Investigation of the Effects of Equal Channel Angular Extrusion on Copper C10100

by

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APPROVED

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Abstract

Equal Channel Angular Extrusion (ECAE) is an innovative process which utilizes plastic deformation to produce materials with unique properties. The process can be applied multiple times to obtain very high levels of deformation without changing the cross-sectional area of the billet. The orientation of the billet can also be varied to change the microstructure of the material. The purpose of this paper is to study the effects of ECAE on copper 10100 with particular interest in increasing the room temperature strength of the copper without increasing the resistivity. Preliminary findings based on the microstructure and hardness data indicates that ECAE has the potential to yield such a material.

Table of Contents

List of Figures

Introduction

Advanced materials are critical to the success of numerous industries. The use of large amounts of plastic deformation to improve the properties of materials is an area of great interest for advanced material development. Equal Channel Angular Extrusion (ECAE) is an innovative process which utilizes plastic deformation to produce materials with unique properties. ECAE has the potential to revolutionize the materials processing field as a completely new manufacturing method for pure metals, alloys, composites, and plastics. The new materials that might be developed, and the enhancement of existing materials as a result of ECAE could have numerous technological benefits for many industries. ECAE is currently being studied as a processing method for several materials. This paper investigates the effect of ECAE on copper C10100 with the objective of increasing the room temperature strength with little loss of conductivity.

History and Background of ECAE

The Equal Channel Angular Extrusion Process (ECAE) was invented in 1972 in the former Soviet Union by Dr. Vladimir Segal while he was a senior researcher at the Physical Technical Research Institute, Academy of Sciences, Minsk, USSR. Dr. Segal has since immigrated to the United States and is currently a research engineer at Texas A&M University in the Mechanical Engineering Department.

Significant work in ECAE started in 1976 in the USSR, and the process was patented in 1978 by Dr. Segal. Research and development of ECAE continues in the former Soviet Union at several institutes. Previous work has provided: development of a new method of plastic deformation, special manufacturing methods for producing various microstructures, the development of process conditions for various materials, and the development of dies, tools, and presses for processing single billets and continuous ingots with ECAE.

Description of ECAE

The ECAE process yields very heavy deformation in a material without changing its cross-sectional area or geometric configuration. A billet is processed by extruding it through a die with intersecting channels of the same diameter (see figure 1). The billet will deform by simple shearing in thin layers at the crossing plane of the channels. Unlike conventional extrusion processes, the cross-section of the billet remains constant. Since the process results in uniform deformation across the entire cross-sectional area of the billet, a homogenous stress state is reached throughout the billet. For a channel angle of 90° the punch pressure required is roughly equal to the flow stress of the material, which is a reduction of 1.5-2.0 times that of conventional extrusion for comparable equivalent reduction ratios [2]. A single pass with a channel angle of 90° produces an equivalent

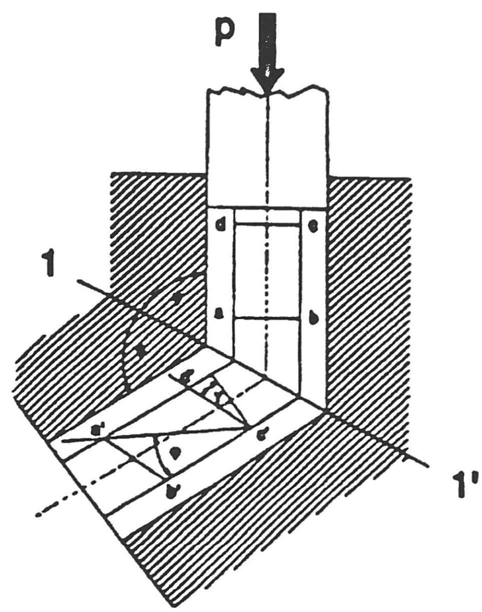


Figure 1: ECAE die

reduction ratio of 3.2 [1]. Thus, ECAE allows a large strain intensity to be reached with low pressure and load.

Multiple passes through the process can be made resulting in the ability to produce extremely high levels of deformation in a billet without changing its cross-section. After eight passes with a channel angle of 90° the equivalent extrusion ratio reaches 10,100 [1]. The planes and directions of shear can also be altered with each pass by rotating the billet. This affects the grain structure allowing various textures to be produced such as equiaxed, laminar, or fibrous orientations. Three different cases of processing orientation which are of interest include:

1) Method A - Billet orientation remains the same for each pass. This produces a laminar grain structure with the grains exhibiting a large aspect ratio.

2) Method B - Billet is rotated 90° about the extrusion axis. This produces a fibrous grain structure with very elongated grains.

3) Method C - Billet is turned 180° about its axis for each even numbered pass. This results in an equiaxed and uniform grain structure after an even number of passes.

Utilizing controlled recrystallization after ECAE can result in sub-micron grain structures. The ductility of the material is thus increased and certain materials will become superplastic at temperatures lower than normal. The ability to produce very fine grain structures and control the orientation of the grain structure yields materials with unique properties.

Area of Investigation

The objective of the research detailed in this paper is to study the effects of ECAE on copper C10100. This is an oxygen-free copper which is 99.99% pure. ECAE is being investigated as a method of improving the room temperature strength of relatively massive sections of the copper with little loss in conductivity. Cu C10100 which has been processed for improved strength without loss of conductivity has applications as a material for high strength magnetic field conductors.

A test matrix was developed for the material to study the hardness, microstructure, mechanical properties, and conductivity for a varying number of passes and orientations through ECAE. Data has been collected for hardness and microstructure; however, research is continuing to determine mechanical properties and conductivity. The microstructure of the material was observed and compared to the microstructure of the original material using light microscopy. The mechanical properties of the material will be determined through tensile testing to determine the tensile strength, yield strength, modulus of elasticity, and ductility. The conductivity will be measured at both room temperature and liquid helium temperature using an eddy current decay method.

Experimental Procedure

The 1" square by 6" long billets of copper 10100 were cut from a single commercial bar. All billets were annealed at 600° for 1 hour in an inert atmosphere (argon gas) furnace and then water quenched. Processing with ECAE was performed at room temperature on a specially designed die which was mounted on a 500 kip hydraulic press (see Figure 2 for a general schematic). The billets were cold deformed with a varying number of

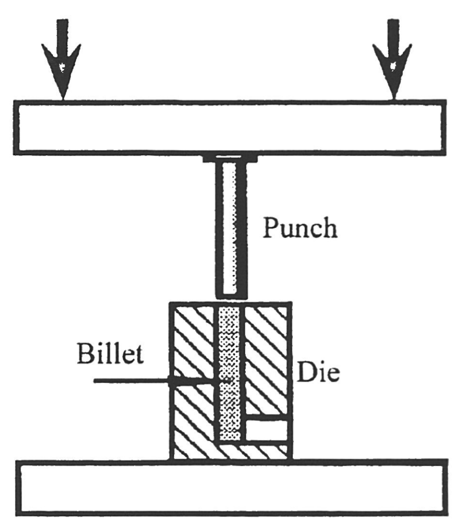


Figure 2: General Apparatus

passes (N = 1, 2, 4, and 8) and orientations (methods A, B, and C) through ECAE. Previous research has shown that processed copper billets can be stored at room temperature for extended periods of time without changes in the material properties; therefore, no special considerations were taken in storing the billets. After processing, the billets were cut into smaller samples using a cut-off wheel. The samples were annealed at temperatures ranging from 100 - 220°C for 1 hour and then water quenched. Hardness testing was performed on the original and annealed samples using a digital Rockwell hardness machine on scales HRB and HRF following standard hardness testing procedures.

Selected samples were prepared to provide a complete evaluation of the effects produced by ECAE and the subsequent annealing on the microstructure. All microstructure studies were performed using light microscopy. The samples were mounted using epoxy and polished with successively finer grits of sandpaper, diamond paste, and aluminum carbide to provide a surface with the necessary smoothness to study the microstructure. Etching times were 40-50 seconds for the samples (see Table 1 for the composition of the etchant).

Amount	Compound
2 g	$K_2Cr_2O_7$
8 mL	H_2SO_4
4 mL	NaCl (saturated solution)
100 mL	H_2O

Table 1: Etchant Composition

Results and Discussion

The test matrix showing the number of passes, orientation, and annealing temperatures for each sample is shown below in Table 2.

Sample	Passes	Method	Annealing Temperatures (°C)
0	0		
1	1		100, 130, 160, 190, 200, 210, 220
2	2	A	100, 130, 160, 190, 220
3	2	C	100, 130, 160, 190, 220
4	4	A	100, 130, 145, 160, 190, 220
5	4	B	100, 130, 138, 145, 160, 190, 220
6	4	C	100, 130, 145, 160, 190, 220
7	8	A	100, 130, 160, 190, 220
8	8	B	100, 130, 160, 190, 220
9	8	C	100, 130, 160, 190, 220

Table 2: Test Matrix

Complete hardness data and microstructure results have been collected for the test matrix from these samples. Mechanical properties and resistivity tests have not been performed and thus will not be presented in this paper.

Hardness data was collected for all samples using a Rockwell HRB scale (see appendix A for complete data). For samples which produced very low or negative hardness numbers on the HRB scale, data was also collected on the HRF scale. The use of the HRB scale for all samples allows annealing curves to be developed for each billet. The HRF scale then provides accurate hardness values for those samples which are very soft (e.g. highest annealing temperatures and strain intensity). Five hardness values were taken for each sample, plotted versus annealing

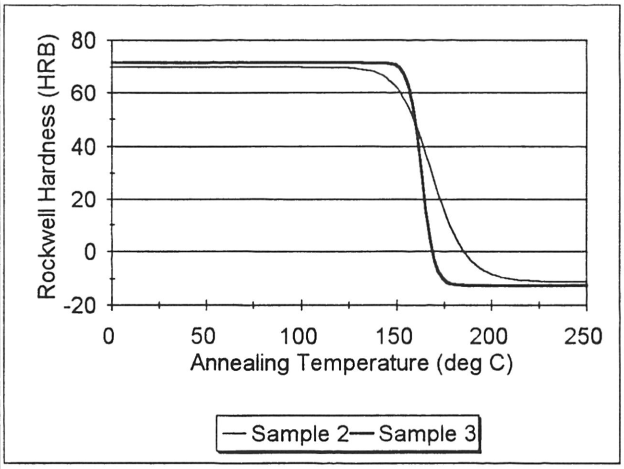


Figure 3: N=2

temperature for each billet, and a curve fit was generated (see appendix B for the curve fits).

Figures 3, 4, and 5 each show the annealing curves for billets processed with the same number of passes, but with different methods. It is evident from these plots that the different methods had little effect on the recrystallization temperature, however method B did produce slightly higher hardness values for the lower shelf. Figures 6, 7, and 8 show the annealing curves for billets processed with the same method, but for a different number of passes. These curves exhibit a significant difference in recrystallization temperature between the one pass and multiple pass samples,

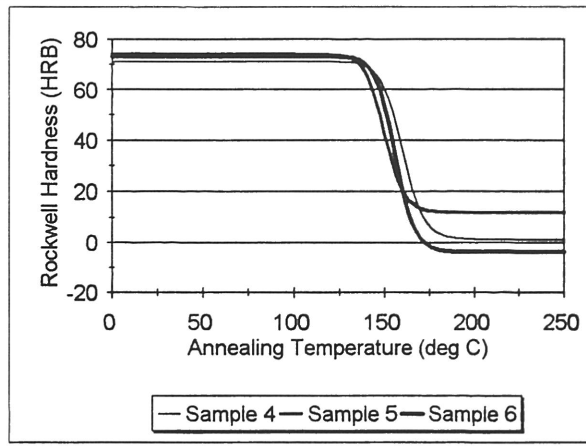


Figure 4: N=4

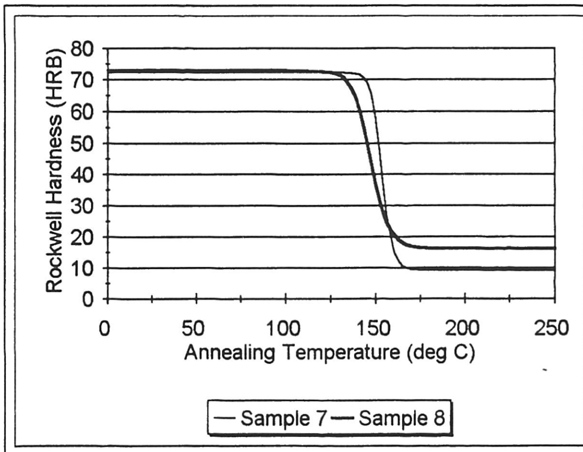


Figure 5: N=8

but little difference among the multiple pass samples. This indicates that the strain intensity after two passes is great enough that any additional strain intensity has no effect on recrystallization temperature. Therefore, the different methods show no difference in recrystallization temperature because the strain

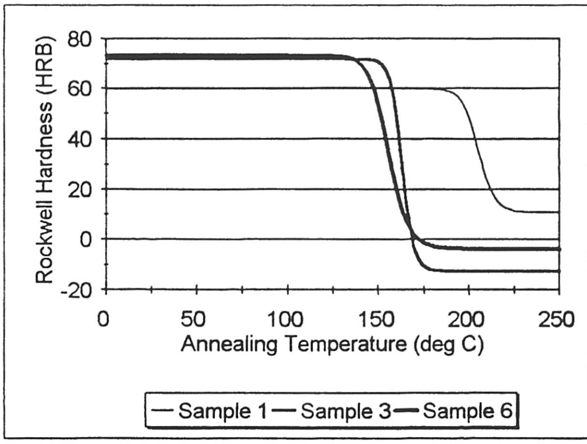


Figure 6: Method C

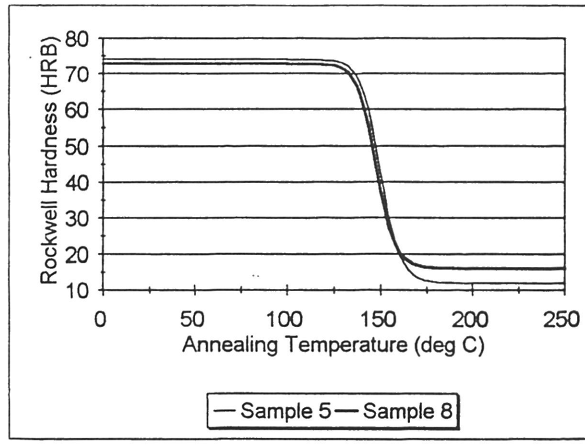


Figure 7: Method B

energy present in the material is great enough to mask any effect the different methods of processing might produce.

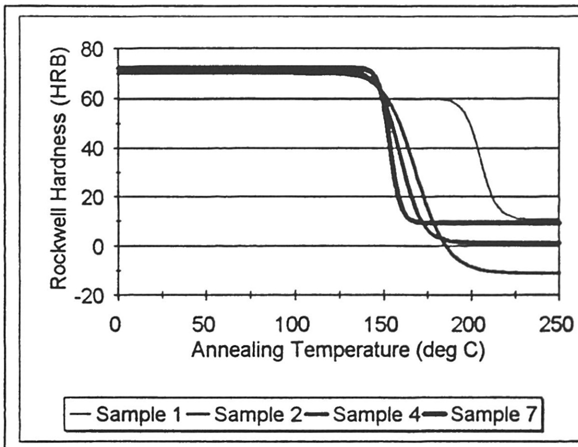


Figure 8: Method A

Examining the microstructure of the samples gives a more detailed picture of the effects of ECAE on copper. Figure 9 shows the microstructure of the copper in its original unprocessed state (Note: all photographs have a magnification of 60X and all photographs of processed samples are in the flow plane unless otherwise noted). The grains are large and well defined with distinct grain boundaries. Figure 10 shows the affect of ECAE on copper after one pass. The sample exhibits heavy deformation, a large number of slip lines, and a large change in the aspect ratio of the grains. Figure 11 is a sample of two passes with Method A. The grains have become even more deformed and now represent a laminar structure. Additional passes with method A continue to develop a heavily deformed laminar structure (Figure 12 - 8 passes, Method A).

Figure 9: Sample 0

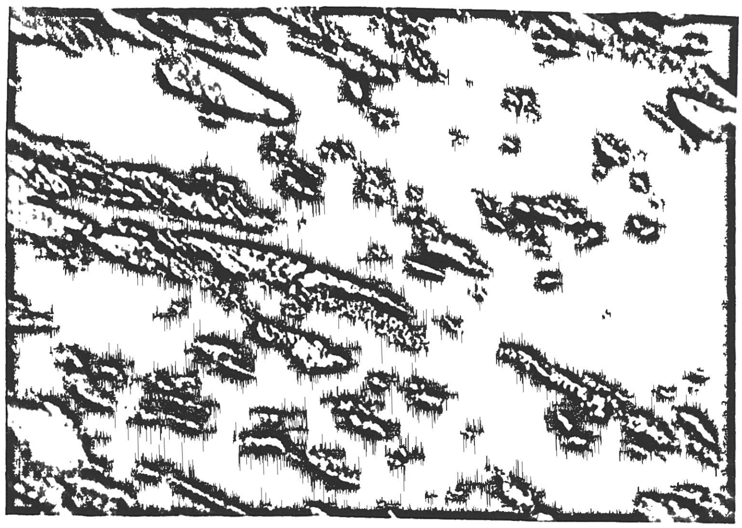


Figure 10: Sample 1

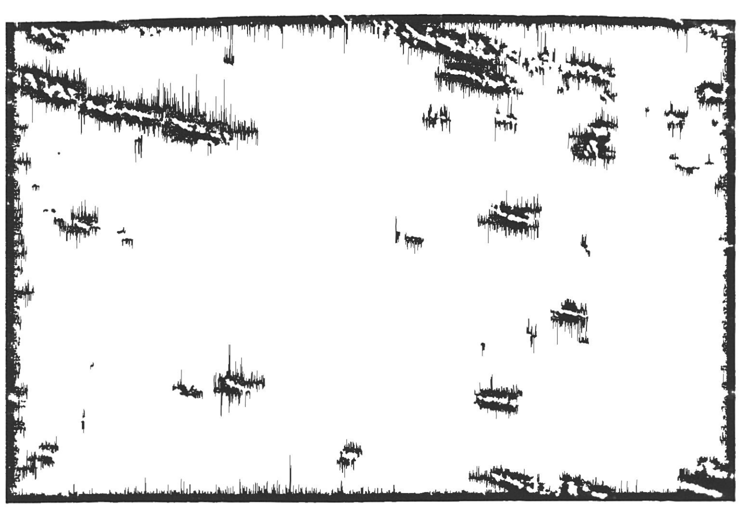


Figure 11: Sample 2

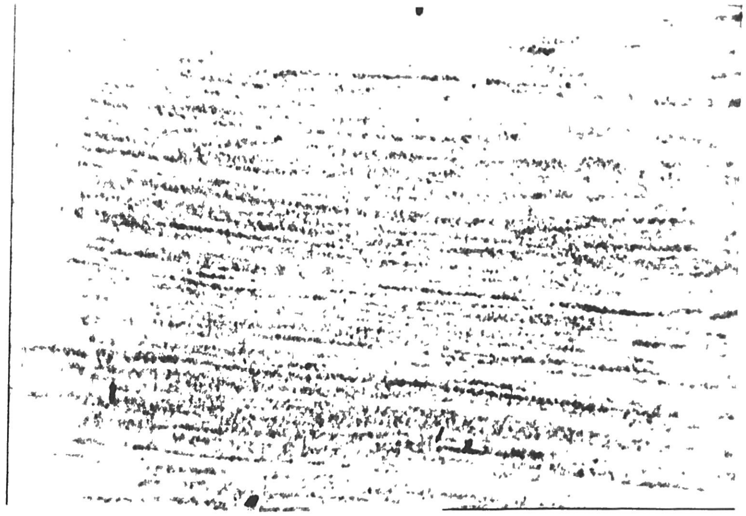


Figure 12: Sample 7

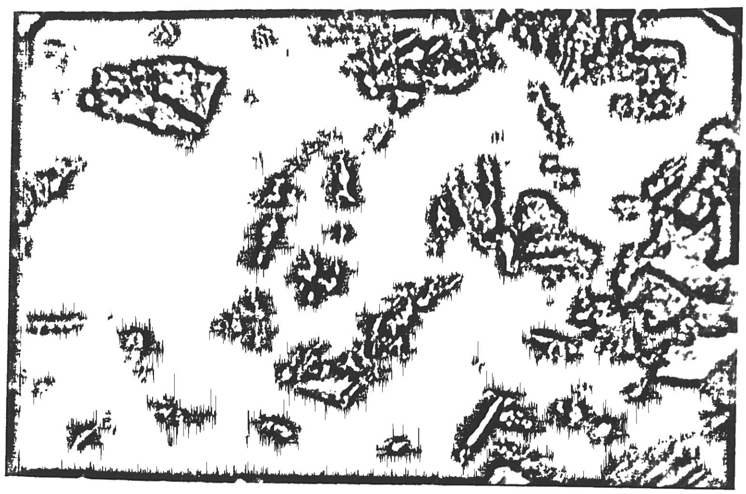


Figure 13: Sample 3

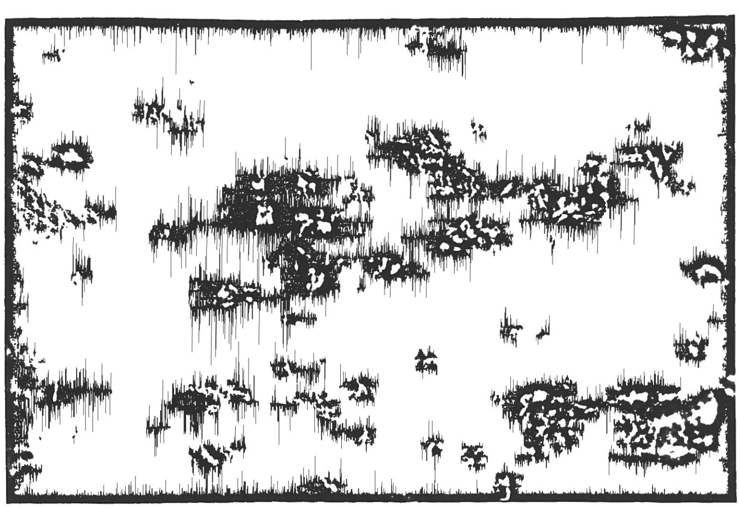


Figure 14: Sample 6

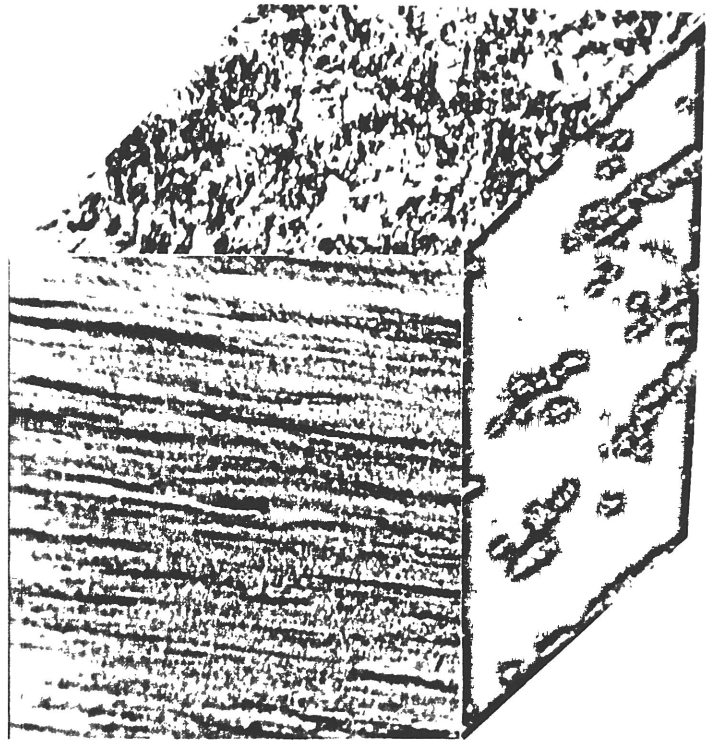


Figure 15: Sample 4, 3-d

Figure 16: Sample 6, 3-d

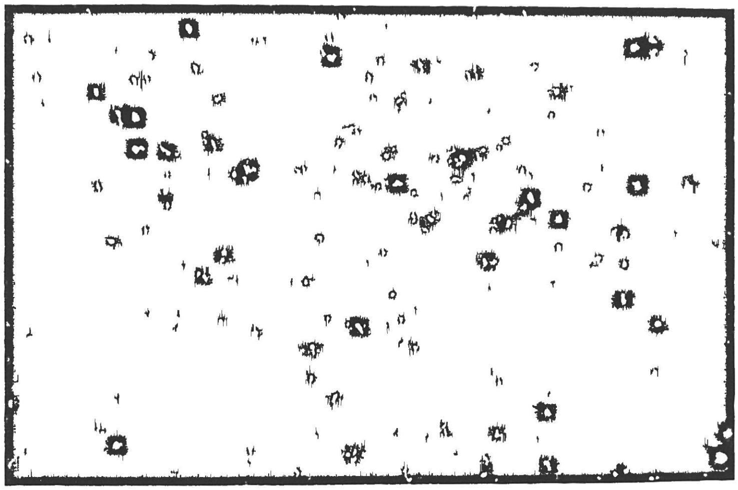


Figure 17: Sample 4, 190°C

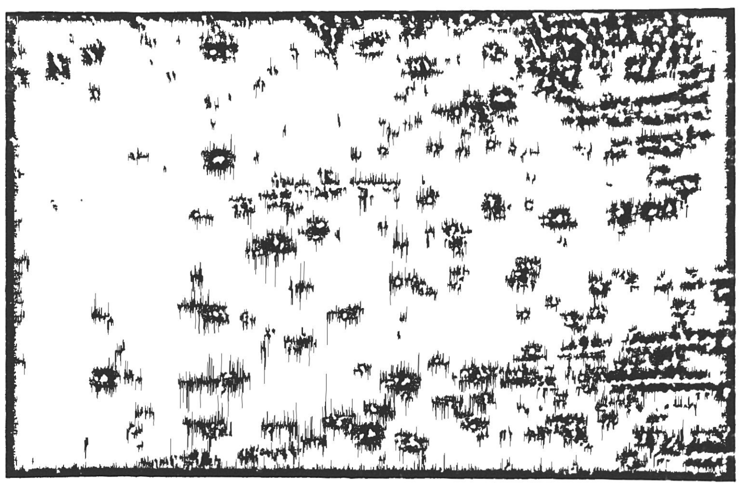


Figure 18: Sample 4, 160°C

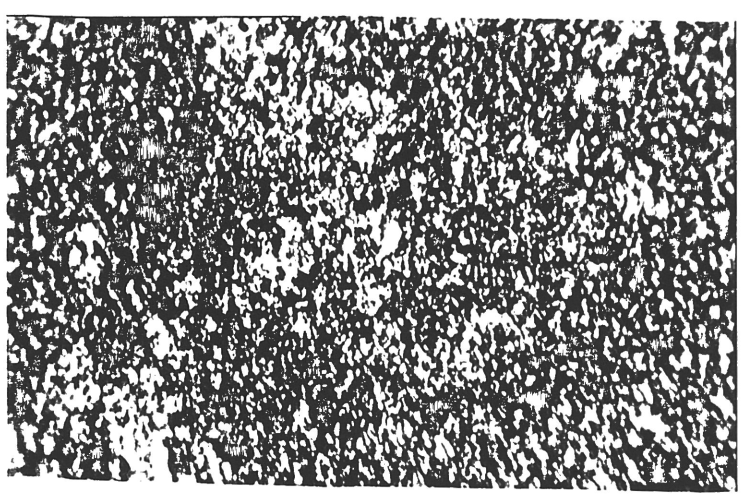


Figure 19: Sample 6, 190°C

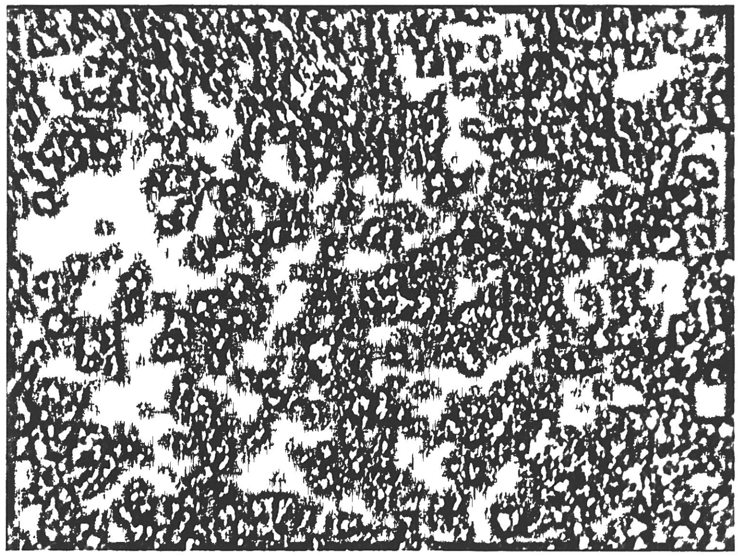


Figure 20: Sample 6, 160°C

Figure 13 demonstrates the effects of Method C after two passes. Notice that the original structure has been restored compared to the sample after one pass (Figure 10). Method C produces a heavily deformed, but uniform and equiaxed grain structure. Additional passes with Method C (see Figure 14 - 4 passes, Method C) yields greater deformation with the grains becoming less sharp and the grain boundaries almost dissolving. Figures 15 and 16 illustrate the grain structure in three dimensions for Methods A and C (four pass samples). The front of the cube represents the flow plane while the sides and the top represent the short transverse plane and the longitudinal transverse plane respectively. In Figure 15 (Method A), the laminar, pancake-like structure is readily visible, while in Figure 16 (Method C) the grain structure is uniform on all three sides as expected.

A very fine grain structure is observed for both Methods A and C after annealing at temperatures high enough to produce recrystallization. Figure 17 shows sample 4 after annealing at 190°C. The grain structure is almost completely recrystallized and shows little similarity to the unannealed sample. Figure 18 shows sample 4 after annealing at 160°C. Recrystallization has begun, but the previous grain structure is still readily visible. Figures 19 and 20 exhibit the same properties for Method C. Additional photographs of microstructure are included in Appendix C.

Conclusion

ECAE can have a profound effect on the structure and properties of a material. Great amounts of plastic deformation can be imparted uniformly on the material. By controlling the orientation and number of passes of the billet, the structure of the material is determined. When applied to copper C10100, ECAE results in significant increases in hardness (and thus strength). By annealing the material, a very fine grain structure can be produced. The different methods of processing allow for the production of very dissimilar structures from the same material. With the proper balance of number of passes, billet orientation, and annealing temperature, a C10100 copper with significantly increased strength and little increase in resistivity should be possible.

References

1. V.M. Segal, "Simple Shear as a Metalworking Process for Advanced Materials Technology"
2. V.M. Segal, "Working of Metals By Simple Shear Deformation Process"

Appendices

Rockwell Hardness

Ave HRB											Ave HRF	
Sample	no anneal	100 deg	130 deg	138 deg	145 deg	160 deg	190 deg	200 deg	210 deg	220 deg	190 deg	220 deg
1	59.33	59.92	60.75			58.83	58.83	48.33	23.58	12.67		
2	68.70	68.10	70.70			49.60	-5.70			-11.10	57.60	51.80
3	69.60	70.30	72.30			53.20	-18.30			-10.50	54.60	48.90
4	71.52	70.74	70.80		66.50	35.36	2.48			0.80	59.30	59.90
5	74.46	72.46	73.40	70.32	55.06	21.08	11.22			12.36	64.22	63.44
6	72.34	74.44	72.30		64.60	19.90	-1.20			-5.86	58.64	57.22
7	72.00	73.70	72.20			13.80	10.70			6.40		
8	71.80	72.00	71.10			20.20	16.80			13.50		
9	72.90	74.10	74.80			6.30	-5.20			-4.40	59.90	58.20

Sample 1 data

	HRB										HRF	
	62.0	60.5	57.0			57.5	58.0	53.0	21.0	19.0		
	58.0	60.0	57.0			61.0	60.5	53.0	19.5	11.0		
	55.0	58.0	61.5			57.0	61.0	51.5	25.0	11.0		
	59.0	60.5	62.0			60.0	57.5	46.0	27.5	10.0		
	59.0	57.0	63.0			56.5	56.0	43.0	28.5	13.5		
	63.0	63.5	64.0			61.0	60.0	43.5	20.0	11.5		
Average	59.33	59.92	60.75			58.83	58.83	48.33	23.58	12.67		

Sample 2 data

	HRB										HRF	
	68.7	68.1	70.7			49.6	-5.7			-11.1	57.6	51.8
	68.5	69.7	69.6			47.1	-2.5			-14.0	55.9	51.4
	69.1	70.3	70.4			50.1	-3.1			-9.5	57.6	51.1
	68.7	70.6	71.3			45.8	0.2			-10.5	56.9	52.4
	69.1	70.2	71.3			49.1	-6.6			-8.7	56.4	54.9
Average	68.82	69.78	70.66			48.34	-3.54			-10.76	56.88	52.32

Sample 3 data

	HRB										HRF	
	69.6	70.3	72.3			53.2	-18.3			-10.5	54.6	48.9
	71.1	72.0	71.8			50.9	-13.3			-12.1	54.9	48.2
	70.9	72.3	72.6			43.0	-10.1			-12.8	55.1	51.9
	71.1	72.4	72.5			42.8	-9.7			-12.6	55.0	50.6
	71.0	72.6	71.9			53.7	-10.4			-14.1	56.7	48.5
Average	70.74	71.92	72.22			48.72	-12.36			-12.42	55.26	49.62

Sample 4 data

	HRB										HRF	
	72.8	71.1	69.2		66.3	35.0	-4.8			-5.9	59.7	59.1
	69.9	70.9	71.7		66.4	35.6	2.0			1.6	56.3	60.7
	71.0	70.0	71.6		66.1	34.8	4.9			3.9	59.5	60.8
	71.6	71.2	70.8		67.3	34.9	5.1			3.7	60.3	59.2
	72.3	70.5	70.7		66.4	36.5	5.2			0.7	60.7	59.7
Average	71.52	70.74	70.80		66.50	35.36	2.48			0.80	59.30	59.90

Sample 5 data

	HRB										HRF	
	74.1	70.2	73.0	70.5	59.3	22.0	9.0			11.0	62.3	64.0
	74.1	73.3	73.1	70.4	57.5	18.9	12.4			11.3	64.5	63.9
	74.9	73.3	72.9	70.1	56.2	21.0	11.0			11.7	64.7	62.6
	74.9	73.2	74.0	69.8	54.5	23.3	12.2			13.2	63.8	62.4
	74.3	72.3	74.0	70.8	47.8	20.2	11.5			14.6	65.8	64.3
Average	74.46	72.46	73.40	70.32	55.06	21.08	11.22			12.36	64.22	63.44

Sample 6 data

	HRB										HRF	
	71.2	74.9	69.6		63.3	22.3	-9.8			-9.3	58.7	54.3
	72.0	74.6	72.5		62.5	20.0	-1.2			-0.9	60.2	55.4
	72.8	74.3	73.6		63.2	20.5	2.2			-4.1	59.0	57.4
	70.8	74.7	73.6		65.6	19.1	0.5			-9.1	57.0	56.7
	74.9	73.7	72.2		68.4	17.6	2.3			-5.9	58.3	62.3
Average	72.34	74.44	72.30		64.60	19.90	-1.20			-5.86	58.64	57.22

Sample 7 data

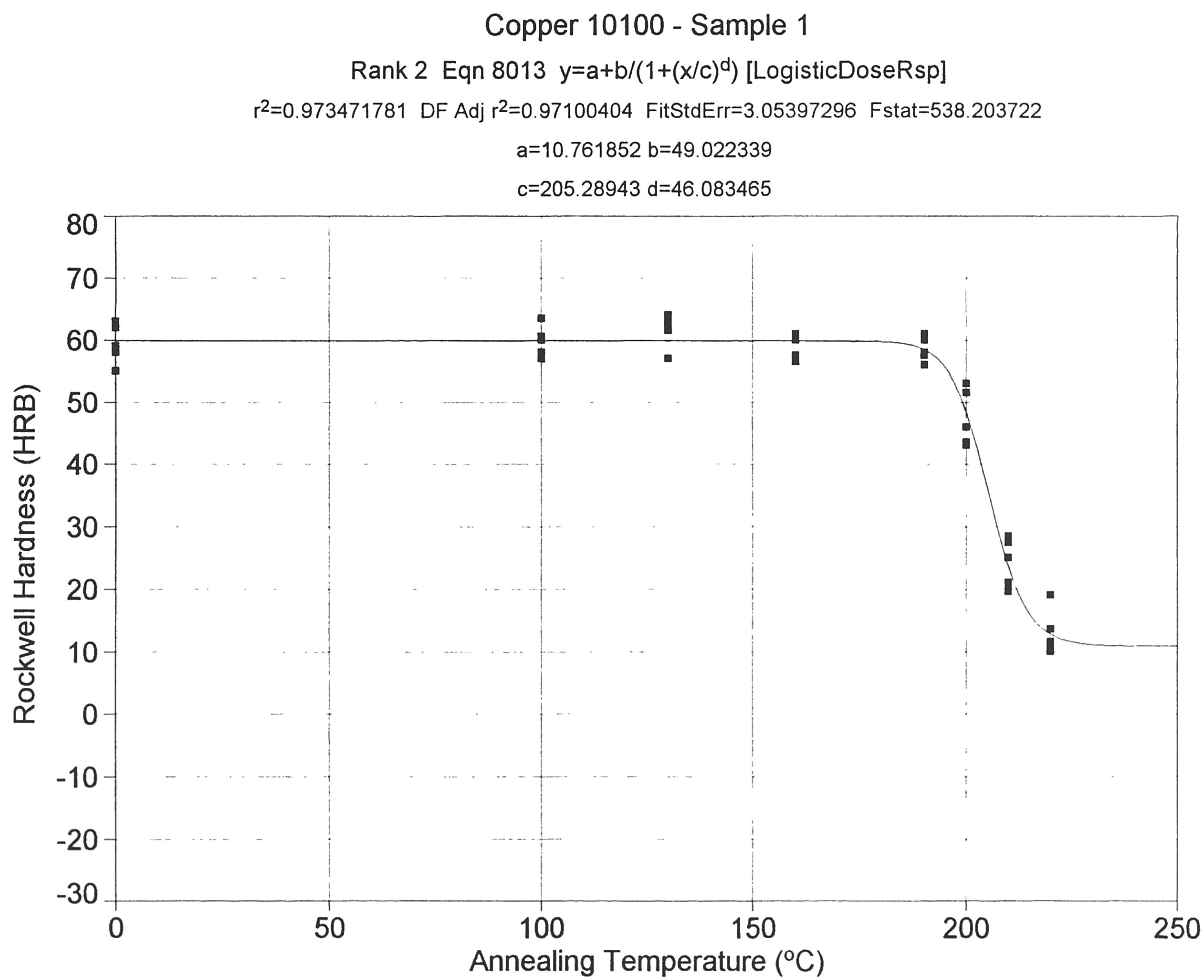
	HRB										HRF	
	72.0	73.7	72.2			13.8	10.7			6.4		
	71.1	74.1	71.9			16.6	10.3			6.2		
	69.6	72.9	72.7			15.9	11.9			5.8		
	71.1	72.3	72.0			17.2	10.1			9.3		
	72.0	72.5	73.3			16.5	13.5			7.7		
Average	71.16	73.10	72.42			16.00	11.30			7.08		

Sample 8 data

	HRB										HRF	
	71.8	72.0	71.1			20.2	16.8			13.5		
	72.2	73.2	71.0			21.2	18.2			14.2		
	72.3	74.0	71.6			21.3	15.6			18.6		
	72.4	73.2	71.9			21.0	16.5			14.1		
	73.1	74.5	71.4			20.6	14.7			19.3		
Average	72.36	73.38	71.40			20.86	16.36			15.94		

Sample 9 data

	HRB										HRF	
	72.9	74.1	74.8			6.3	-5.2			-4.4	59.9	58.2
	73.6	74.1	74.1			5.1	1.3			-1.1	60.0	58.3
	72.3	74.2	74.7			6.7	0.8			-1.8	59.6	59.5
	73.1	74.1	73.0			7.9	0.9			0.2	61.2	61.0
	73.8	75.3	73.1			7.9	1.3			2.0	61.7	59.0
Average	73.14	74.36	73.94			6.78	-0.18			-1.02	60.48	59.20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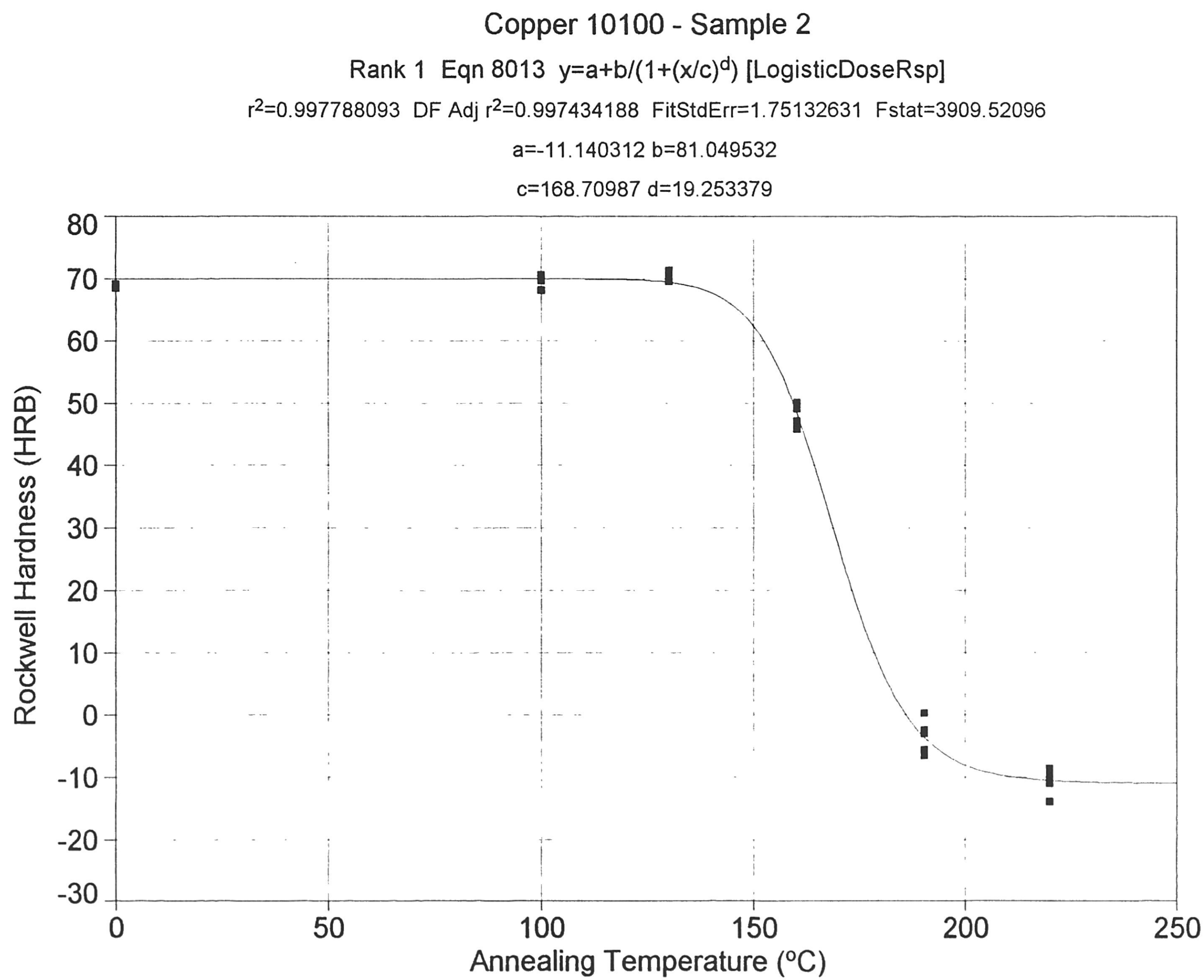
Rank 2 Eqn 8013 y=a+b/(1+(x/c)^d) [LogisticDoseRsp]
r^2=0.973471781 DF Adj r^2=0.97100404 FitStdErr=3.05397296 Fstat=538.203722
a=10.761852 b=49.022339
c=205.28943 d=46.083465
Rockwell Hardness (HRB)
80
70
60
50
40
30
20
10
0
-10
-20
-30
0
50
100
150
200
250
Annealing Temperature (°C)


Copper 10100 - Sample 2
Rank 1 Eqn 8013 y=a+b/(1+(x/c)^d) [LogisticDoseRsp]
r^2=0.997788093 DF Adj r^2=0.997434188 FitStdErr=1.75132631 Fstat=3909.52096
a=-11.140312 b=81.049532
c=168.70987 d=19.253379
Rockwell Hardness (HRB)
80
70
60
50
40
30
20
10
0
-10
-20
-30
0
50
100
150
200
250
Annealing Temperature (°C)

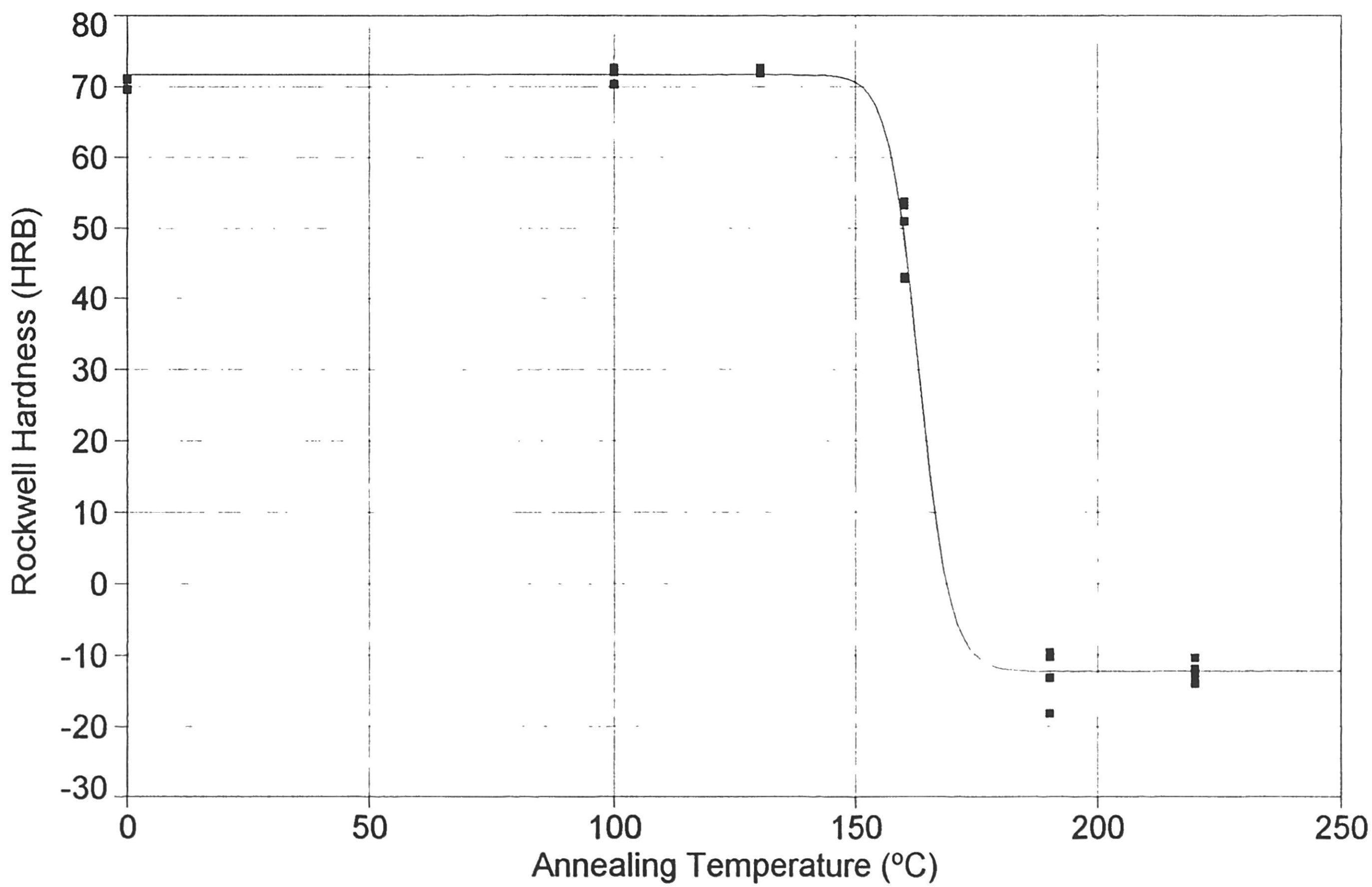
Copper 10100 - Sample 3

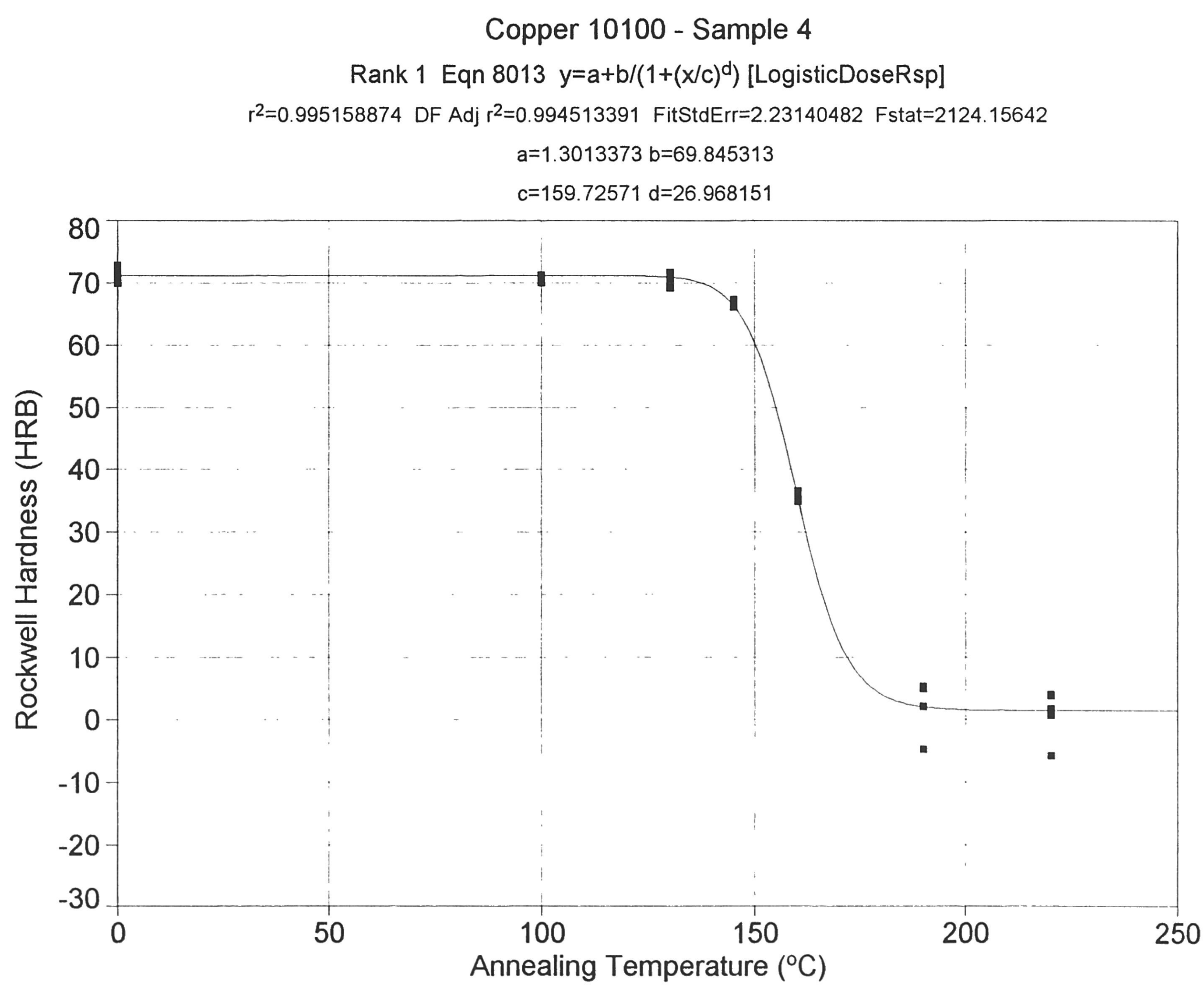
Rank 3 Eqn 8013 $y=a+b/(1+(x/c)^d)$ [LogisticDoseRsp]

r^2=0.995626863 DF Adj r^2=0.994927161 FitStdErr=2.68989194 Fstat=1973.12951

a=-12.404412 b=84.031276

c=163.04049 d=52.137513




Copper 10100 - Sample 4
Rank 1 Eqn 8013 y=a+b/(1+(x/c)^d) [LogisticDoseRsp]
r^2=0.995158874 DF Adj r^2=0.994513391 FitStdErr=2.23140482 Fstat=2124.15642
a=1.3013373 b=69.845313
c=159.72571 d=26.968151
80
70
60
50
40
30
20
10
0
-10
-20
-30
Rockwell Hardness (HRB)
0
50
100
150
200
250
Annealing Temperature (°C)

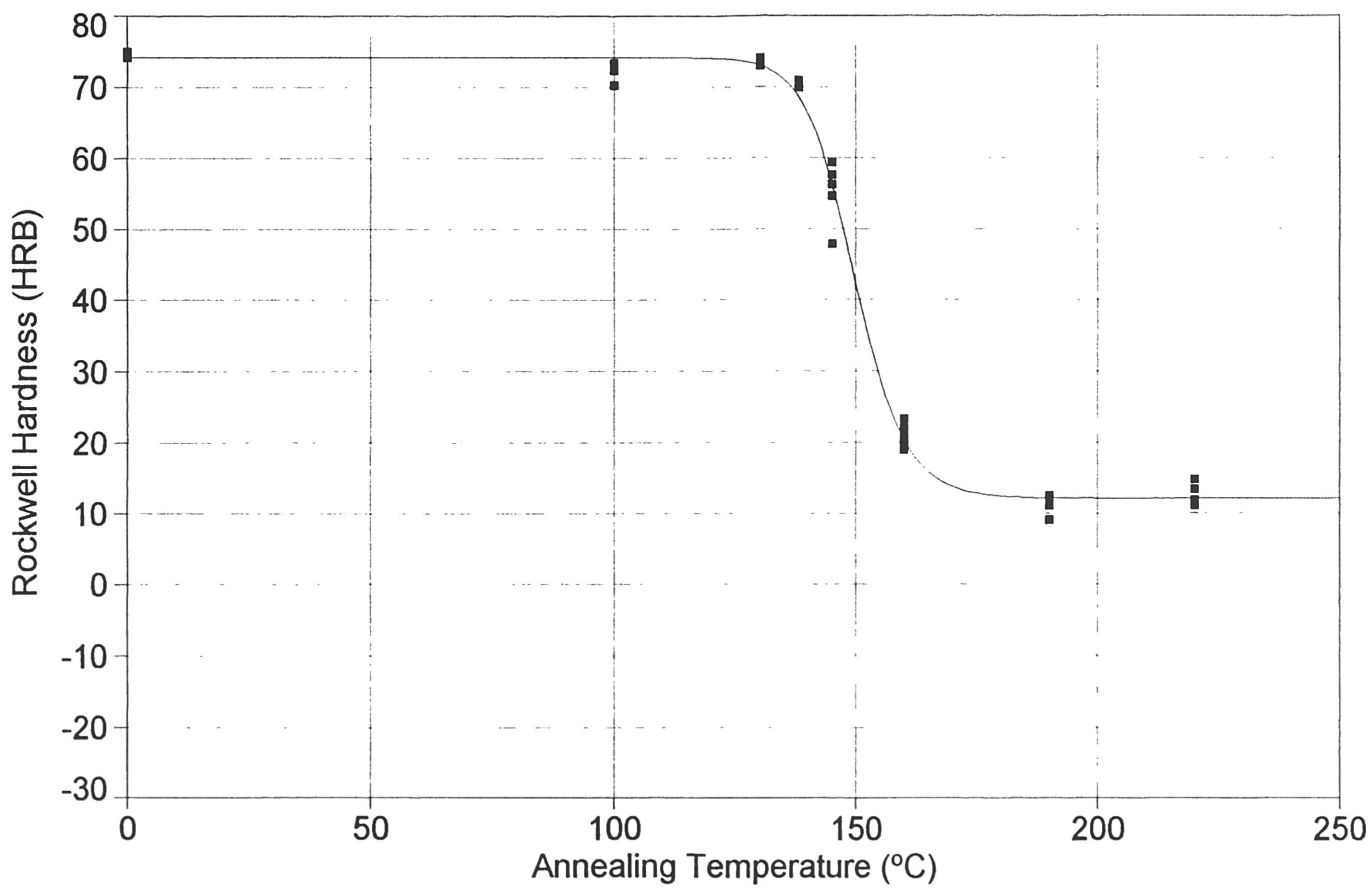
Copper 10100 - Sample 5

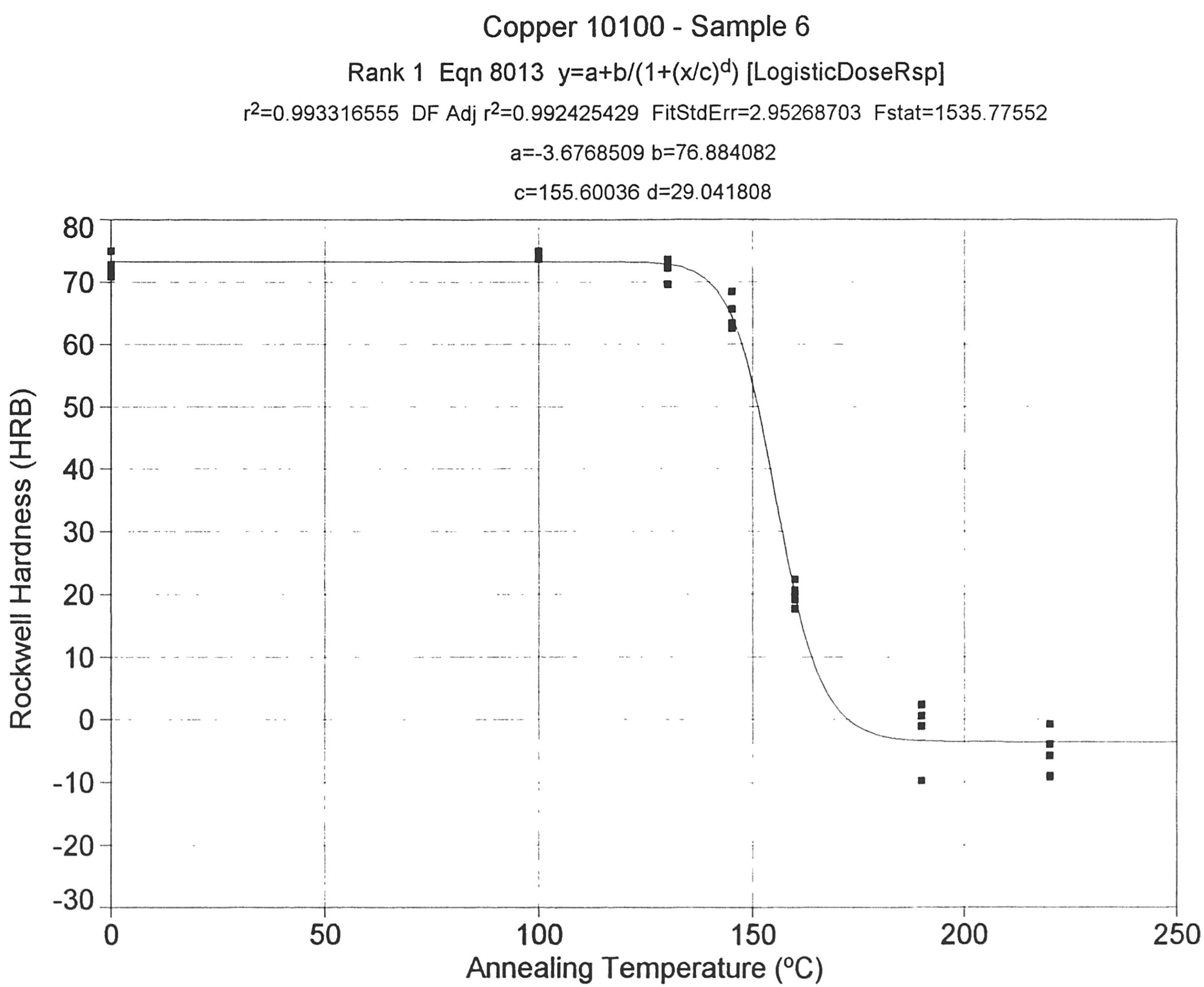
Rank 2 Eqn 8013 $y=a+b/(1+(x/c)^d)$ [LogisticDoseRsp]

r^2=0.994455135 DF Adj r^2=0.993821436 FitStdErr=2.12387969 Fstat=2152.16432

a=11.993789 b=62.129375

c=149.74914 d=28.172322





Copper 10100 - Sample 6
Rank 1 Eqn 8013 $y=a+b/(1+(x/c)^d)$ [LogisticDoseRsp]
r^2=0.993316555 DF Adj r^2=0.992425429 FitStdErr=2.95268703 Fstat=1535.77552
a=-3.6768509 b=76.884082
c=155.60036 d=29.041808
Rockwell Hardness (HRB)
80
70
60
50
40
30
20
10
0
-10
-20
-30
0
50
100
150
200
250
Annealing Temperature (°C)

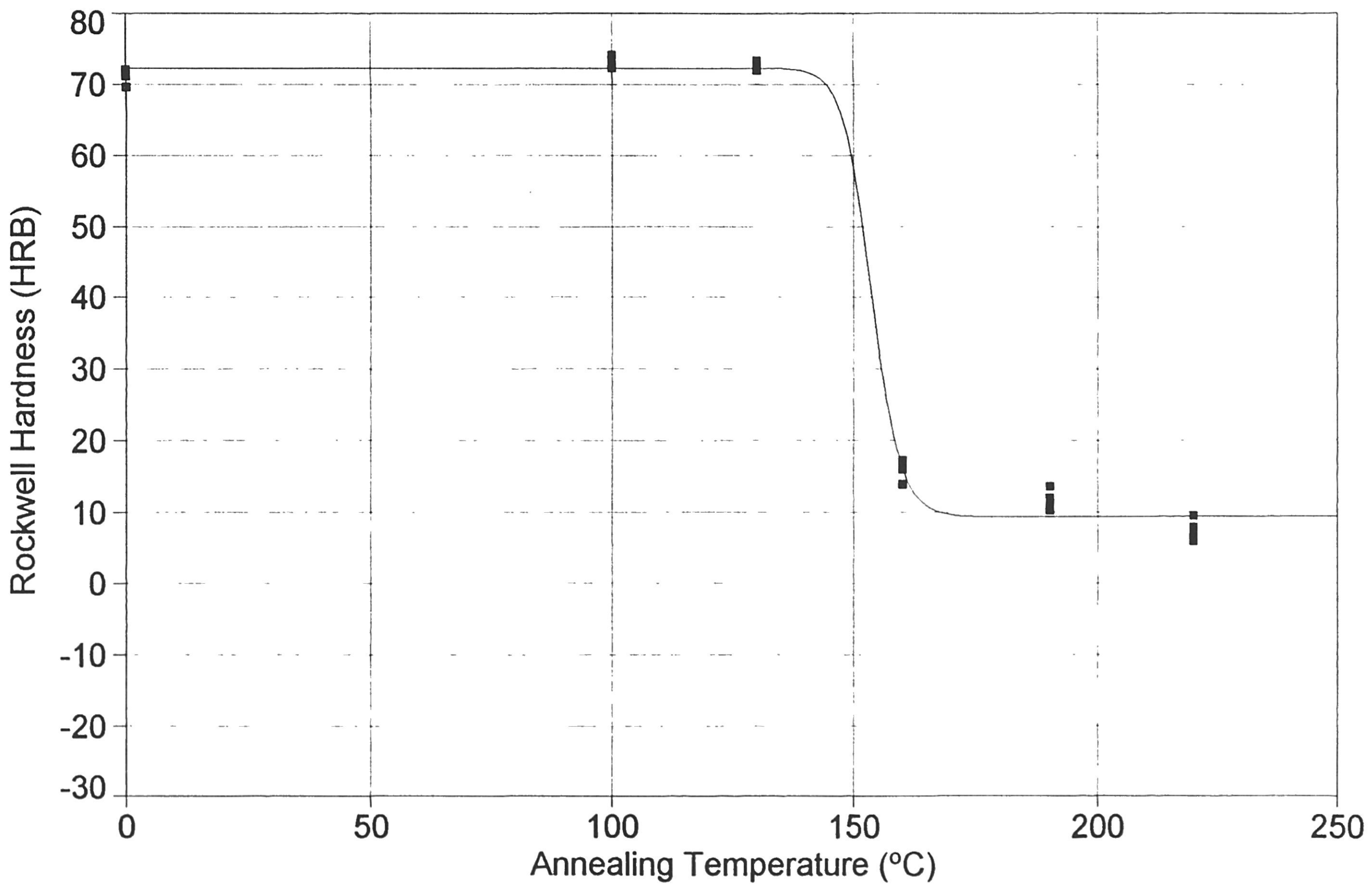
Copper 10100 - Sample 7

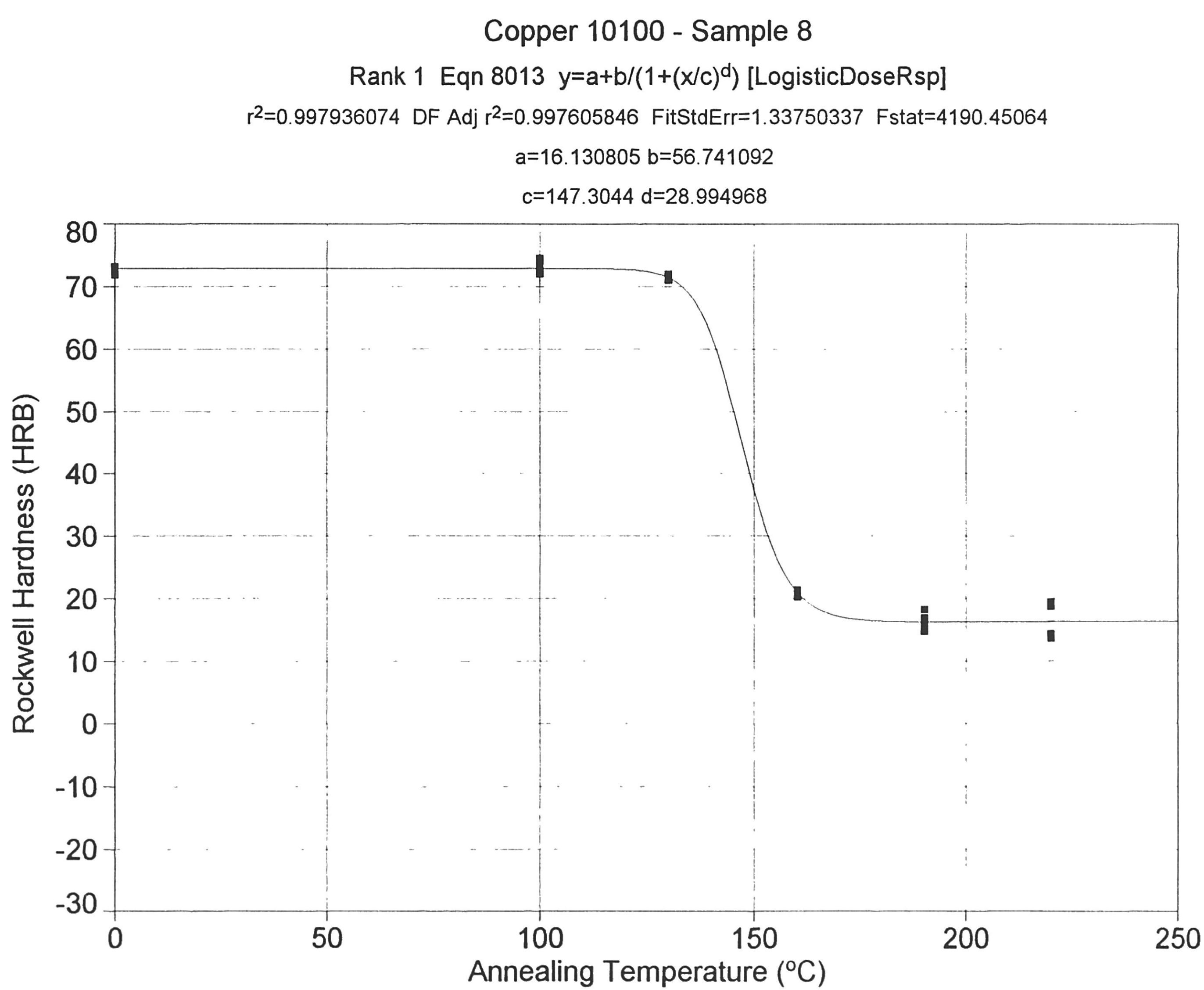
Rank 1 Eqn 8013 $y=a+b/(1+(x/c)^d)$ [LogisticDoseRsp]

$r^2=0.99696075$ DF Adj $r^2=0.99647447$ FitStdErr=1.80701704 Fstat=2842.9144

a=9.1892424 b=63.04082

c=153.649 d=52.103245




Copper 10100 - Sample 8
Rank 1 Eqn 8013 y=a+b/(1+(x/c)^d) [LogisticDoseRsp]
r^2=0.997936074 DF Adj r^2=0.997605846 FitStdErr=1.33750337 Fstat=4190.45064
a=16.130805 b=56.741092
c=147.3044 d=28.994968
Rockwell Hardness (HRB)
80
70
60
50
40
30
20
10
0
-10
-20
-30
0
50
100
150
200
250
Annealing Temperature (°C)

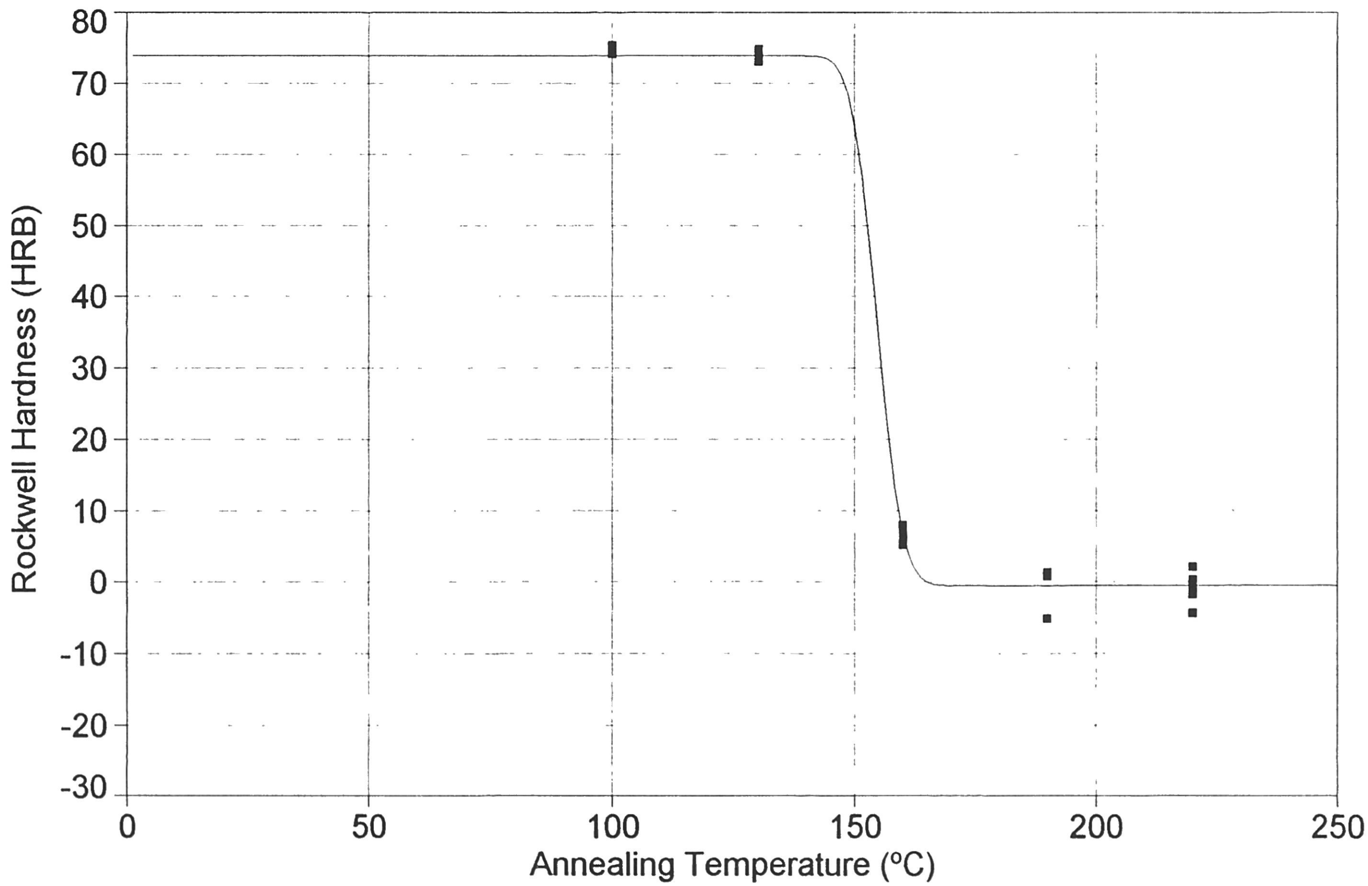
Copper 10100 - Sample 9

Rank 1 Eqn 8001 y=CumLogNorm()

r^2=0.998183689 DF Adj r^2=0.997893079 FitStdErr=1.65233324 Fstat=4762.91005

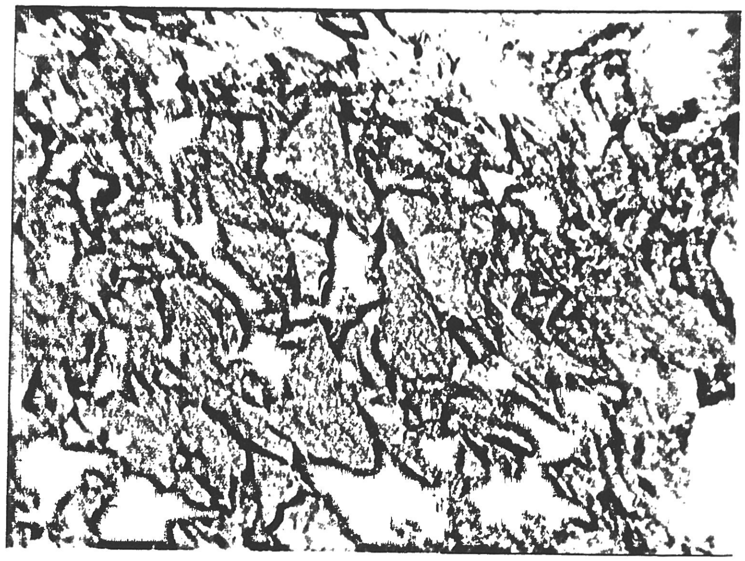
a=-0.6 b=74.413333

c=154.44773 d=0.026895566

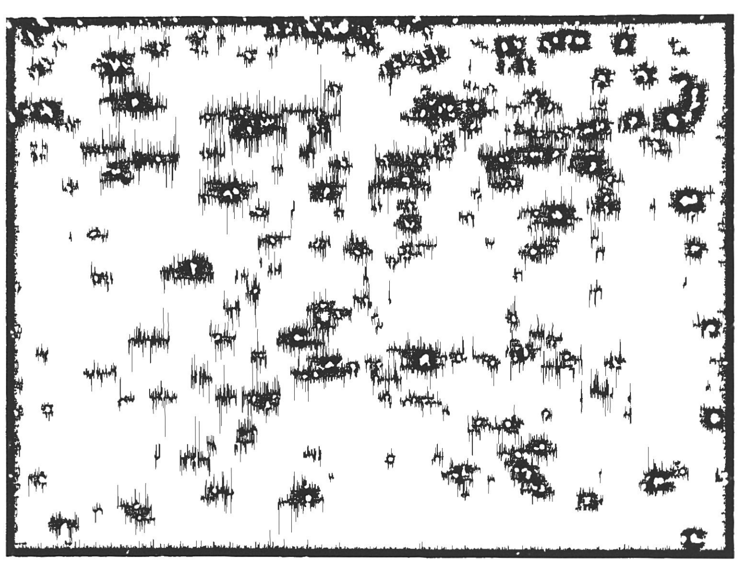


Additional Microstructure Photographs

Magnification=60X

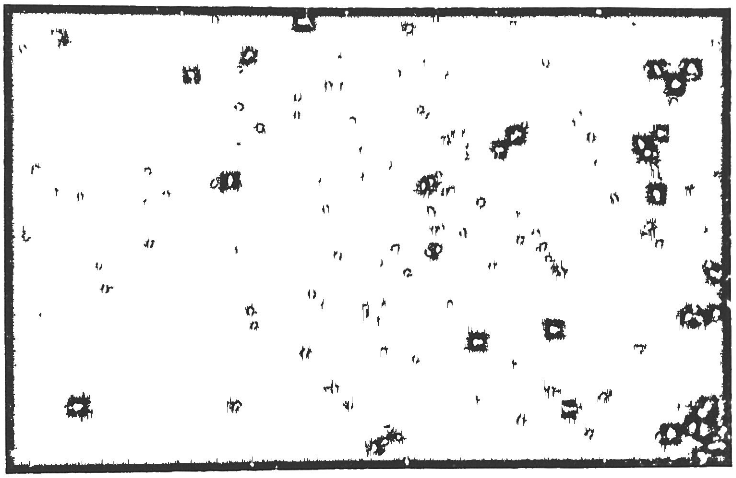


Sample 9 (8 passes, Method C)



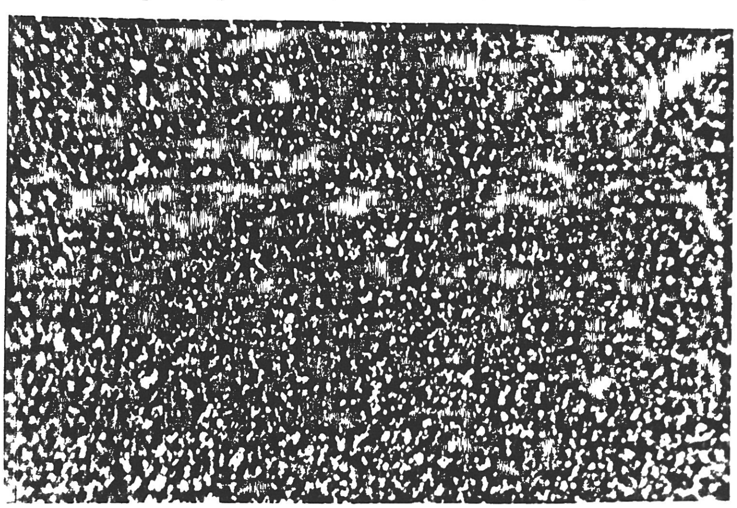
Sample 4 (4 passes, Method A, 220°C)

Magnification = 60X



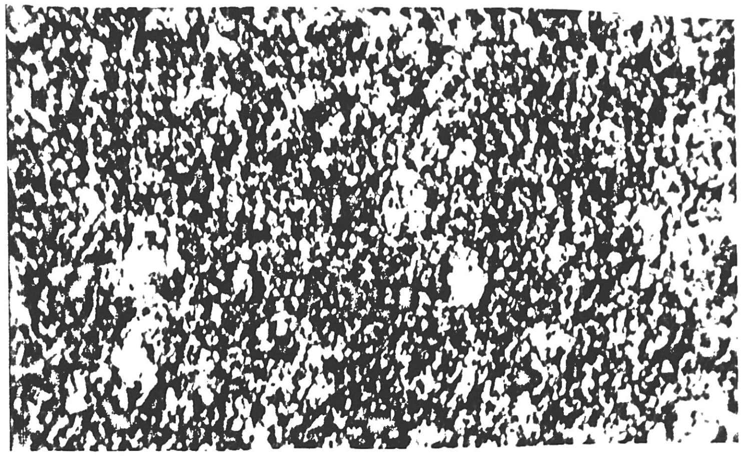
Sample 7 (8 Passes, Method A, 160°C)

Sample 7 (8 Passes, Method A, 190°C)

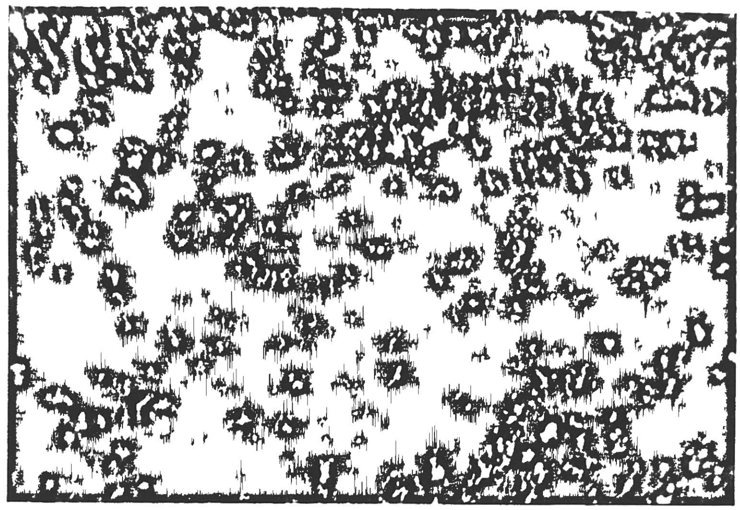


Sample 7 (8 passes, Method A, 220°C)

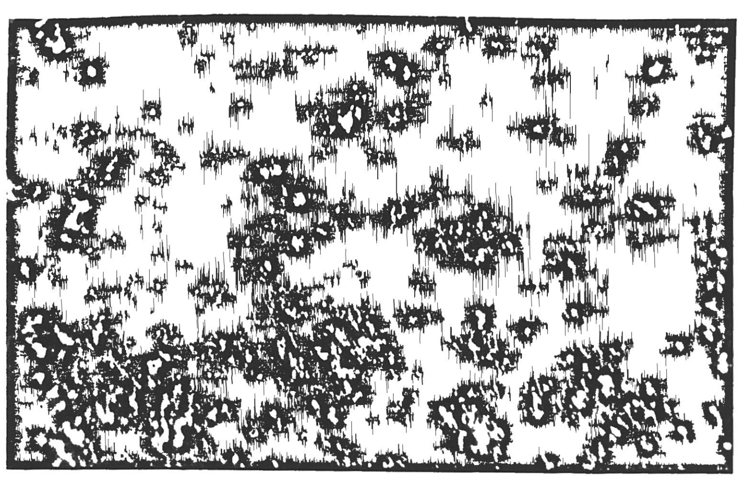
Magnification = 60X



Sample 6 (4 passes, Method C, 220°C)

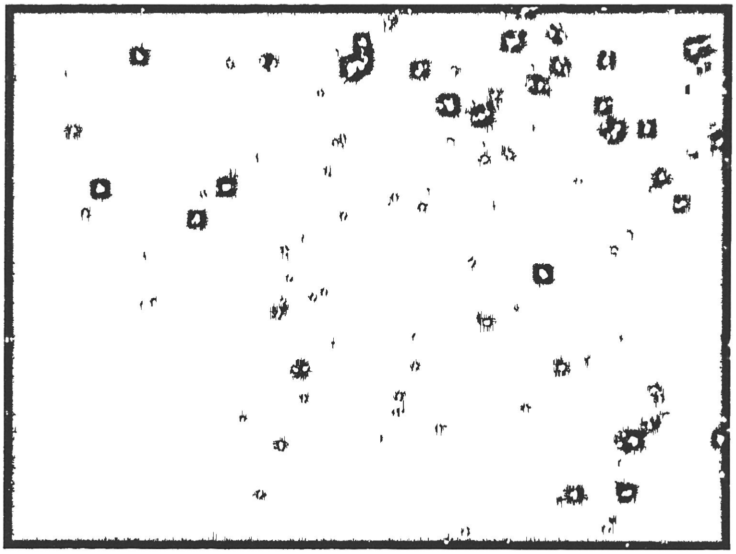


Sample 9 (8 passes, Method C, 160°C)



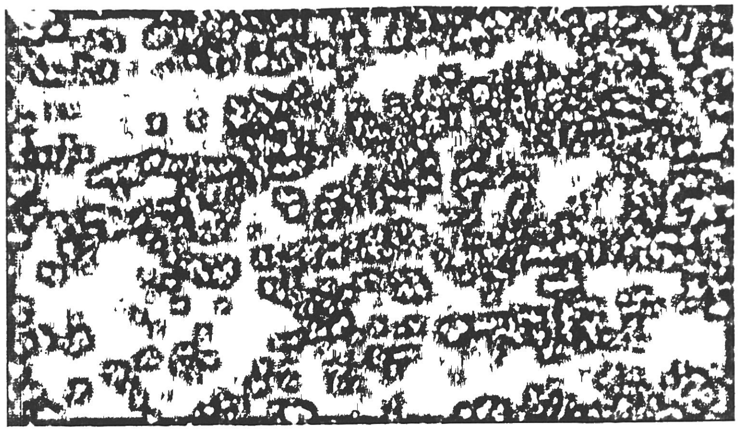
Sample 9 (8 passes, Method C, 190°C)

Magnification = 60X

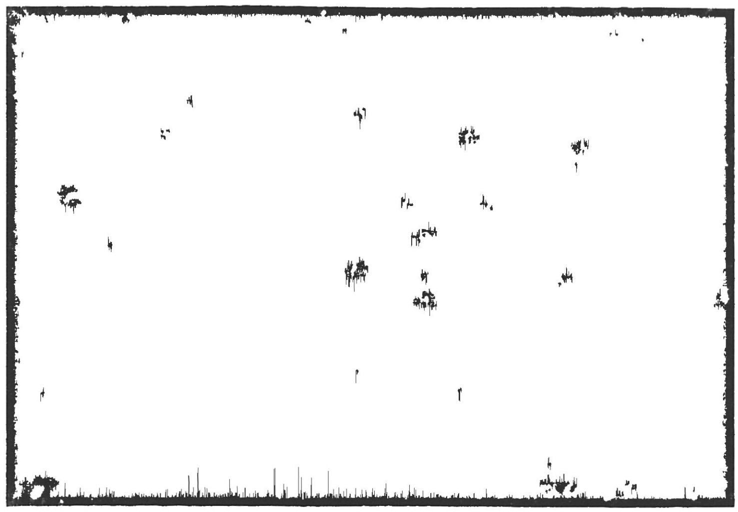


Sample 9 (8 passes, Method C, 220°C)

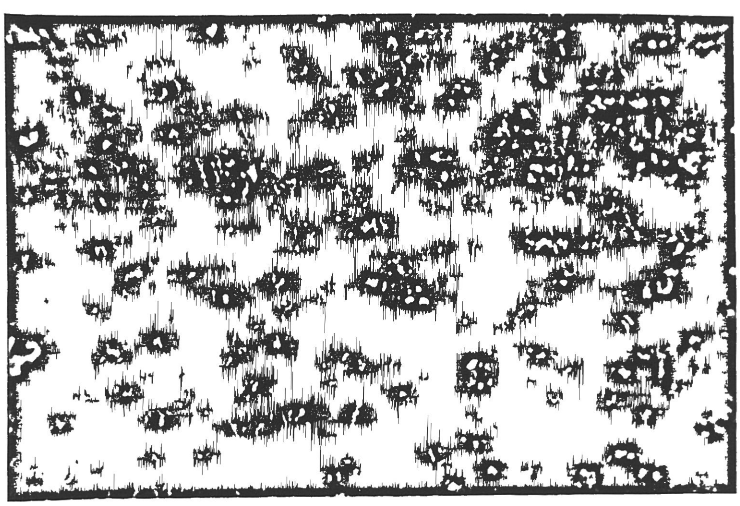
Magnification = 240X



Sample 4 (4 passes, Method A, 160°C)

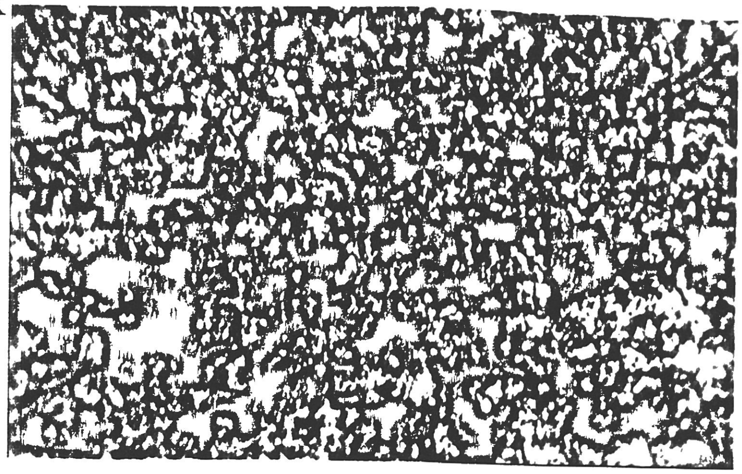


Sample 4 (4 passes, Method A, 190°C)

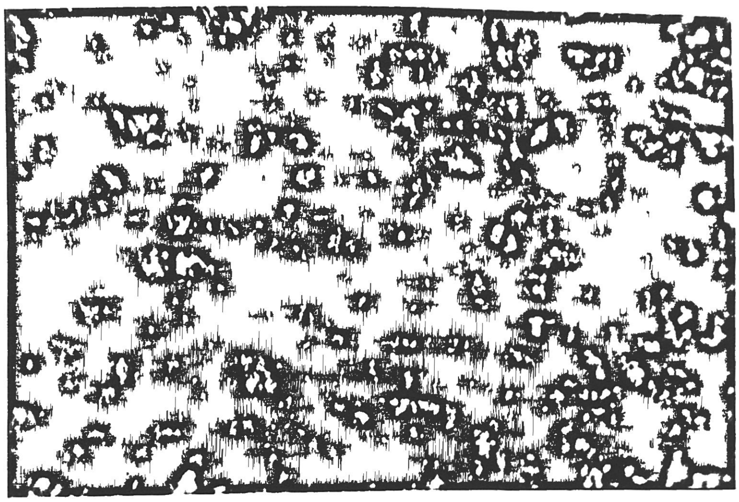


Sample 4 (4 passes, Method A, 220°C)

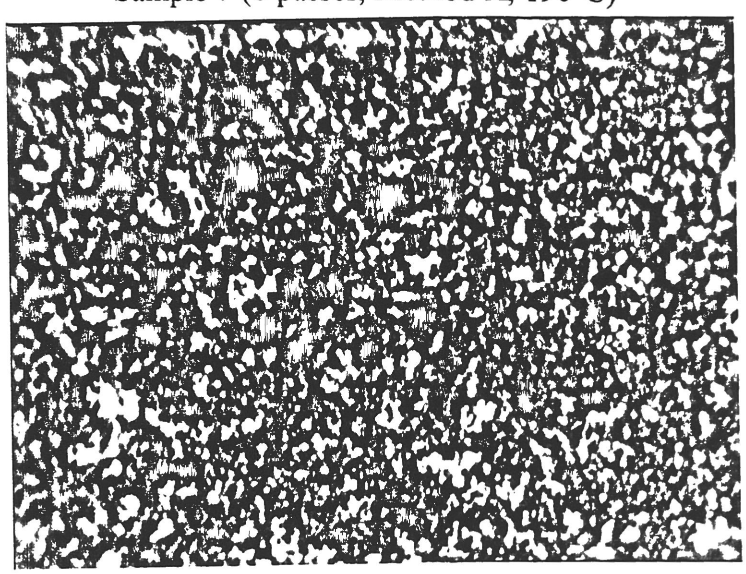
Magnification = 240X



Sample 7 (8 passes, Method A, 160°C)



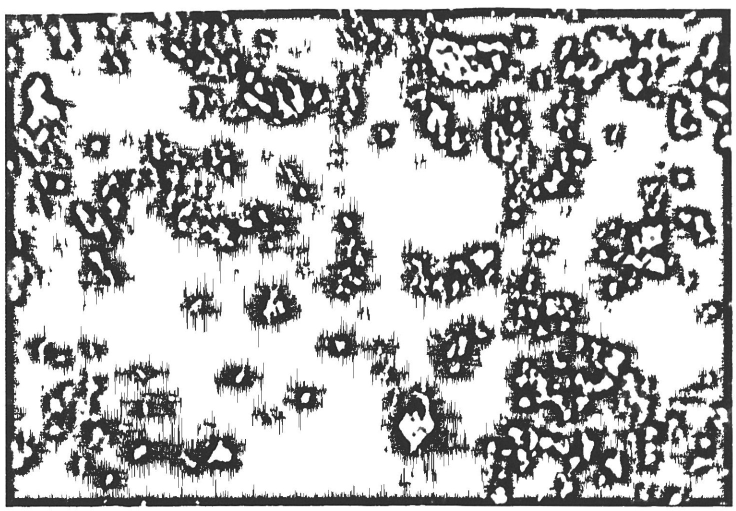
Sample 7 (8 passes, Method A, 190°C)



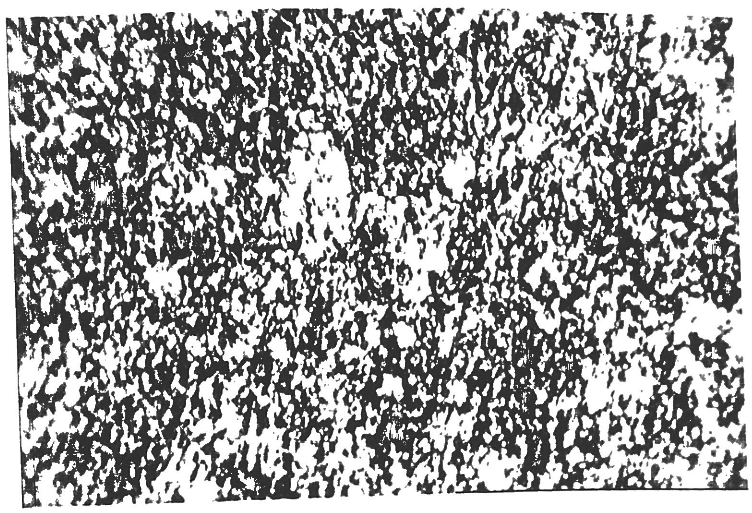
Sample 7 (8 passes, Method A, 220°C)

Magnification = 240X

Sample 6 (4 passes, Method C, 160°C)

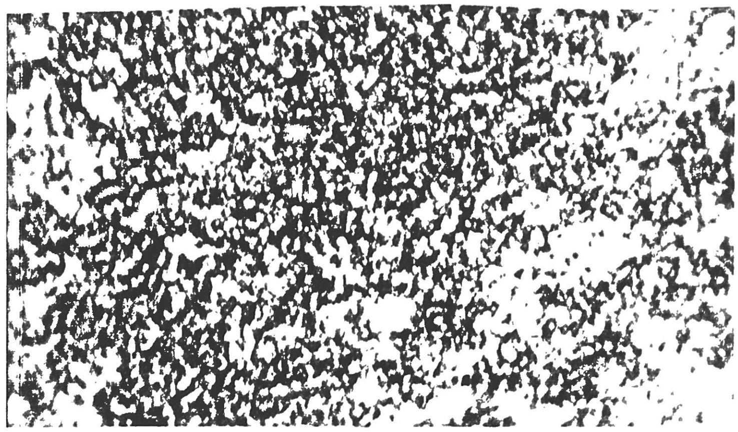


Sample 6 (4 passes, Method C, 190°C)

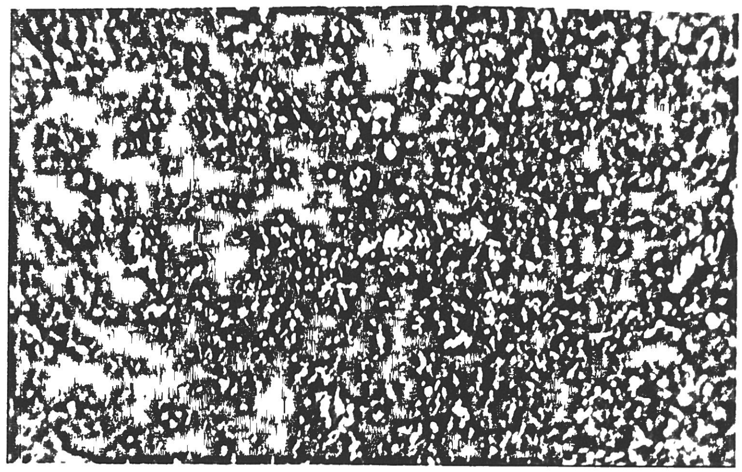


Sample 6 (4 passes, Method C, 220°C)

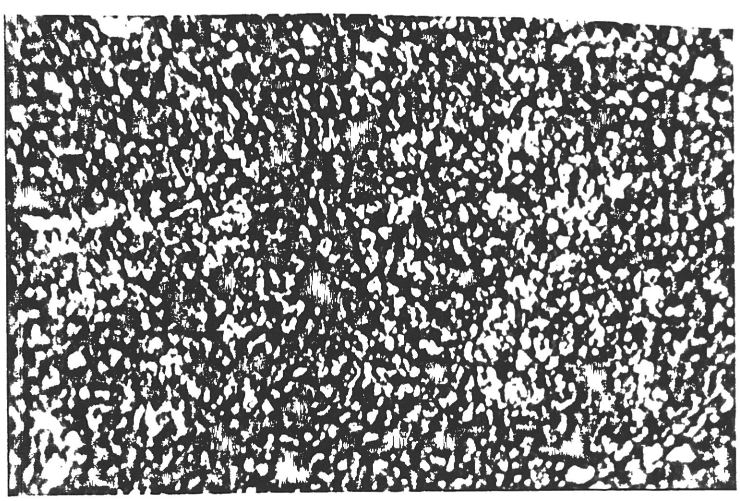
Magnification = 240X



Sample 9 (8 passes, Method C, 160°C)

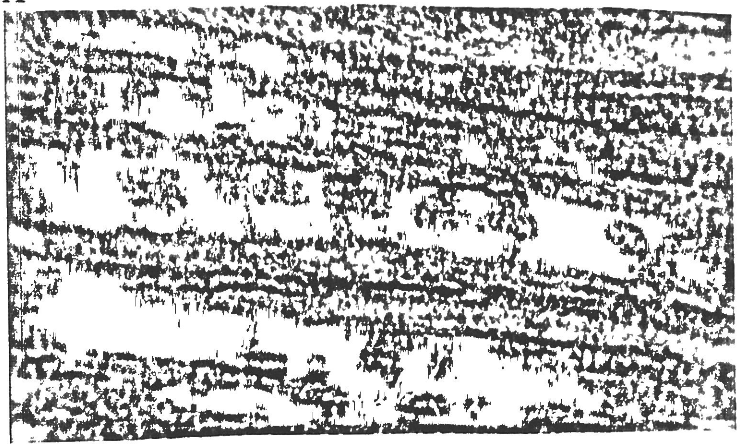


Sample 9 (8 passes, Method C, 190°C)



Sample 9 (8 passes, Method C, 220°C)

Magnification = 240X

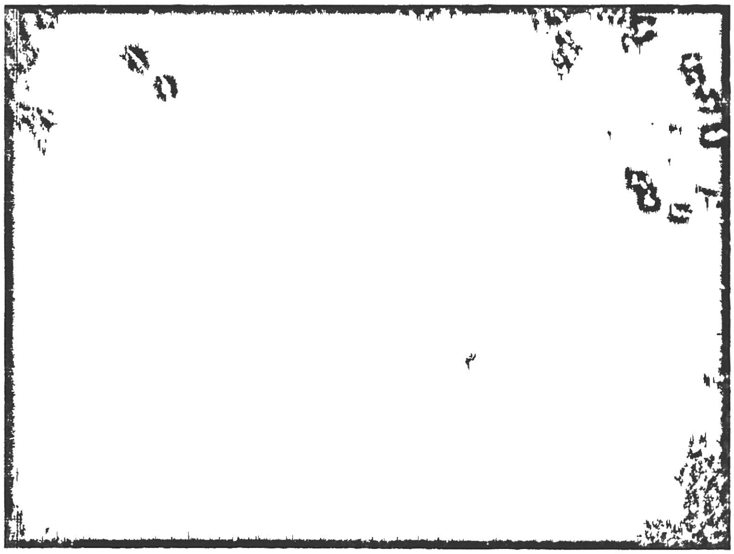


Sample 4 (4 passes, Method A)

Sample 7 (8 passes, Method A)

Sample 6 (4 passes, Method C)

Magnification = 240X



Sample 9 (8 passes, Method C)