September 1998

LA-SUB--99-3

REPORT

TO THE LOS ALAMOS NATIONAL LABORATORY

AT THE UNIVERSITY OF CALIFORNIA

AGREEMENT NUMBER: 4528 N 0004-35

TASK ORDER 004

CONTRACTOR: A.A.BOCHVAR ALL-RUSSIAN RESEARCH

INSTITUTE OF INORGANIC MATERIALS

PROJECT TITLE: **PROCESS OPTIMIZATION FOR ADVANCED HIGH CONDUCTIVITY - HIGH STRENGTH MATERIALS**

MASTER

PROJECT DURATION: OCTOBER , 1994 - OCTOBER, 1998

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ABSTRACT

On the basis of investigations carried out earlier, two types of high strength - high conductivity Cu-Nb wires have been designed and appropriate manufacturing processes have been proposed and experimentally approved. Long length conductors with rectangular cross sections 4x6 mm^2 and 2x3 mm^2 have been fabricated by the «in situ» process and by the «bundle and deform» process, which eliminates the operation of melting, accordingly.

Investigation on the microstructure of both types of the fabricated wires has been conducted. Mechanical properties and electrical conductivity parameters have been measured also.

INTRODUCTION

One of the two main objectives of this stage of work was to search for the possibility to enhance the strength of Cu-Nb microcomposite wires produced by the «bundle and deform» process, which eliminates the operation of melting. The wire with 2x3 mm^2 cross section has been designed and fabricated.

The second goal was to adjust and stabilize the previously developed «in situ» process by fabrication of 4x6 mm^2 Cu-Nb wire.

The ingots of Cu-18 wt.% Nb alloy were produced by consumable arc melting process described in details in [Report on Task Order 001].

At the previous stage of work [Report on task Order 003] we initiated the investigation on the ««bundle and deform» process. Several experimental Cu-Nb wires which contained different amounts of Nb filaments in the range of 2mln - 104 mln were fabricated. Mechanical and electric properties of these experimental wires were investigated.

It was shown that the decisive factor influencing on the achievement of high strength level in Cu-Nb microcomposite wires, fabricated by «bundle and deform» method, was the volume fraction of Nb in the first order strand. The volume fraction of Nb also be expressed in term of d_{Nb}/d_{Cu} ratio (where d_{Nb} and d_{Cu} - characteristic dimensions of Nb filaments and Cu matrix accordingly). It was experimentally found that decrease of d_{Nb}/d_{Cu} ratio for the wire with 39 mln Nb filaments in a factor of 1.5 comparing with the d_{Nb}/d_{Cu} ratio for the wire with 104 mln Nb filaments had resulted in the increase of maximum attainable UTS value. The Cu-Nb wire with 39 mln Nb filaments had exhibited UTS (RT) ≥ 1400 MPa. Some guidelines for further improvement of Cu-Nb wires produced by the «bundle and deform» method have been proposed. In particular the necessity to optimize the dimensions (d_{Nb}/d_{Cu} ratio) of first order composite billet has been proposed for attaining of the maximum extended surface of interphase Cu/Nb boundary.

Cu-Nb microcomposite «in situ» processed wires.

On the basis of the results obtained at the earlier stages of work [Reports on Task Orders 001, 002, 003] the following initial materials were chosen for fabrication of Cu-Nb alloys: - high purity copper (OFHC type - 99.99%) with $RRR_{4.2} \geq 200$ and electron beam melted high purity niobium. The composition of alloy was Cu-18wt.%Nb. Consumable arc melting process which was optimized earlier [Report on Task Order 002] provided the uniform distribution of Nb (in the range of ± 0.5 wt.%) in the ingots 100 mm in dia.

The ingots produced were machined and deformed by extrusion and consequent drawing. Hexagonal rod was so produced with «key size» ~ 10 mm, which then was cut on measured lengths. The measured lengths were chemically cleaned and assembled in a secondary consumable electrode which was then remelted forming the final ingot of Cu-18 wt.% Nb with more uniform microstructure.

As it was shown [Reports on Task Orders 001, 002, 003] the only possible way to attain high level of strength in the wires with large cross section 4x6 mm^2 is the process which comprise forming of «in situ» cores in a copper cladding and following assembling of them in a copper can. The scheme of manufacturing process is presented in Fig.1.

Geometrical dimensions of «in situ» fabricated hexagonal Cu-Nb rods are defined by the necessity to obtain the optimal microstructure in the final wire. As it was established at the stage of model «in situ» Cu-Nb wires investigation the dimension of Cu-18%Nb in situ cores (or subelements) should be approximately o.3 mm in dia. That is why for the fabrication of 4x6 mm^2 wire the number of Cu-18%Nb «in situ» subelements was chosen to be equal to 391.

Investigations on the microstructure have been carried out by SEM on the final wire's samples. The typical micrographs are presented in fig.2-5.

As it could be seen on fig.2 the regular arrangements of Cu-18%Nb «in situ» cores remained in the final 4x6 mm^2 wire. It should be especially noted that all Cu-18%Nb «in situ» subelements had high homogeneity of microstructure (fig.2, fig,3). Previously developed regimes of deformation with intermediate heat treatments [Report on Task Order 003] have enabled to attain the highly expended Cu matrix -Nb filaments interphase surface due to Nb 110 texture formation during the deformation by drawing. The aspect ratio of Nb filaments cross section was close to 10 (fig. 4, fig.5), which was in a good agreement with previous results [Report on Task Order 003] (look at fig.6).

The results on mechanical strength and electrical conductivity properties measurements are presented in Table 1.

The data given in Table 1 have evidenced that in spite of significant increase of cross section (24 mm^2 instead of 6-12 mm^2) the mechanical properties of designed conductor were in good coincidence with calculated parameters. The calculated, in accordance with ROM, strength of «in situ» Cu-18%Nb individual cores in designed and fabricated 4x6 mm^2 wires was in good agreement with the data for similar «in situ» Cu-18%Nb cores in model wires with small cross sections. Analyzing the data presented in Table 1 it could be stated that the decrease of microcomposite Cu-Nb wires cross section from 24 mm^2 (4x6 mm^2) to ~ 12 mm^2 (∅ 4 mm) leads to increase of strength properties without significant decrease of conductivity. Also the data presented in Table 1 show that the introduction of intermediate heat treatments during the cold deformation process leads to improvement of combination of strength - conductivity properties.

Table 1

Mechanical strength and electrical conductivity properties of Cu-Nb microcomposite wires

Definition	Cross section, mm^2	UTS, MPa	YS, MPa	δ, %	RRR	IACS, %
IS 46 391	4x6	1063	736	2	4.20	72
IS 5.7 t 391*	∅ 5.73	1000	780	1	4.92	75
IS 4 391	∅ 4.0	1170	930	2	4.2	71
IS 4 t 391*	∅ 4.0	1100	920	3.5	4.7	73
D 23 62 Zr	2x3	980	577	2	5.11	75
D 23 62	2x3	965	568	2.7	5.22	75
D 2.86 t 62*	∅ 2.86	900	370	2.5	5.6	79
D 3.4 62	∅ 3.4	880	395/720	3.8	5.39	80
D 3.06 62	∅ 3.06	930	560	3.4	5.31	79
D 2.86 62	∅ 2.86	940	495	3.7	5.17	77
D 2.07 62	∅ 2.07	1040	600	3.4	5.1	75
D 1.72 62	∅ 1.72	1100	630	3.2	5.0	74
D 1.42 62	∅ 1.42	1170	680	3.3	4.7	71
D 1.21 62	∅ 1.21	1210	690	2.2	4.6	70
D 1.0 62	∅ 1.0	1280	770	1.9	4.6	66
D 0.8 62	∅ 0.8	1330	915	1.2	4.5	64
D 2.86 t 62*	∅ 2.86	930	520	5.6	5.34	78

* t - Derformation with intermediate heat treatment.

Cu-Nb microcomposite wires produced by the «bundle and deform» process, which eliminates the operation of melting.

On the basis of previously fulfilled investigations [Reports on Task Orders 002 and 003] the following initial materials have been chosen: high quality oxygen free electron beam melted copper (99.99%) with $RRR_{300/4.2K} \geq$ 200 and high quality electron beam melted niobium.

At the earlier stages of work it was shown that for Cu-Nb microcomposite wires produced by the «bundle and deform» process the proper choosing of Nb volume fraction in first order strands is very crucial for extensive deformation. The second very important factor is the uniformity of geometrical array of first and second strands in wires cross section. That is why it was proposed to use as an initial billet not a bimetal initial element but a little bit more complicated billet consisted of 12 copper and 7 niobium rods in a copper can. The drawing of such initial billet is presented in Fig.7. The scheme of whole manufacturing process is presented in Fig.8. The numbers of Nb filaments in the strands are given in a Table 2.

Table 2

Distribution of Nb filaments in Cu-Nb microcomposite wire with continuous filaments

Definition of strand	Number of Nb filaments	Volume fraction of Nb
First order	7	0.24
Second order	1477 (7x211)	0.205
Third order	311,647 (7x211x211)	0.175
Forth order	62,017,753 (7x211x211x199)	0.149

Investigations on the microstructure have been carried out by SEM on the final wire's samples. The typical micrographs are presented in fig. 9-14.

As it could be seen out of presented microstructures in a final wire the regular geometrical arrangement of components was successfully maintained - the dimensions and cross section areas of forth, third and second order strands (Fig.9 x100, Fig.10 x300, Fig.11 x5000). It should be noted that as it was predicted earlier [Report on Task Order 003] the decrease of Nb volume fraction in first order strand from 41% to 23% led to appropriate enhancement of tendency to the formation of ribbon like cross section of Nb filaments with large aspect ratio. The schematic process of transformation of initially round Nb filaments cross section to ribbon like final cross section was suggested earlier (Fig.13) [Report on Task Order 003, Fig.17]. The real microstructure of 2x3 mm^2 Cu-Nb wire designed and fabricated now is presented in Fig.12 x10000. It was shown that the decrease of initial Nb volume fraction (down to 24%) in a first order strand guaranteed the possibility to avoid any problems with mechanical breakage during the manufacturing of long length (≥ 100 m) 2x3 mm^2 wires with 62 mln Nb filaments maintaining the reasonably high level of mechanical strength.

The results of the mechanical strength and electrical conductivity investigations for the wires designed are presented in Table 1 and Fig.14-16. The comparison of strength and conductivity properties for wire with 62 mln Nb filaments, which was fabricated now with appropriate parameters for the wire with 39 mln Nb filaments, which was fabricated under the Task Order 003, shows the following. First - «bundle and deform» process, which eliminates the operation of melting showed the reasonably high reproducibility (Fig.14). Second - proposed modification of first order billet's design (Fig.7) improved the homogeneity (uniformity) of Nb filaments arrangement in Cu matrix which as a consequence narrowed the deviations of electrical conductivity values from linear dependence ρ vs 1/D (Fig.15, 16): Summarizing the results of microstructure, strength, conductivity investigations, it could be stated that there is the possibility to further design optimization of Cu-Nb microcomposite wires produced by the «bundle and deform» process. The main aspect of this optimization is connected with the application of larger scale billets dimensions (130 - 200 mm in dia) which will make feasible further increase of homogeneity (uniformity) of Nb filaments arrangement in Cu matrix.

Analyzing the data presented in Table 1 It could be seen that Cu-Nb microcomposite wires produced by the «bundle and deform» process, which eliminates the operation of melting having the similar level of mechanical properties which are typical for traditional «in situ» produced wires at the same time are characterized by slightly higher values of conductivity at room temperature and RRR_{77} values. It could stated that the better combination of strength and conductivity properties for Cu-Nb wires produced by the «bundle and deform» process compensates higher cost of more complicated manufacturing process.

Conclusion

- Two types of high strength - high conductivity Cu-Nb wires have been designed and appropriate manufacturing processes have been proposed and experimentally approved. Long length conductors with rectangular cross sections 4x6 mm^2 (~15 m) and 2x3 mm^2 (~100 m) have been fabricated by the «in situ» process and by the «bundle and deform» process, which eliminates the operation of melting, accordingly.
- Investigation on the microstructure of both types of the fabricated wires has been conducted. Mechanical properties and electrical conductivity parameters have been measured. UTS value of fabricated wires attained 1000 MPa; RT conductivity was in the range of 75-78% IACS and $RRR_{300/77K}$ values were equal to ≥ 5.0.

List of Captions

Fig.1 Scheme of Cu-Nb microcomposite wires «in situ» processing procedures

Fig.2 Microstructure of Cu-Nb «in situ» produced wire with 4x6 mm^2 cross section (x100)

Fig.3 Microstructure of Cu-Nb «in situ» produced wire with 4x6 mm^2 cross section (x300)

Fig.4 Microstructure of Cu-Nb «in situ» produced wire with 4x6 mm^2 cross section (x5,000)

Fig.5 Microstructure of Cu-Nb «in situ» produced wire with 4x6 mm^2 cross section (x10,000)

Fig.6 Aspect ratio of Nb filaments in Cu-Nb «in situ» produced wire

Fig.7 Scheme of first order billet for Cu-Nb microcomposite wires produced by «bundle and deform» process, which eliminates the operation of melting

Fig.8 Scheme of Cu-Nb microcomposite wires processing procedures by «bundle and deform» method, which eliminates the operation of melting

Fig.9 Microstructure of Cu-Nb microcomposite wires with 2x3 mm^2 cross section produced by «bundle and deform» process, which eliminates the operation of melting (x100)

Fig.10 Microstructure of Cu-Nb microcomposite wires with 2x3 mm^2 cross section produced by «bundle and deform» process, which eliminates the operation of melting (x300)

Fig.11 Microstructure of Cu-Nb microcomposite wires with 2x3 mm^2 cross section produced by «bundle and deform» process, which eliminates the operation of melting (x5,000)

Fig.12 Microstructure of Cu-Nb microcomposite wires with 2x3 mm^2 cross section produced by «bundle and deform» process, which eliminates the operation of melting (x10,000)

Fig.13 Scheme of Nb filaments transformation during the Cu-Nb microcomposite wires deformation for the wires produced by «bundle and deform» process, which eliminates the operation of melting.

Fig.14 Strength - $(1/D_{Nb})^{-2}$ dependence for Cu-Nb microcomposite wires with 62 mln and 39 mln Nb filaments, produced by «bundle and deform» process.

Fig.15 Conductivity - $(1/D_{Nb})$ dependence for Cu-Nb microcomposite wires with 62 mln Nb filaments, produced by «bundle and deform» process.

Fig.16 Conductivity - $(1/D_{Nb})$ dependence for Cu-Nb microcomposite wires with 39 mln Nb filaments, produced by «bundle and deform» process.

Cu - Nb (in-situ) Wire Processing Procedures

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Client: Bochvar lab
Job: Study
Res: Ultrafine
Label: 25-2-d5.8 x100 (16 Sep 98 18:41:07)

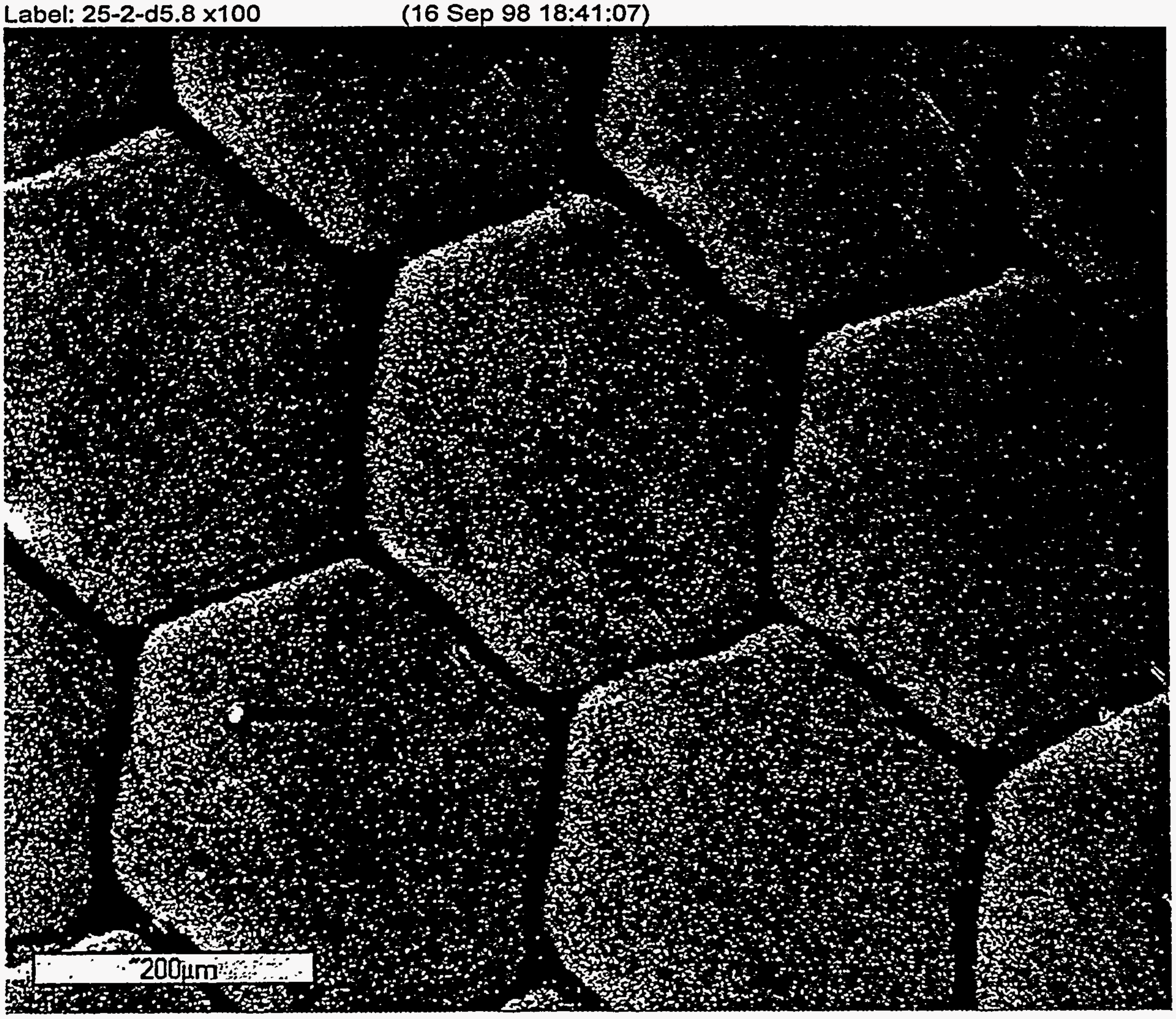

Fig. 2

Operator: Sergey Soudiev
Client: Bochvar lab
Job: Study
Res: Ultrafine
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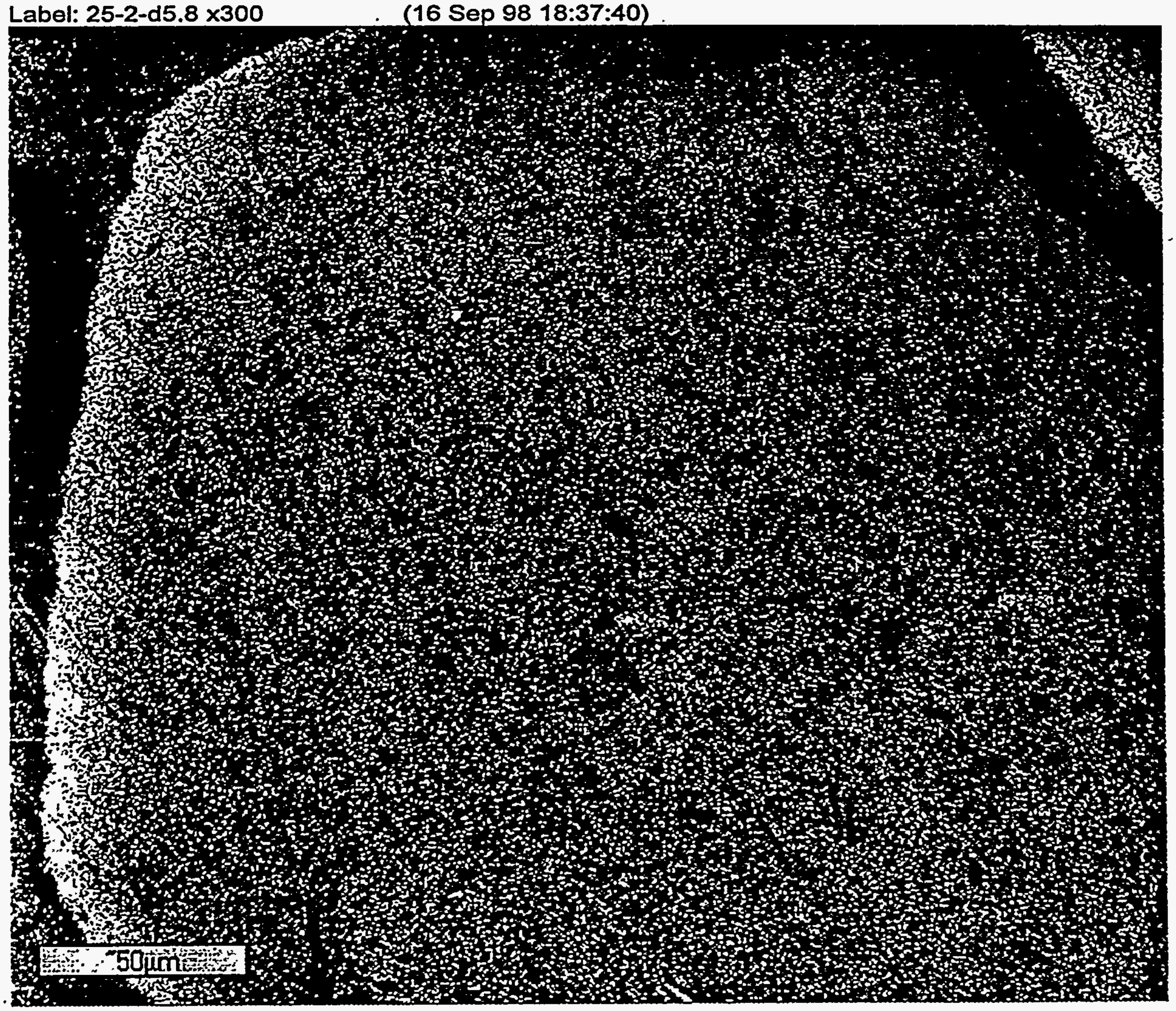

Fig. 3

Operator: Sergey Soudiev
Client: Bochvar lab
Job: Study
Res: Ultrafine
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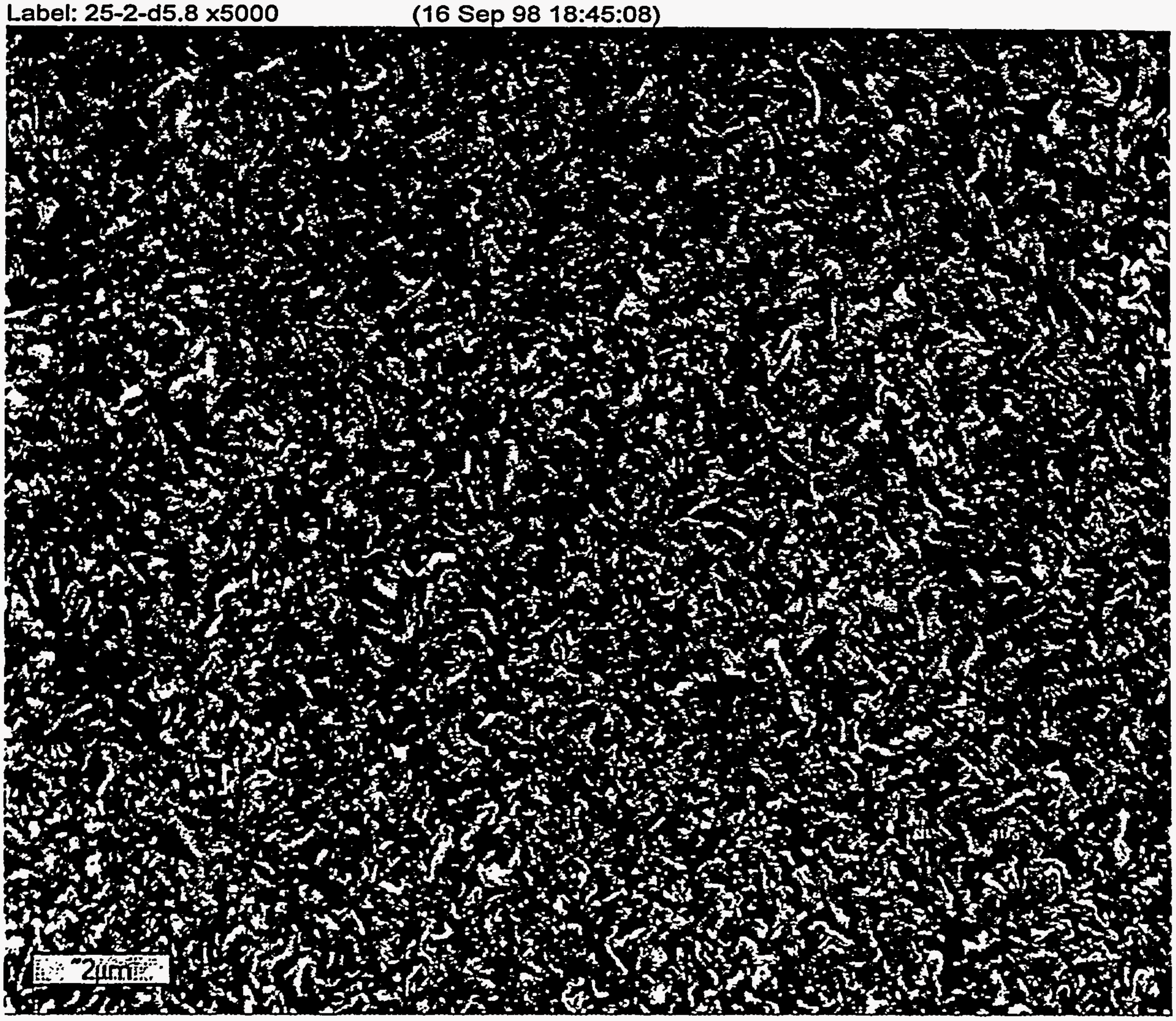

Fig. 4

Operator: Sergey Soudiev
Client: Bochvar lab
Job: Study
Res: Ultrafine
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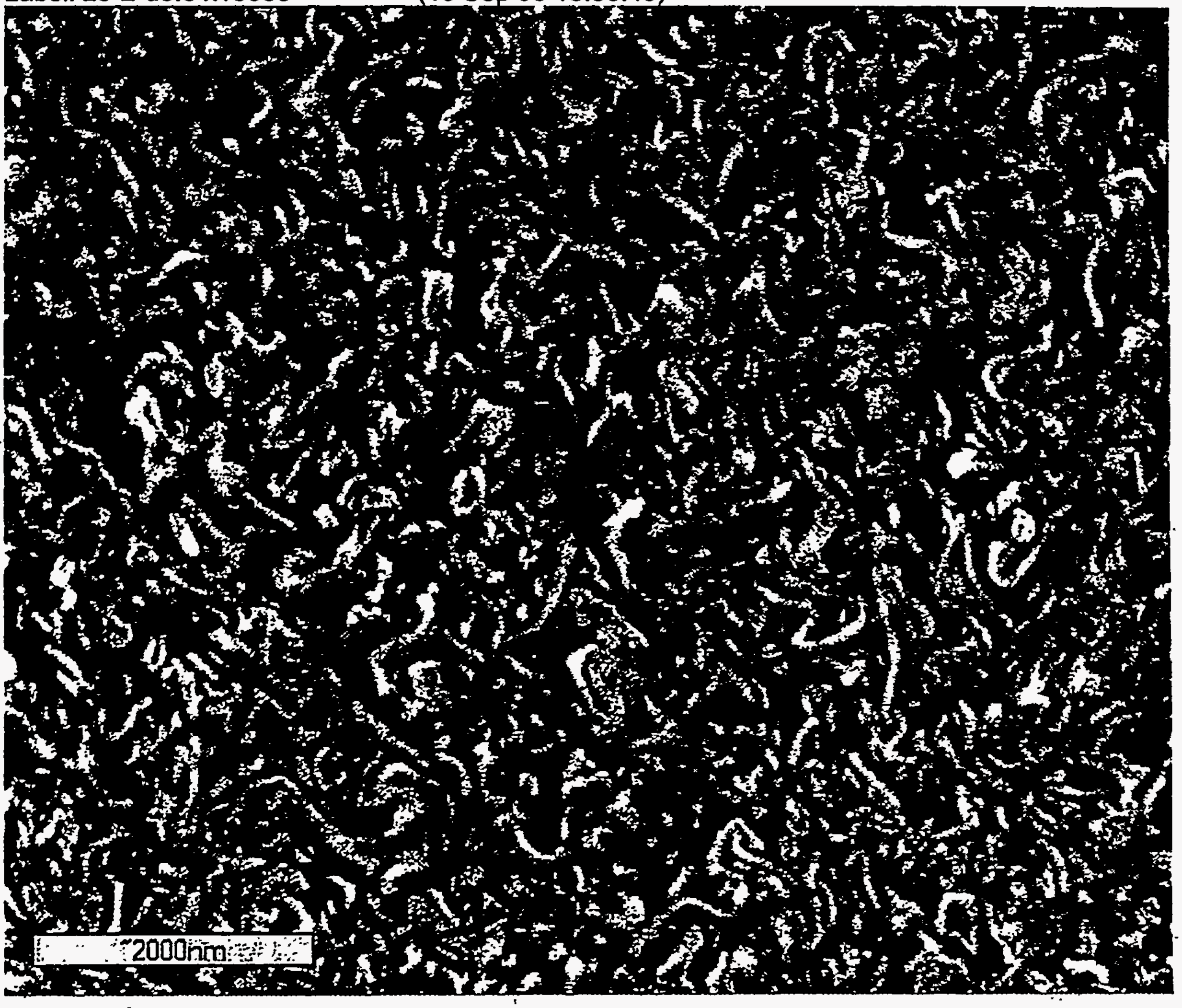
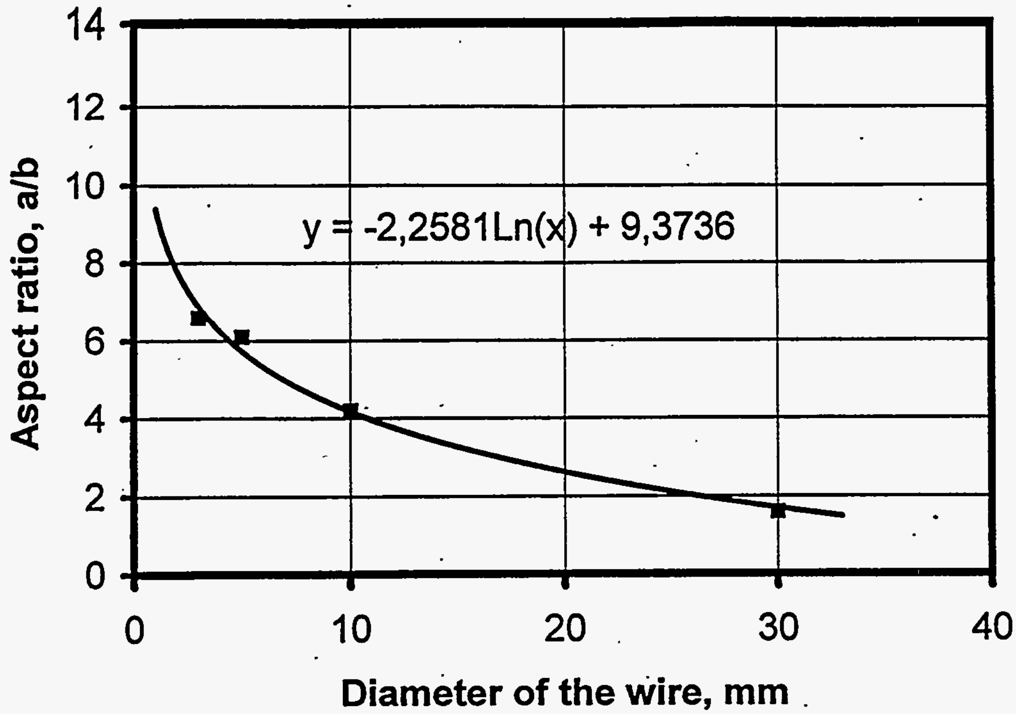
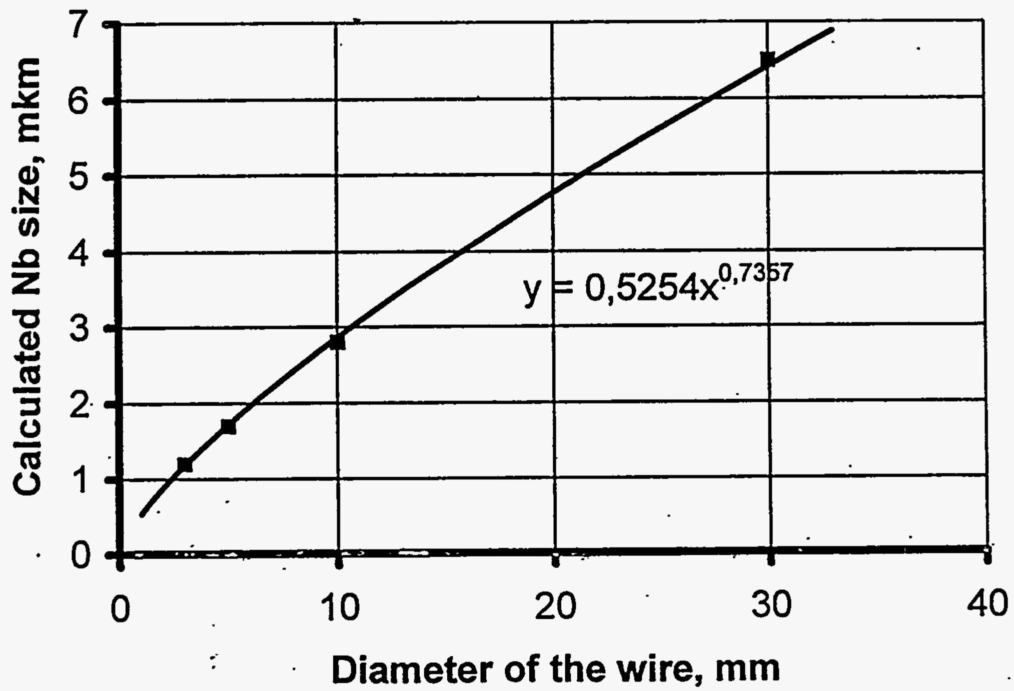


Fig. 5



14
12
10
8
6
4
2
0
0
10
20
30
40
Aspect ratio, a/b
Diameter of the wire, mm
y = -2,2581Ln(x) + 9,3736



7
6
5
4
3
2
1
0
0
10
20
30
40
Calculated Nb size, mkm
Diameter of the wire, mm
y = 0,5254x^0,7357

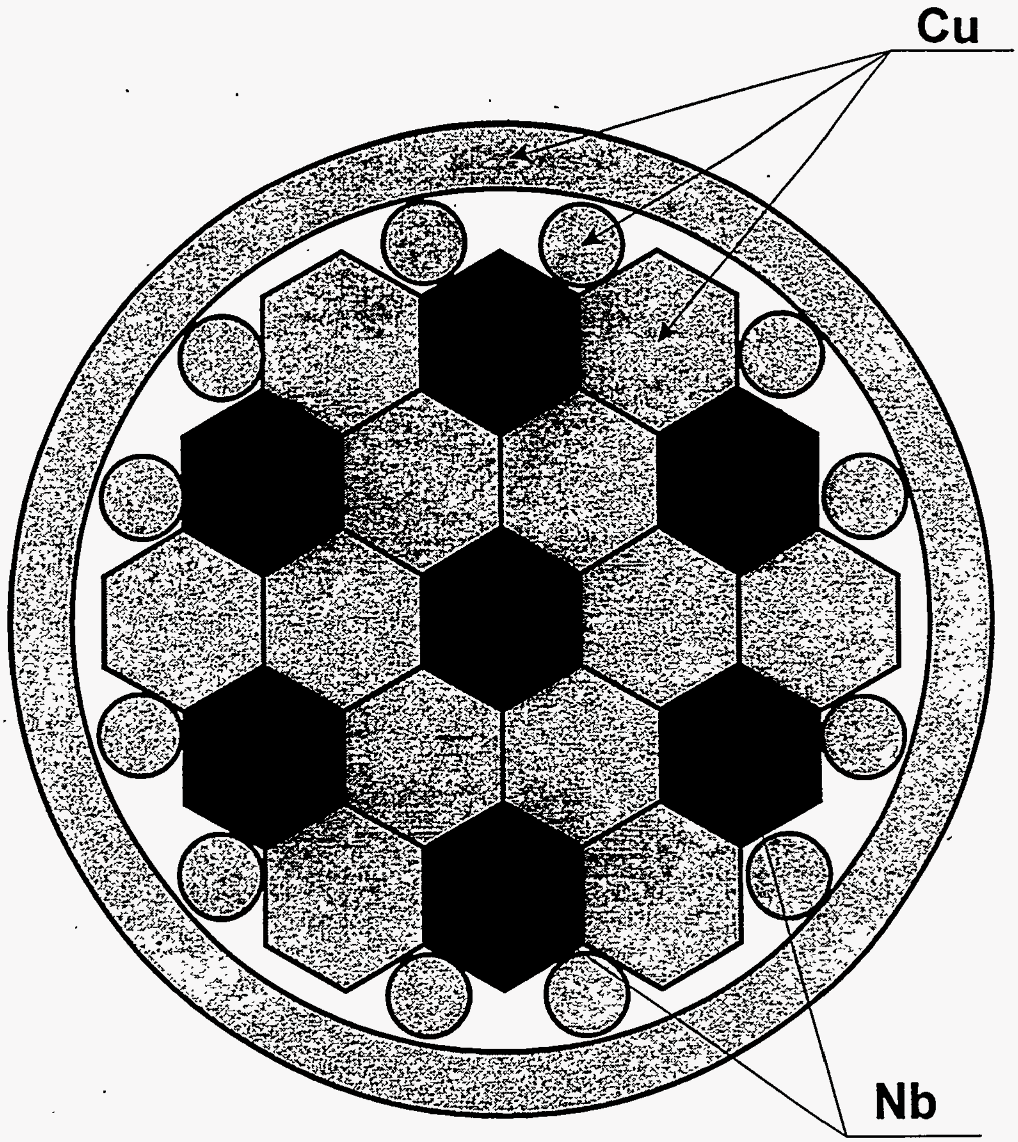
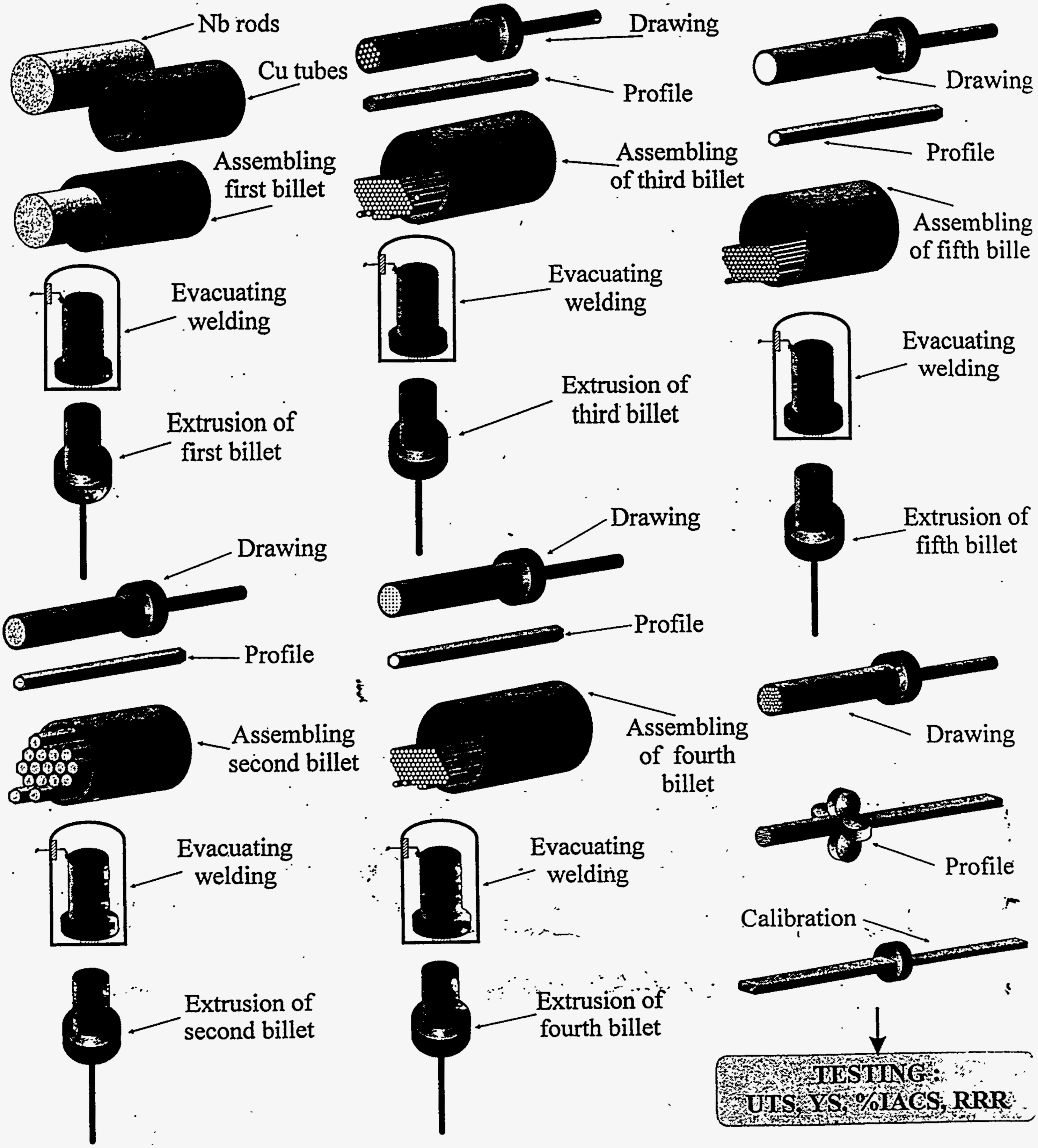


Fig.7

Cu - Nb (composite) Wire Processing Procedures



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Client: Bochvar lab
Job: Study
Res: Ultrafine
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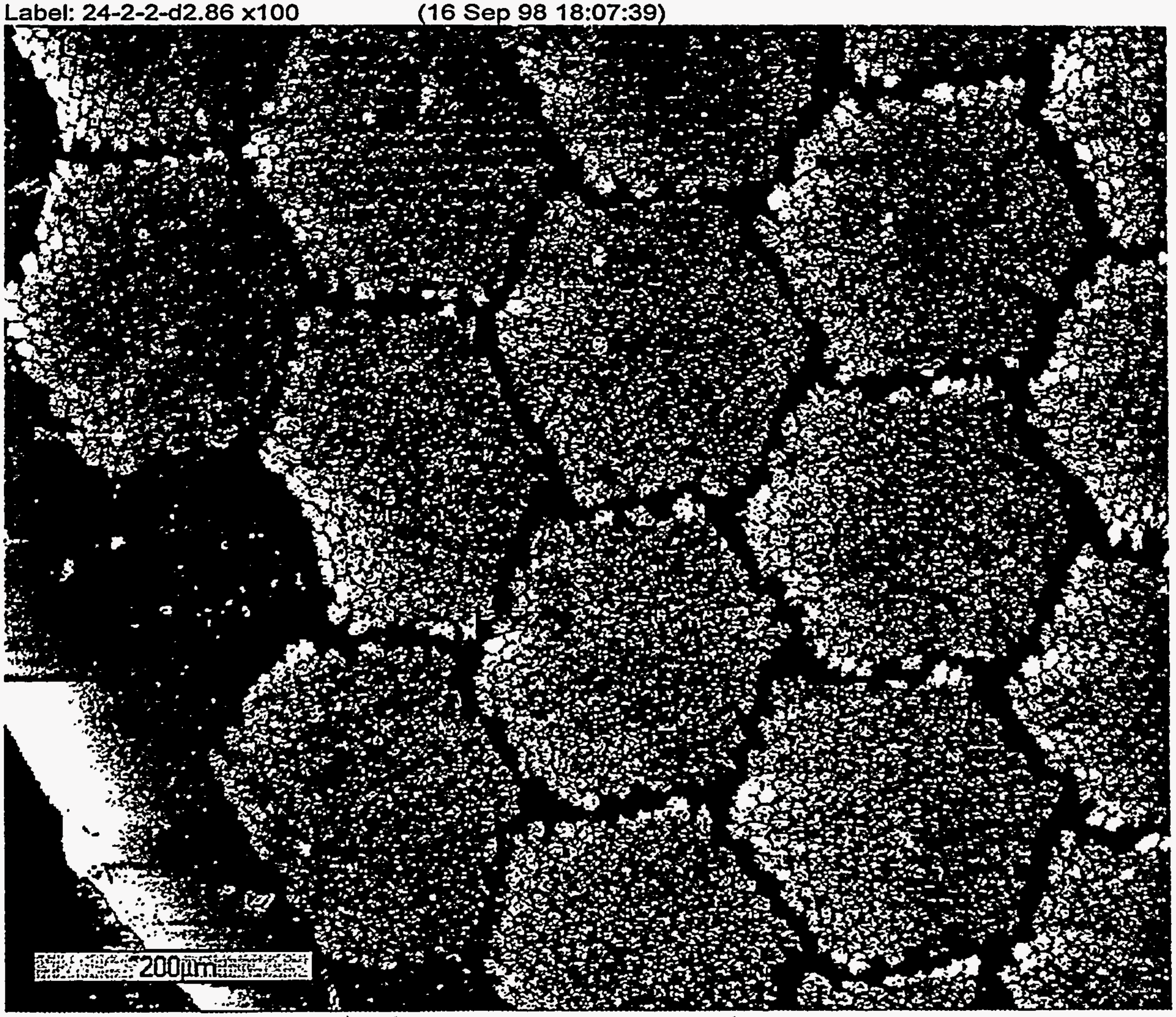

Fig. 9

Operator: Sergey Soudiev
Client: Bochvar lab
Job: Study
Res: Ultrafine
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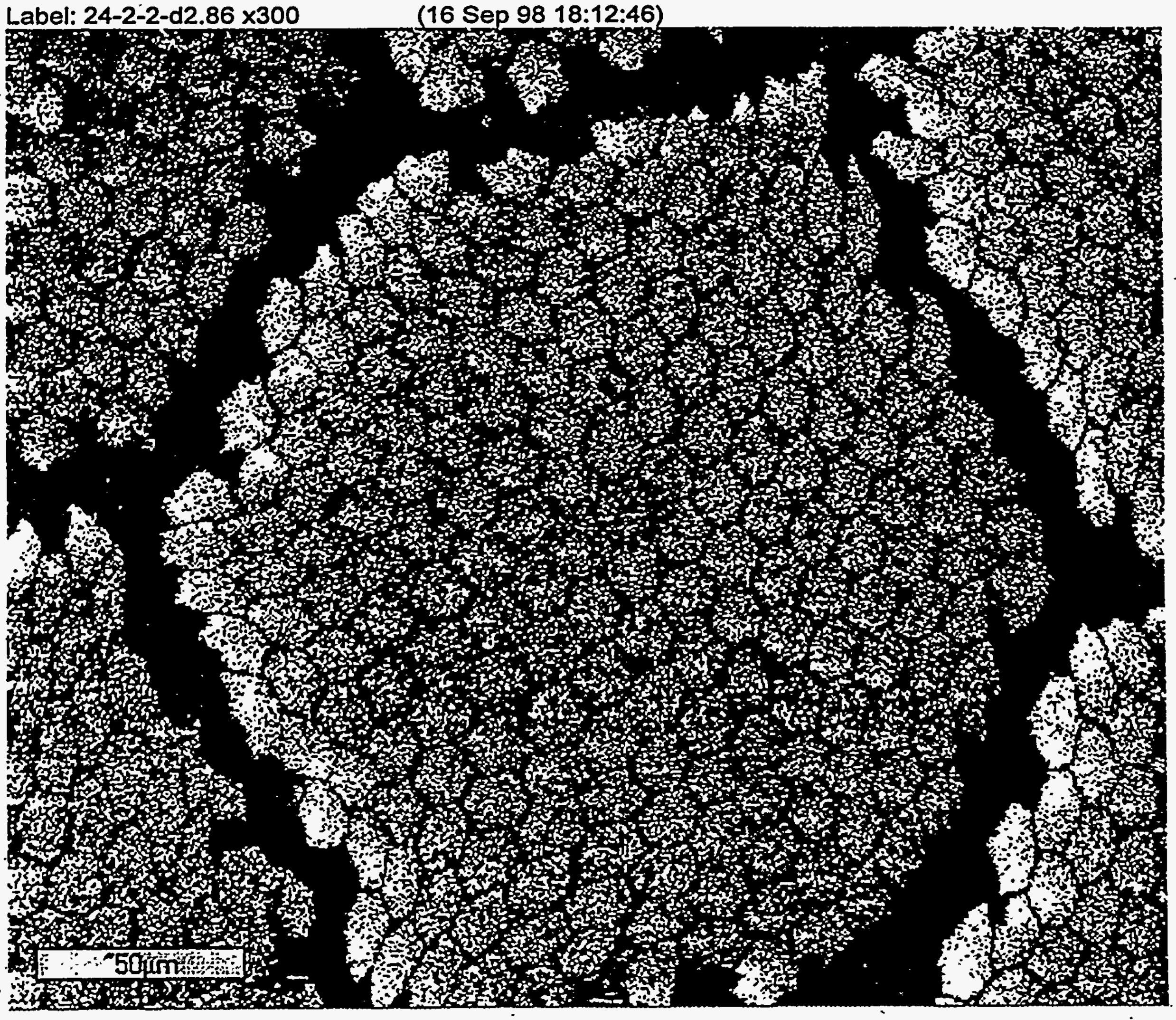

Fig. 10

Operator: Sergey Soudiev
Client: Bochvar lab
Job: Study
Res: Ultrafine
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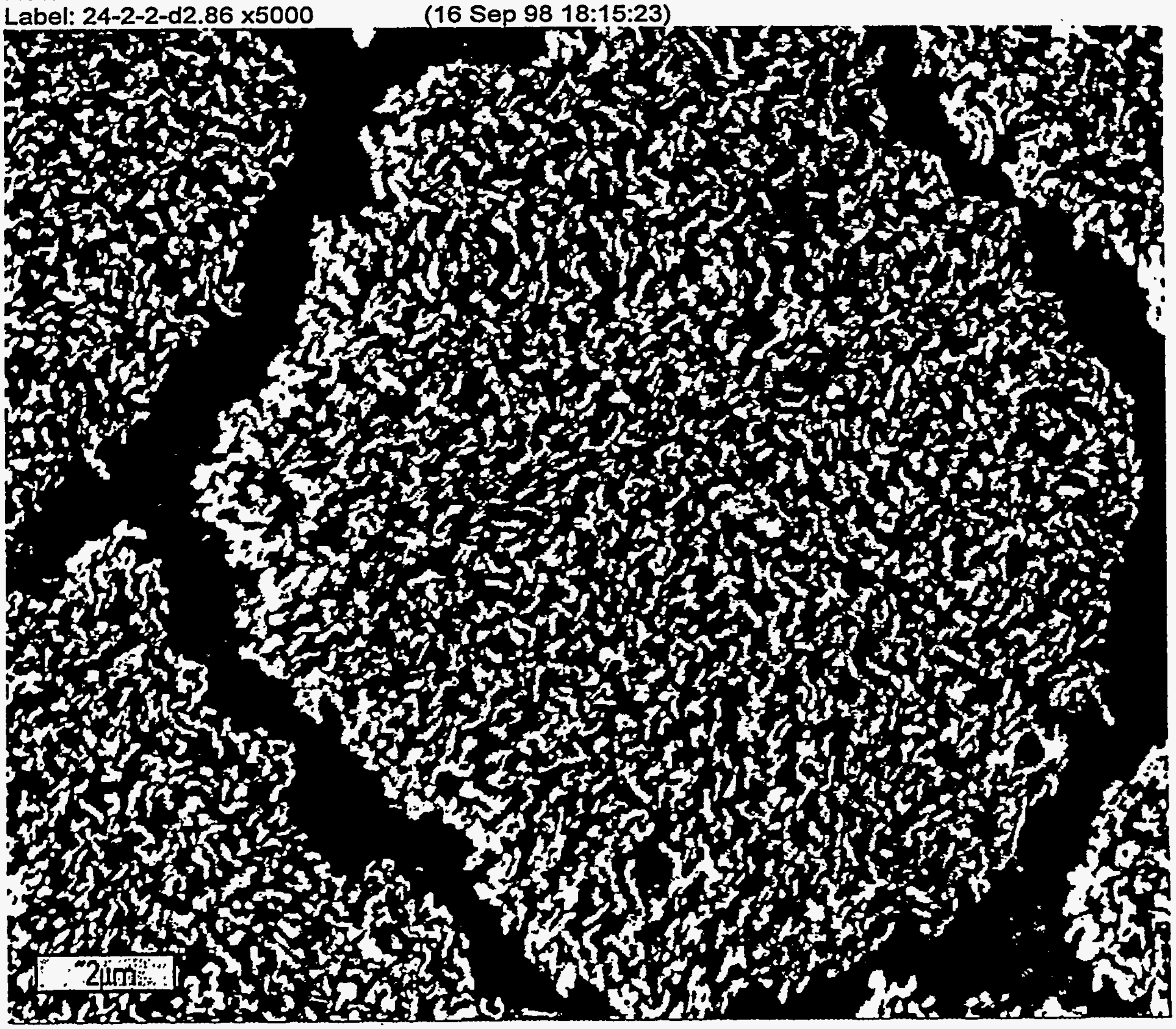

Fig. 11

Operator: Sergey Soudiev
Client: Bochvar lab
Job: Study
Res: Ultrafine
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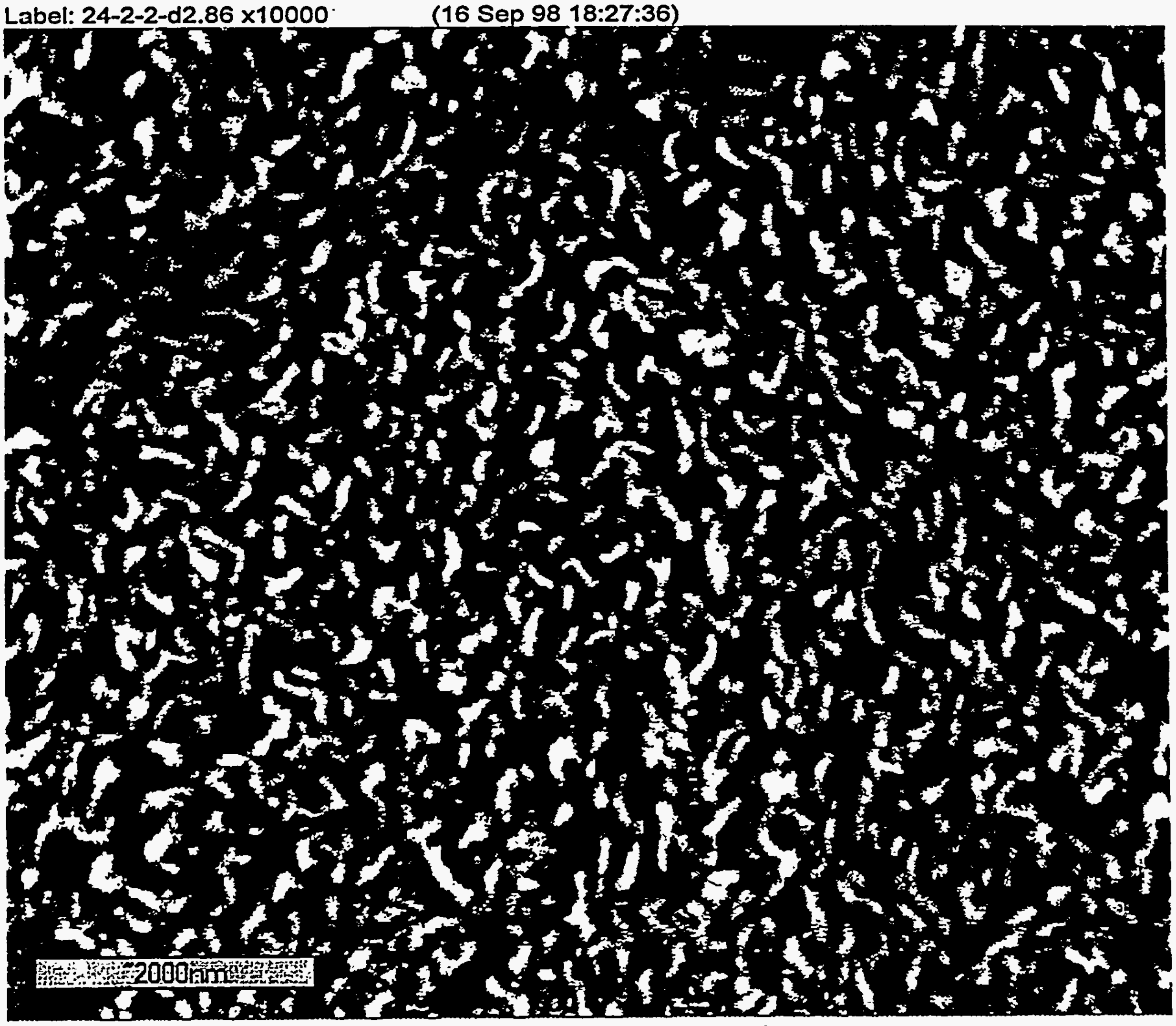


Fig. 12

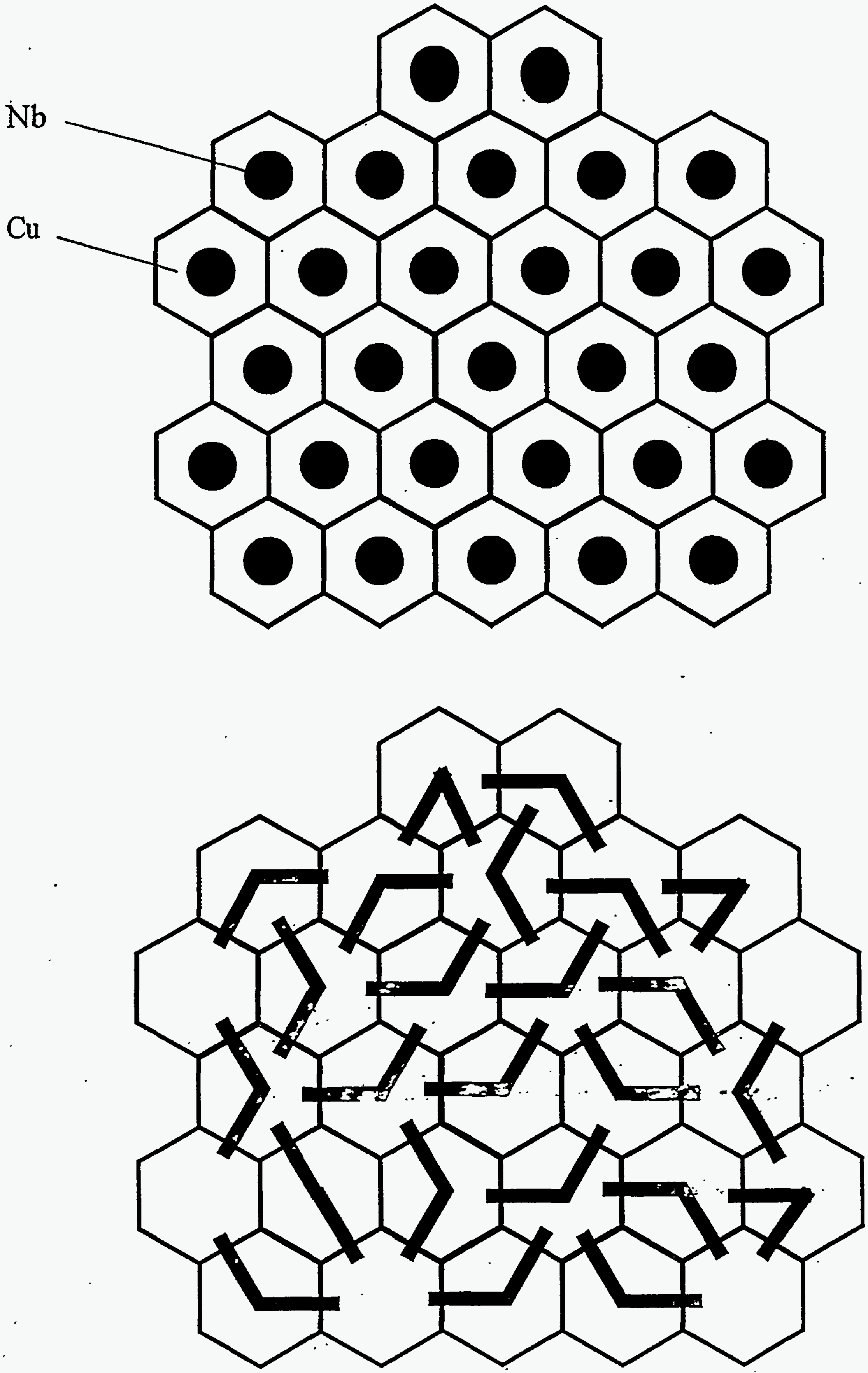


Fig 13

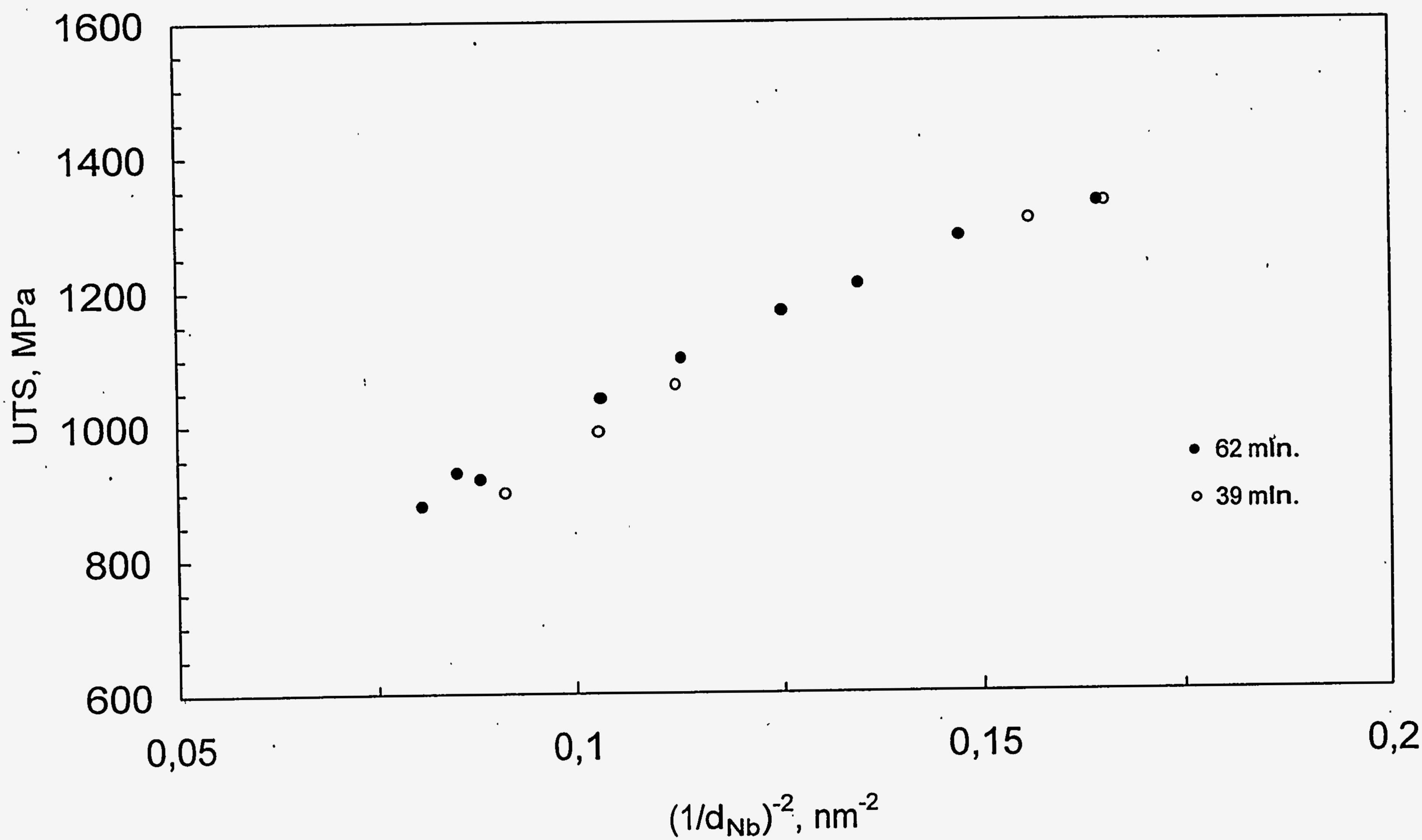
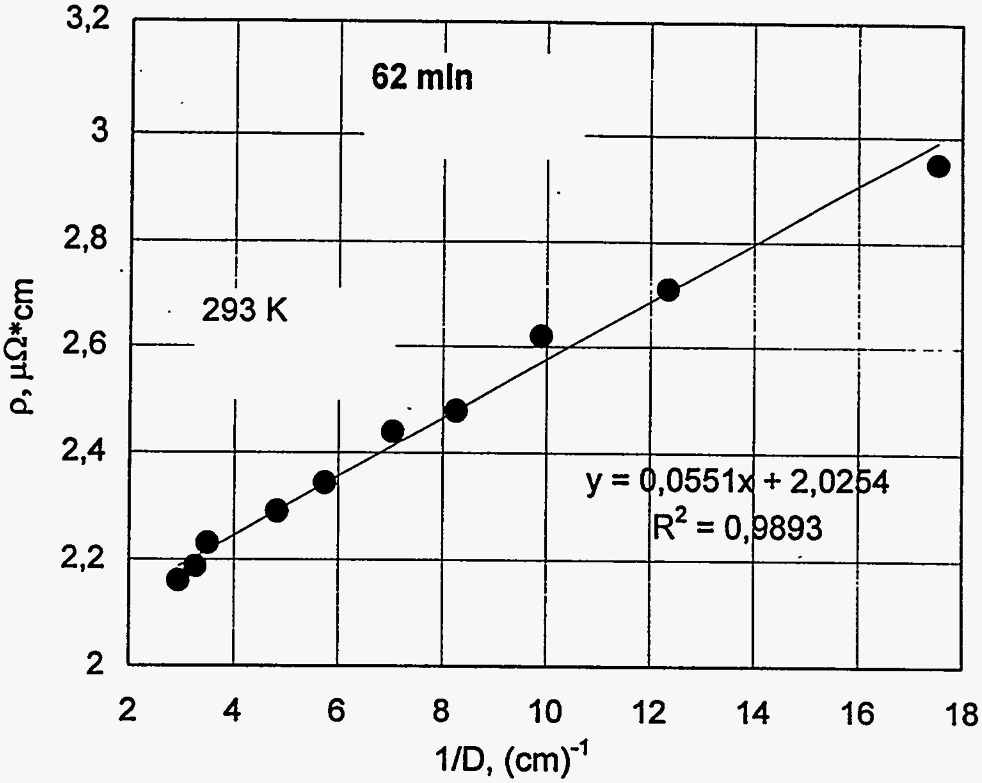
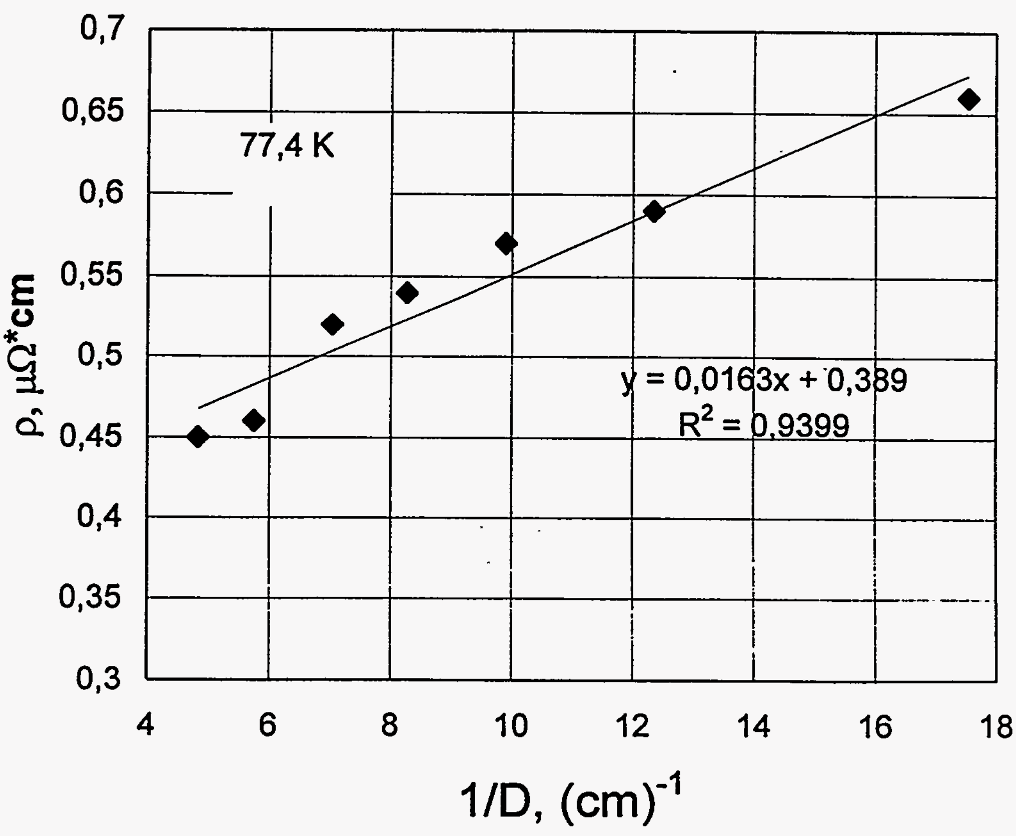

1600
1400
1200
1000
800
600
UTS, MPa
0,05
0,1
0,15
0,2
(1/d_Nb)^-2, nm^-2
62 mln.
39 mln.

Fig.14

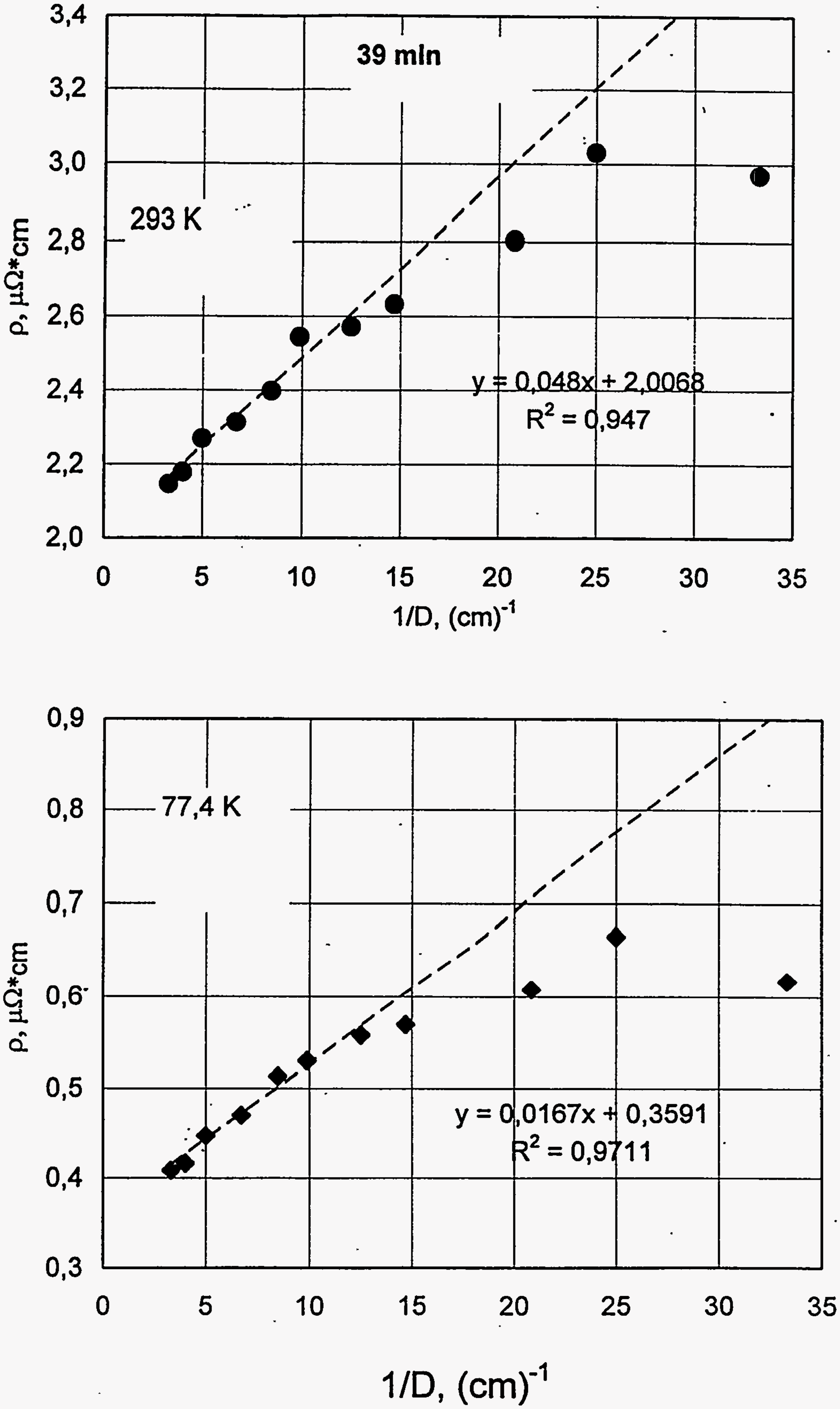


3,2
3
2,8
2,6
2,4
2,2
2
62 min
293 K
ρ, μΩ*cm
y = 0,0551x + 2,0254
R2 = 0,9893
2
4
6
8
10
12
14
16
18
1/D, (cm)-1



0,7
0,65
0,6
0,55
0,5
0,45
0,4
0,35
0,3
77,4 K
ρ, μΩ*cm
y = 0,0163x + 0,389
R2 = 0,9399
4
6
8
10
12
14
16
18
1/D, (cm)-1

Fig.15



39 min
293 K
y = 0,048x + 2,0068
R2 = 0,947
ρ, μΩ*cm
1/D, (cm)-1
3,4
3,2
3,0
2,8
2,6
2,4
2,2
2,0
0
5
10
15
20
25
30
35
77,4 K
y = 0,0167x + 0,3591
R2 = 0,9711
ρ, μΩ*cm
0,9
0,8
0,7
0,6
0,5
0,4
0,3
1/D, (cm)-1

Fig.16