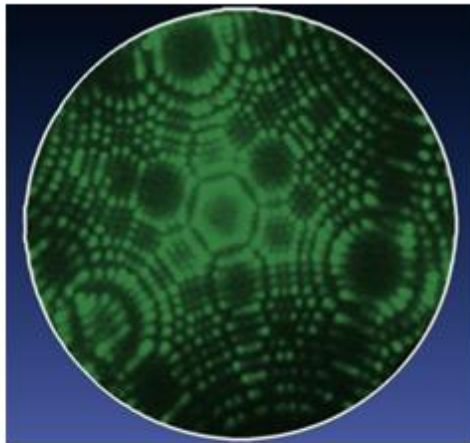
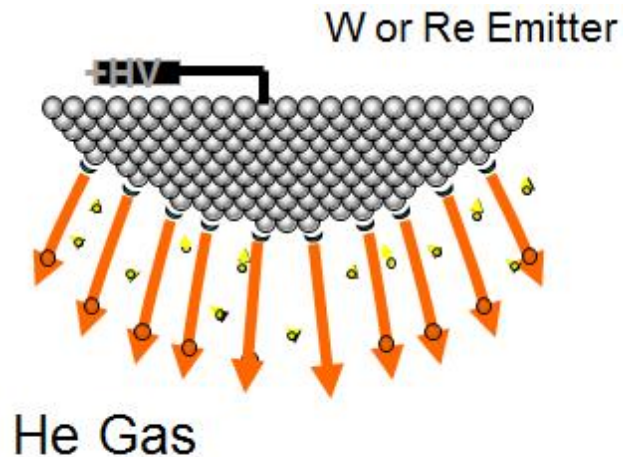
  $\text{Au}_{62}\text{Si}_{23}\text{Be}_{15}$  Source

  $\text{Au}_{60}\text{Be}_{40}$



## 2. Gas field ionization sources

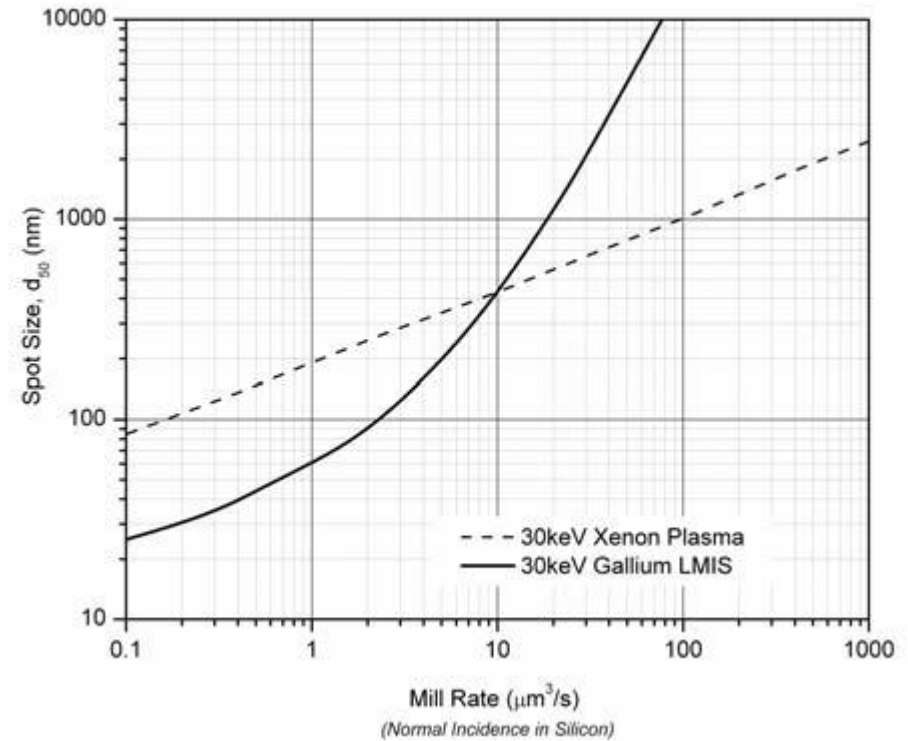
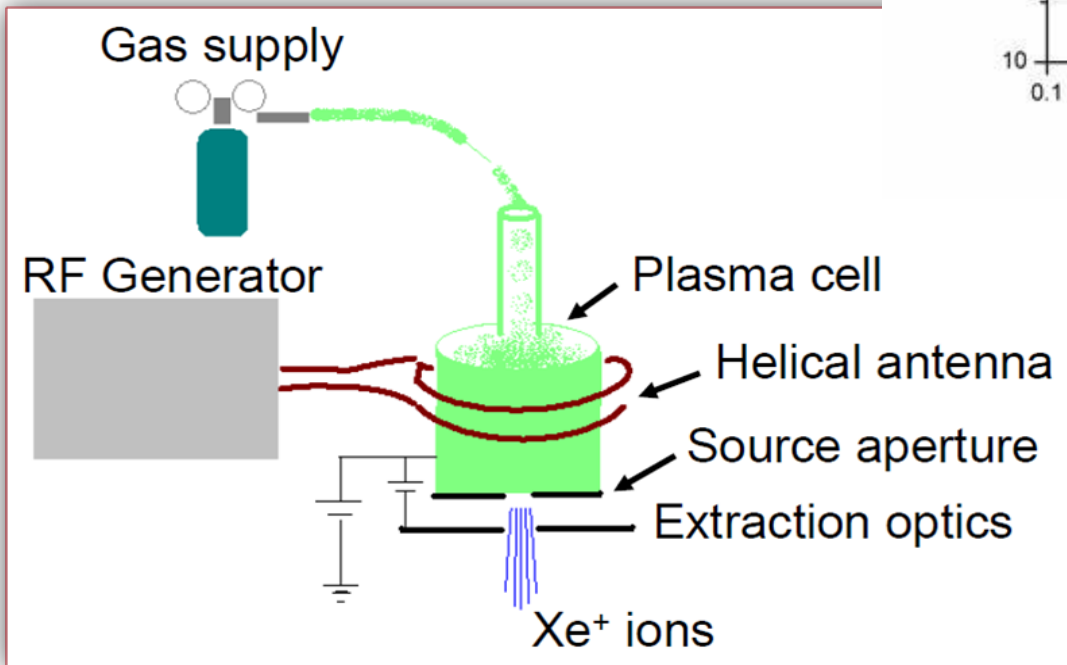
surface field ionization of gas atoms at the tip of a metal needle



# GFIS - considerations

1. Incredible Br but has a lot of limitations: species, current, complicated
2. Virtual Source size  $< 0.25$  nm,  $Br \approx 1-5 \times 10^9$  A/m<sup>2</sup>-sr –V  
Angular Intensity: 0.25 – 0.5 uA/sr
3. Not a source for milling – clearly best ion source for imaging
4. Maximize current you want to column magnification (M) to be high: If M is high then source mechanical vibration becomes an issue.
5. High column magnification means gun lens aberrations become more important
6. Limited to He and Ne commercially. Light mass ions have very low sputter rates.
7. Very low beam currents  $< 5$  pA .
8. Challenging source environment requirements: UHV ( $< 10^{-9}$  torr), low temperature ( $< 80$ K), and extremely pure gas supply.

# Plasma source



# Plasma sources - considerations

1. Extremely large angular intensity  $I' = 50 \text{ mA/sr}$  ( $Ga = 20 \text{ uA/sr}$ )
2. Very large virtual source size:  $15 \text{ um}$  ( $Ga = 50 \text{ nm}$ )
3. Very large de-magn is required to get the smaller beam sizes which means it is challenging to get high beam currents into smaller beam spot sizes.
4. There are a significant amount of neutrals in beam – column may need a bend to keep neutrals from reaching the sample
5. Wide variety of different ions; all natural elemental gases including mixed gases which would allow fast switching between different gases with use of a mass filter.
6. Gas type is easy to change: same source can produce many different ions.
7. Beam of molecular single element will contain multiple peaks ( $O^+$ ,  $O_2^+$ ) therefore a mass filter is required if only one ion specie is desired.
8. Extraction elements/optics has significant effect on brightness and dE

# Použitá literatura

- [1] Handbook of charged particle optics / edited by Ion Orloff - 2<sup>nd</sup> ed., ISBN : 978-1-4200-4554-3
- [2] Reimer L., Scanning Electron Microscopy - Physics of Image Formation and Microanalysis, ISBN: 3-540-63976-4
- [3] Williams D., Carter B., Transmission Electron Microscopy, vol. I, ISBN: 0-306-45247-2
- [4] Karlík M., Úvod do transmisní elektronové mikroskopie, ISBN: 978-80-01-04729-3
- [5] Smith N.S. et al., A High Brightness Plasma Source for Focused Ion Beam Applications, Microsc. Microanal. 13 (Suppl 2), 2007
- [6] Dahl P., Introduction to Electron and Ion Optics, ISBN 0-12-200650-X
- [7] Greg Schwind (FEI), CPO-9 Brno conference presentation on ion sources