

# Autoemitters and sensors based on CNT experimental researches.

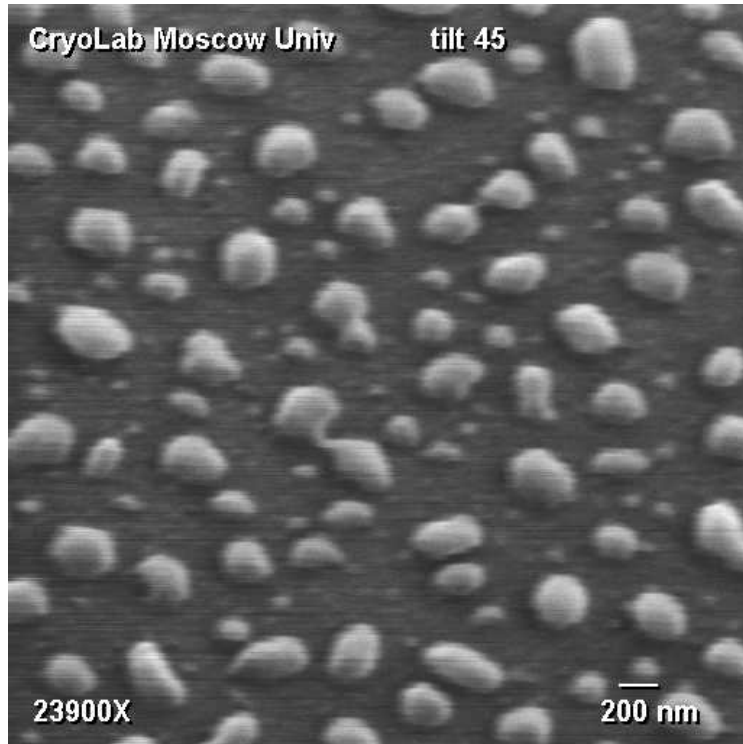
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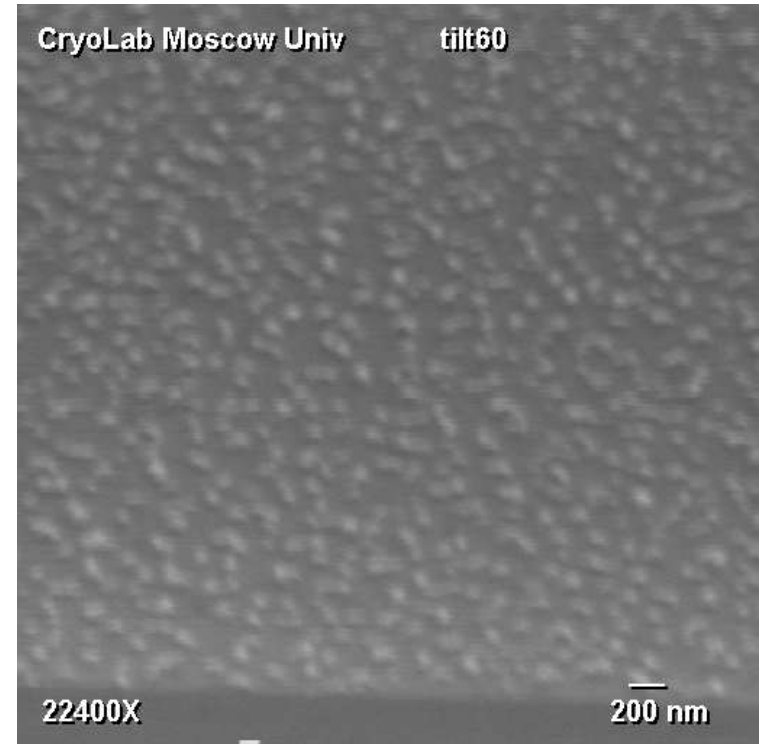
# Agenda

- Growth mechanism
  - metal film melting and correlation of its thickness with CNT diameters
  - growth modes
- Experimental conditions and methods
  - growing plant
  - measurement stand
- Experimental results
  - Tables with emission parameters
  - Tables with emission current degradation parameters
- Analysis of obtained results
  - Emission current noise problem
  - The way to solve noise problem (lateral autoemitter construction)
  - Emission current degradation problem
- Conclusion

# Growth mechanism.



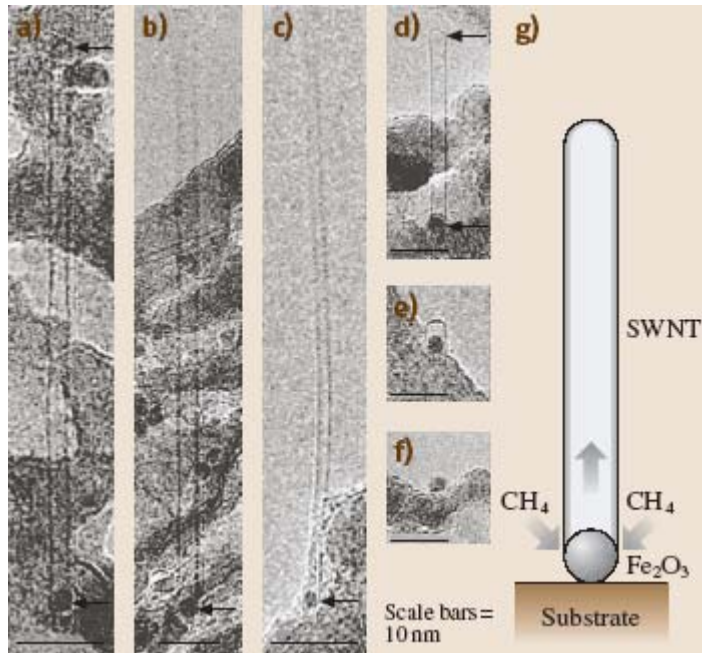
a)



b)

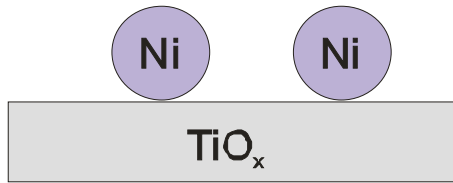
The catalyst after annealing in hydrogen plasma.  
Nickel layer thickness a)  $\sim 100$  nm, b)  $\sim 20$  nm.

# Growth mechanism.

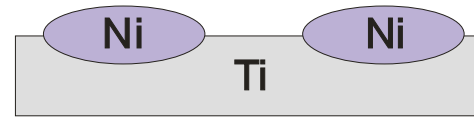


- High resolution transmission electron microscopy images of several SWNTs grown from iron-based nanoparticles by CVD method, showing that particle sizes determine SWNT diameters in that case.

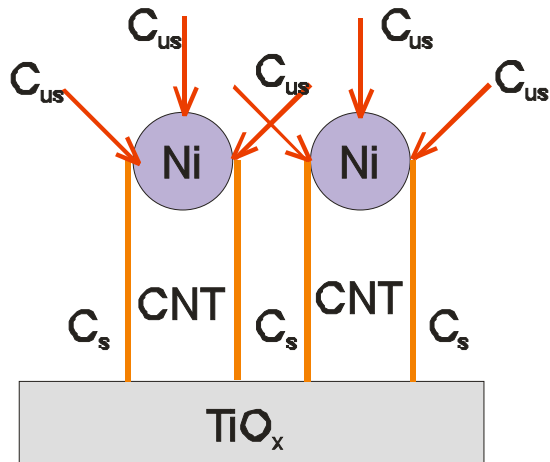
# Growth mechanism.



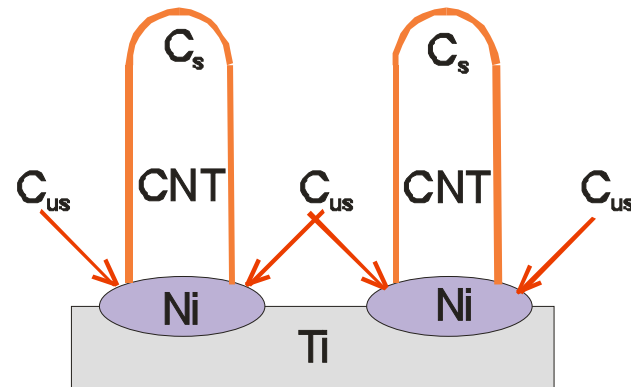
a)



b)



c)



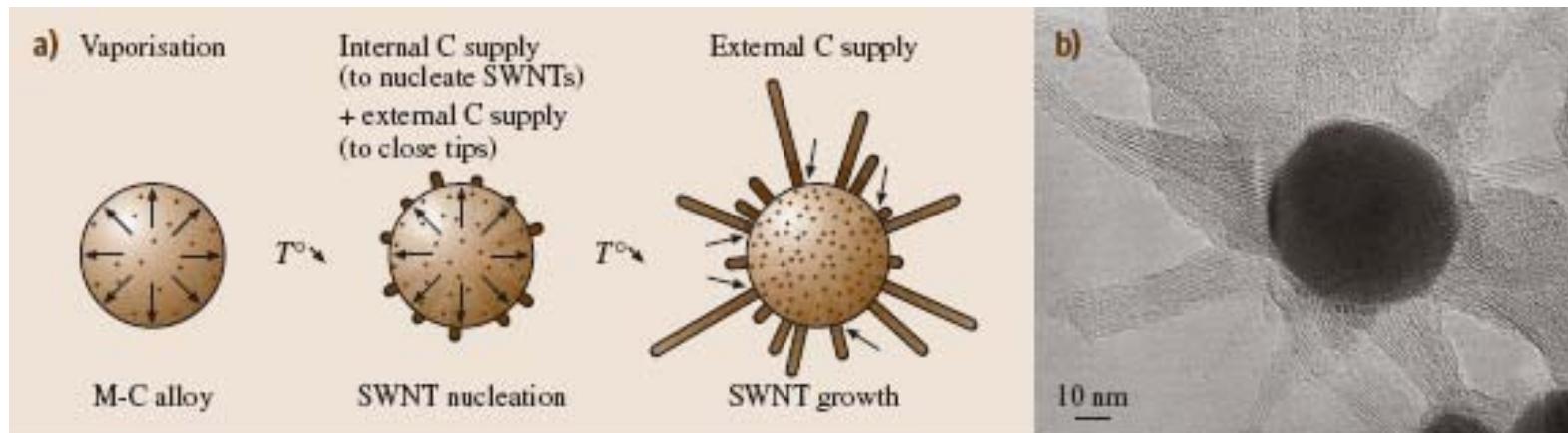
d)

# Growth mechanism.

Guidelines indicating the relationships between possible carbon nanofilament morphologies and some basic synthesis conditions

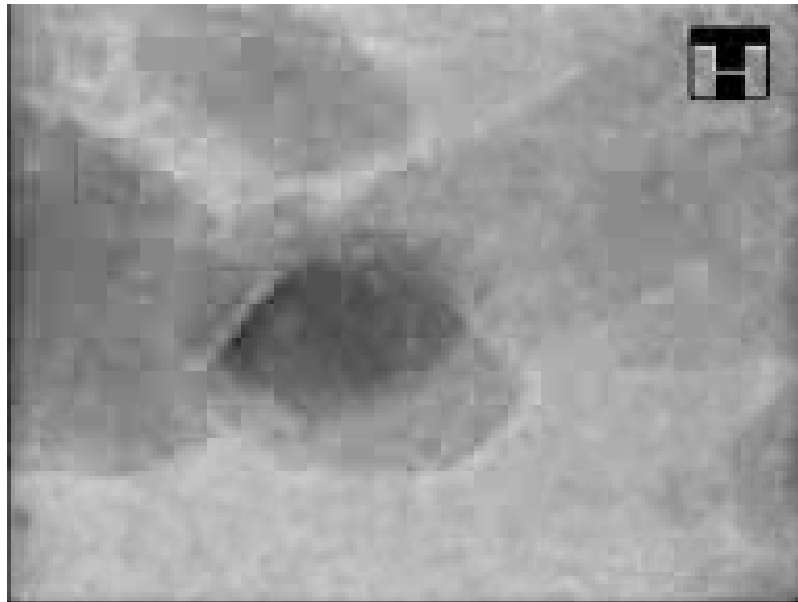
		Increasing temperature ... ... and physical state of catalyst			Substrate		Thermal gradient	
		Solid (crystallized)	Liquid from melting	Liquid from clusters	Yes	No	Low	High
Catalyst particle size	<~ 3 nm	SWNT	SWNT	?	base-growth			
	>~ 3 nm	MWNT (c,h,b) platelet nanofiber (heterogeneous related to catalyst particle size)	c-MWNT	SWNT	tip-growth	tip-growth	long length	short length
Nanotube diameter								
Nanotube/particle		one nanotube/particle		several SWNTs/particle				

# Growth mechanism.



(a) Mechanism proposed for SWNT growth. (b) Transmission electron microscopy image of SWNT growing radial to a large Ni catalyst particle surface in the electric arc experiment.

Video file. Nanotube or nanofilament growth.





## Growth modes.

- “Low temperature” mode, in which carbon-bearing gas is injecting into the chamber previously heated up to the 530 degrees centigrade. The additional heating is not carrying out.
- “High temperature” mode, which concludes in that the work chamber heating up to the 580 degrees centigrade and higher. And additional heating is carrying out after the working gas has been injecting, to compensate its cooling effect.

# Growing plant

- Chamber with forvacuum eviction ability
- Working gas injection system
- UHF plasma
  
- Substrate: silicon, sapphire, polikor
- Catalyst: nickel
- Buffer layers: titanium, vanadium

A fragment of the measurement stand (vacuum system, the management block).



# Autoemission parameters measured

- threshold voltage  $E_{thv}$  (given in the tables in corrected to the micrometers value)
- threshold current  $I_{thv}$  (the starting autoemission current)
- maximum emission current  $I_{max}$
- medium emission current  $I_{med}$  (current that suites to the stable emission current)

The best values of autoemission parameters are represented

№	structure	T (°C)	t (min)	Pressure (atm.)	resume
K13	Ni on polikor	600	20	0.3 -0.5	$E_{thv}=3,5 \text{ V}/\mu\text{m}$ ; $I_{max}=50\mu\text{A}$
K15(2)	Ni(15) on polikor	433 – 532	20	0.9 -0.7	$E_{nop}=4,24 \text{ V}/\mu\text{m}$ $I_{max}=31\mu\text{A}$
K17	Ni(30)V(20)Ti(40) on Si	485-531	20	0.9 -0.8	$E_{thv}=3,03 \text{ V}/\mu\text{m}$ ; $I_{max}=25\mu\text{A}$
K18	Ni(30)V(20)Ti(40) on Si	474-532	40	0.8	$E_{nop}=2,2 \text{ V}/\mu\text{m}$ $I_{max}=22\mu\text{A}$
K19	Ni(30) Ti(40) on Si	485-532	40	0.7	$E_{nop}=3,67 \text{ V}/\mu\text{m}$ $I_{max}=23,5\mu\text{A}$
K23	Ni on sapphire	649-614	15	0.9	$E_{nop}=1,54 \text{ V}/\mu\text{m}$ $I_{max}=6,5\mu\text{A}$
K28(1)	Ni(40)V(20)Ti(30) on Si	531	60	0.7	$E_{nop}=3,23 \text{ V}/\mu\text{m}$ $I_{max}=10\mu\text{A}$
K32	Ni(30) Ti(40) on Si	625	10	0.9	$E_{nop}=10,75 \text{ V}/\mu\text{m}$ $I_{max}=40\mu\text{A}$

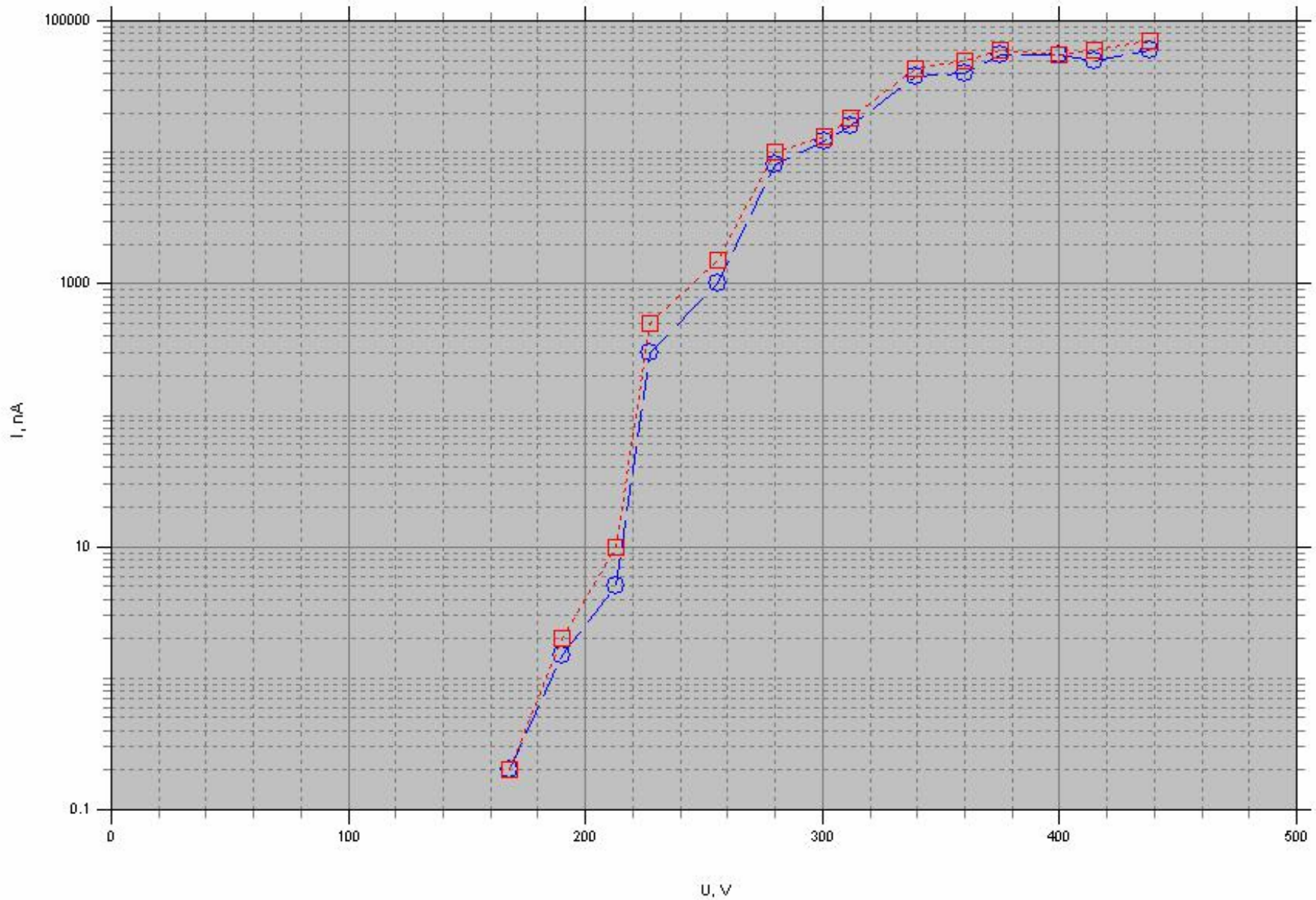
Here are the worst emission parameters for compare

№	structure	T (°C)	t (min)	Pressure (atm.)	resume
K5	Ni(30)V(20)Ti(40) on Si	628...606	3	0,9	no emission
K14	Ni on polikor	450 - 550			no emission
K21	Ni(30) V(20)Ti(40) on Si	660-648	20	0,9	no emission
K22	Ni on sapphire	649-626	20	0,85	no emission
K25	Ni on Si	649-623	5	0.94	$E_{thv} = 8,29 \text{ V}/\mu\text{m}$ , $I_{thv} = 200\text{nA}$ $I_{max} = 0,7 \mu\text{A}$
K27(1)	Ni V on Si	542-507	60	0.8	no emission
K29(1)	Ni on Si	531	20	0.9	no emission
K29(2)	Ni on Si	531	20	0.9	$E_{thv} = 18 \text{ V}/\mu\text{m}$ , $I_{thv} = 7\text{nA}$ $I_{max} = 8 \mu\text{A}$
K34	Ni(30)V(20)Ti(40) on Si	590	20	0.9	no emission

## Medium voltage and current values

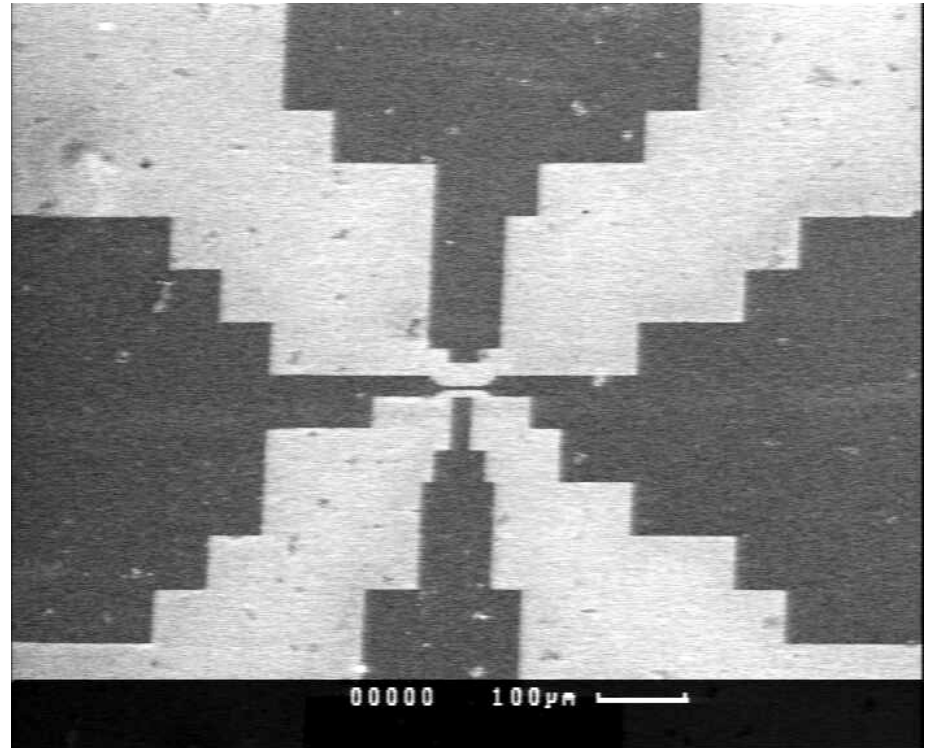
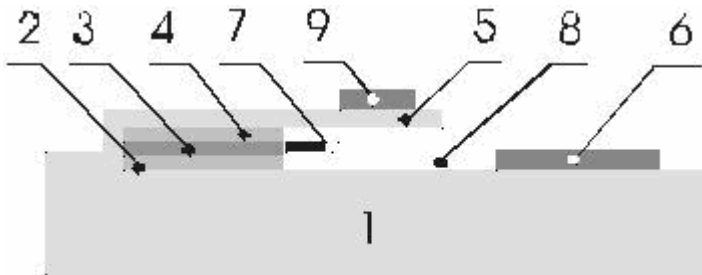
№	structure	T (°C)	t (min)	Pressure (atm.)	resume
K15(1)	Ni(15) on polikor	433 - 532	20	0.9	$E_{thv} = 3,92 \text{ V}/\mu\text{m}$ $I_{thv} = 60 \text{ nA}$ $I_{max} = 18 \mu\text{A}$ $I_{med} = 8 \mu\text{A}$
K16	Ni(30)Ti(40) on Si	485-520	10	0.8 - 0.9	$E_{thv} = 5,58 \text{ V}/\mu\text{m};$ $I_{thv} = 363 \text{ nA}$ $I_{max} = 15 \mu\text{A},$ $I_{med} = 7 \mu\text{A}$
K20	Ni(30)Ti(40) on Si	648-660	20	0.9	$E_{thv} = 5,31 \text{ V}/\mu\text{m};$ $I_{thv} = 425 \text{ nA}$ $I_{max} = 0,8 \mu\text{A},$ $I_{med} = 0,4 \mu\text{A}$
K24	Ni on Si	649-614	10	0.9	$E_{thv} = 4,44 \text{ V}/\mu\text{m};$ $I_{thv} = 333 \text{ nA}$ $I_{max} = 5 \mu\text{A},$ $I_{med} = 2 \mu\text{A}$
K26(2)	Ni(30)V(20)Ti(40) on Si	543-508	20	0.8	$E_{thv} = 4,53 \text{ V}/\mu\text{m};$ $I_{thv} = 400 \text{ nA}$ $I_{max} = 10 \mu\text{A},$ $I_{med} = 2 \mu\text{A}$
K27(2)	Ni(30)V(20)Ti(40) on Si	542-507	60	0.8	$E_{thv} = 8 \text{ V}/\mu\text{m};$ $I_{thv} = 300 \text{ nA}$ $I_{max} = 9 \mu\text{A},$ $I_{med} = 5 \mu\text{A}$
K30(2)	Ni on Si	590	15	0.9	$E_{thv} = 11,68 \text{ V}/\mu\text{m};$ $I_{thv} = 120 \text{ nA}$ $I_{max} = 7 \mu\text{A},$ $I_{med} = 3 \mu\text{A}$
K33	Ni(30)Ti(40) on Si	590	20	0.9	$E_{thv} = 9,25 \text{ V}/\mu\text{m};$ $I_{thv} = 15 \text{ nA}$ $I_{max} = 15 \mu\text{A},$ $I_{med} = 5 \mu\text{A}$

# Emission current noise problem





# The way to solve noise problem (lateral autoemitter construction)



## Table with emission current degradation parameters

№	U, V	I, $\mu\text{A}$ (before)	$\Delta t$ , min	I, $\mu\text{A}$ (after)	Uthv, V/ $\mu\text{m}$	I <sub>max</sub> , $\mu\text{A}$
K15(2)	319	1,6 - 2,1	5	0,8 - 0,9	4,24	31
	791	28 - 31	3	0,00015		
K18	299	0,8 - 1,4	2	0,5 - 0,8	2,2	22
	750	7,0 - 15,0	2	5,0 - 10,0		
K19	502	18 - 18,5	30	23,0 - 22,0	3,67	23,5
K23	284	6,0 - 6,5	17	1,2 - 1,6	1,54	
	320	3,4	6	3,4		
K28(1)	328	2,5 - 3,0	5	1	3,23	10
	427	5,0 - 6,0	5	5,0 - 5,5		
	489	10	2	0,5		
K32	473	2,5 - 3,5	2	1,5 - 2	10,75	40
	701	20 - 25	15	18 - 21		
	782	25 - 27	10	13 - 14		

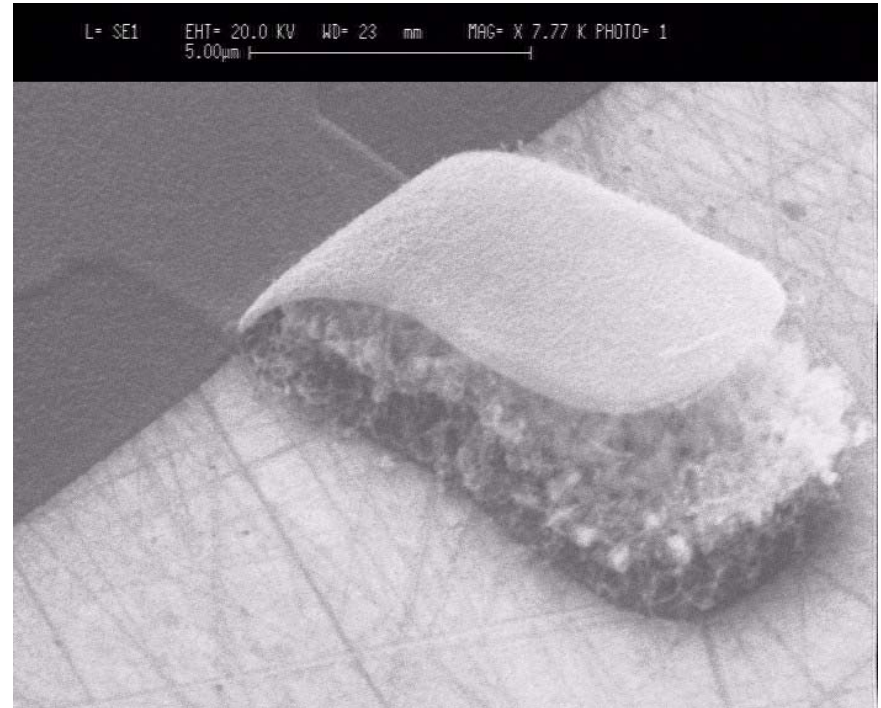
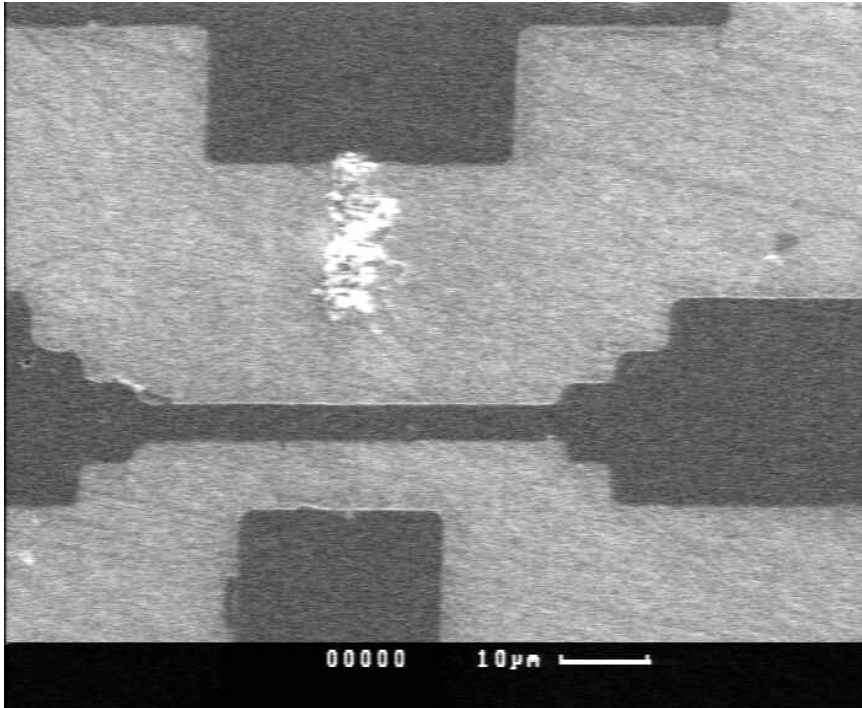
## Table with emission current degradation parameters

№	U, V	I, $\mu\text{A}$ (before)	$\Delta t$ , min	I, $\mu\text{A}$ (after)	$E_{\text{thv}}$ , V/ $\mu\text{m}$	$I_{\text{max}}$ , $\mu\text{A}$
K15(1)	465	10,0 - 11,0	5	8	3,92	18
K24	485	3	5	2	4,44	5
	570	2,5	10	1,3		
K26(2)	284	7.0	10	7.0	4,53	10
	476	3,0 - 3,2	2	1,8 - 2,0		
K27(2)	414	2,2 - 2,5	5	2,2 - 2,5	8	9
	492	8,0 - 9,0	30	5		
K30(2)	657	3	3	7	11,68	7
	659	7	60	0,3		
K33	400	6,5 - 7	60	5 - 5,5	9,25	15
	480	15	2	2		

# Nanotubes degradation



# Degradation of autoemitters



# Conclusion

- Two main problems (emission current noise and degradation) that was revealed during this work are related with technical realization. And the way to solve this problems is to improve technical equipment that we use and control each step and each parameter of the process from the beginning to the end.