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DEPARTMENT OF ENERGY, MINES AND RESOURCES

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EXAMINATION OF COLD-ROLLED HIGH-PURITY NICKEL PRODUCED BY SHERRITT GORDON MINES LIMITED, FOR COIN PRODUCTION

by

C. F. DIXON AND N. S. SPENCE

PHYSICAL METALLURGY DIVISION

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SUMMARY OF RESULTS

The effect of metallurgical properties on the fabrication of fully dense nickel strip prepared by roll compacting powder by Sherritt Gordon Mines Limited was investigated. Microstructure, hardness, and magnetic permeability were determined on two different gauges of strip in three different routes of production to finished gauges, embracing material at from zero (annealed) to 80% cold reduction. Cold reductions of 20 and 30% (depending upon gauge) had no effect on grain size but greater cold reduction resulted in larger grains parallel to the strip surface. The greatest increase in hardness was found to occur with the first 20% cold reduction of annealed material, the thinner gauge being harder for the same amount of cold reduction. Magne Gage permeability tests showed a progressive reduction in magnetic response with increased cold work and resulting hardness.

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INTRODUCTION

In late 1961, due to increasing industrial use, public and commercial hoarding, and other factors, the price of silver came under upward pressure and broke through $1.00 an ounce. By 1964, the price had risen to $1.29 US and was held at this level by the release of US Treasury stocks to satisfy all demands. It became increasingly evident that silver would soon become too expensive for use in common coinage, particularly because consumption for coinage itself was a major cause of short supply for other markets. In the United States, a decision was reached to switch 10¢ and 25¢ coin production to a cupro-nickel clad laminate of copper and to a composite 50¢ coin containing only 40% silver. The Royal Canadian Mint, facing the same problem, instituted a review of the situation. The Committee on New Coinage, chaired by the Master of the Mint, was formed and the Mines Branch representative acted as metallurgical consultant. After considering all relevant factors, the Committee recommended that Canadian coins, hitherto of silver, be made of pure nickel, a decision which was facilitated by the concurrent development of more sophisticated mechanical and electronic coin testing devices for automatic vending machines. These improved devices were designed to accept both existing silver coinage and pure nickel but to reject slugs or counterfeits of the type usually encountered. In the course of this study, it became evident from work in the Electric Engineering Division of NRC, during development of a novel electronic sensing unit, that small variations in magnetic permeability of pure nickel could be very important and that such variations bore a relationship to the amount of cold work done during the production of coin blanks or of coins. It was therefore desirable to investigate this matter in order to provide information which might be of future value in establishing production and quality control over the minting of nickel coinage.

The work, reported here, constitutes a study of the effects of cold work on metallographic structure, hardness, and magnetic permeability of rolled nickel produced by Sherritt Gordon Mines Limited (SGM).

MATERIAL

The material examined was SGM 99.9% nickel strip produced by the roll compacting of nickel powder, sintering at 980°C (1800°F), and hot rolling to 40% reduction. The nickel strip was finished to gauge by the manufacturer by three different procedures, designated as Series A, B, and C.

Series A. Hot-rolled, 0.180-in. gauge, nickel strip was cold rolled to pre-determined intermediate gauges and then given an intermediate anneal for 10 min at 900°C (1650°F) in a hydrogen atmosphere. This was followed by cold rolling to 0%, 10%, 20%, 30%, 40%, and 50% reduction to produce 0.051-in. gauge strip and by cold rolling to 0%, 10%, 20%, 30%, 40%, 50%, 60%, and 70% reduction to produce 0.036-in. gauge strip.

Series B. Hot-rolled, 0.180-in. gauge, nickel strip was cold rolled, without any intermediate anneal, to 0.051-in. gauge strip (71.7% reduction) and to 0.036-in. gauge strip (80% reduction).

Series C. Hot-rolled, 0.090-in. gauge, nickel strip was cold rolled, without any intermediate anneal, to 0.051-in. gauge strip (43.3% reduction) and to 0.036-in. gauge strip (60% reduction).

The grain size and hardness of intermediate annealed and hot-rolled material were supplied by Sherritt Gordon and are listed in Table 1 below.

TABLE 1

Grain Size and Hardness of SGM Nickel Strip

	Grain Size (mm)	Hardness R_{30T}
Series A, Annealed (0.051 Gauge)	0.050	24
Series A, Annealed (0.036 Gauge)	0.032	45
Series B, 0.180-In. Gauge, Hot-Rolled	0.025	35
Series C, 0.090-In. Gauge, Hot-Rolled	0.030	29

PROCEDURES

Sample Preparation

Samples, 1/2 x 2 in., were cut from nickel strips in each metallurgical condition. These samples were sectioned into three specimens as shown in Figure 1.

Polishing and Etching

For metallographic examination and hardness determinations, specimens were mounted in diallyl phthalate, and the surface planes, identified as X, Y and Z in Figure 1, were polished and etched.

Specimens were first ground on 600-grit silicon carbide abrasive papers and then polished on Microcloth with 9-micron and 1/4-micron diamond paste. This was followed by etching in Carapella's reagent for 10 to 15 sec and re-polishing on Gamal cloth with magnesium oxide as the abrasive. The final etch was for 3 to 15 sec in Carapella's reagent.

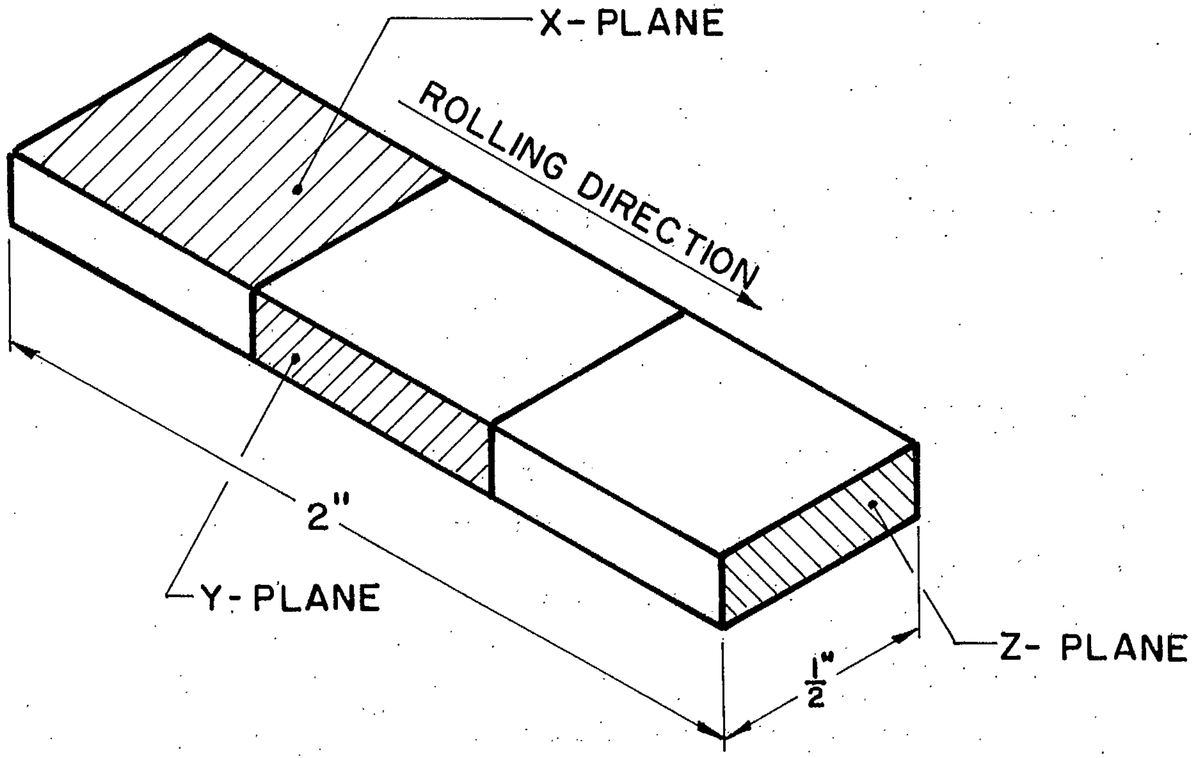


FIGURE 1. NICKEL SAMPLE

Grain Size Determination

Grain sizes were determined on specimens containing equiaxed grains by examining polished and etched surfaces with a Quantitative Television Microscope (QTM). Grains in the X-plane (see Figure 1) of all specimens in the 0.051-in. strip in Series A were measured. Equiaxed structures were also observed in: the Y- and Z-planes of the 0.051-in. strip in Series A that had been cold reduced by 20% or less and by 30% or less, respectively; the X-plane of the 0.051-in. strip in Series C, and in all planes of the 0.036-in. strip in Series A that had been cold reduced by 30% or less.

Degree of Elongation

The degree of elongation of grains was determined on the QTM and is the ratio of their mean linear intercepts taken at right angles to each other.

Hardness Tests

Three superficial hardness tests: Rockwell 30T (R_{30T}), Tukon (Knoop hardness number) using a 0.5-kg load and Vickers (VHN) using a 5-kg load were carried out on polished sections of the mounted specimens. In addition R_{30T} and Vickers hardness values were determined for the X-plane on unmounted specimens.

Magnetic Permeability

The magnetic characteristic of the nickel strips was determined by a Magne Gage (American Instrument Company) which measures the attraction of a small permanent magnet to the material being tested. The test yields relative magnetic permeability on two arbitrary scales. On one scale, the values are inversely proportional to the magnetic permeability and, on the other scale, the values are directly proportional. Values from the latter scale were used in this investigation.

RESULTS AND DISCUSSION

Grain Structure of 0.051-In. Gauge, Nickel Strip

Figures 2 to 13 are photomicrographs of the grain structures of the 0.051-in. specimens in the annealed 0%, 30%, 40%, and 50% cold-rolled (CR) conditions (Series A). In the X-plane, the grains increase in size and become elongated with increased cold reduction although little change is evident until the material is cold rolled 30%. This change in size and shape of grains can be seen by comparing the annealed structure in Figure 2 with the grain structure of specimens cold rolled 30%, 40%, and 50% in Figures 3, 4, and 5, respectively.

The grains in the Y-plane are equiaxed in the annealed specimen (Figure 6) and in specimens cold rolled to 10% and 20% reduction but have a pronounced elongation in the 30%, 40%, and 50% CR specimens as can be seen in Figures 7, 8, and 9, respectively.

In the Z-plane, with cold reductions up to 30%, there is very little change in the shape of the grains (Figure 10, annealed, and Figure 11, 30% CR). Some distortion occurred in specimens cold rolled to 40% and 50% reduction as is shown in Figures 12 and 13, respectively.

Photomicrographs of specimens in Series B show some elongation of grains in the X-plane, Figure 14, and the effect of cold rolling from 0.180-in. to 0.051-in. gauge (71.7% CR) is evident in the worked structure in the Y-plane shown in Figure 15. The structure in the Z-plane was difficult to resolve (Figure 16).

The 43.3% cold reduction of specimens in Series C had little effect on the grains in the X-plane as is evident in Figure 17; however, there is a worked structure in the Y- and Z-planes (Figures 18 and 19, respectively).

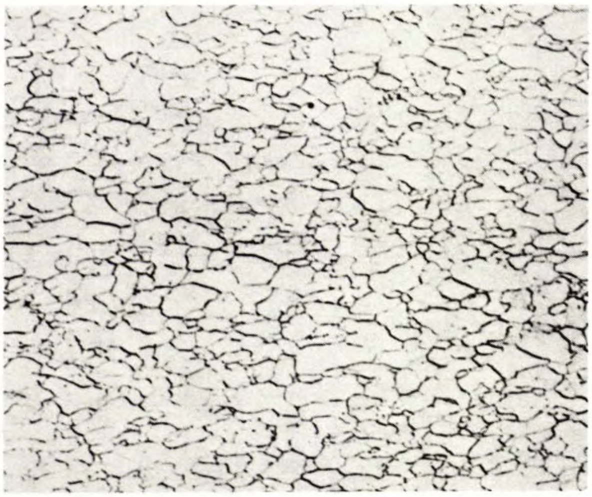
Table 2 lists the ASTM grain sizes and degrees of elongation for specimens in each series. The distortion of the grain structure in specimens with high percentages of cold work is evident in the degree of elongation found. This distortion of the grains in the Y-plane is indicated by comparing the 1:1 ratio of elongation for grains in the 10% CR specimens to the 1.6:1 ratio of elongation for grains in specimens cold rolled to 50% reduction.

X 100

Figure 2. 0.051-In. Gauge, Nickel Strip, Series A, Ann, X-Plane, ASTM Grain Size-8.

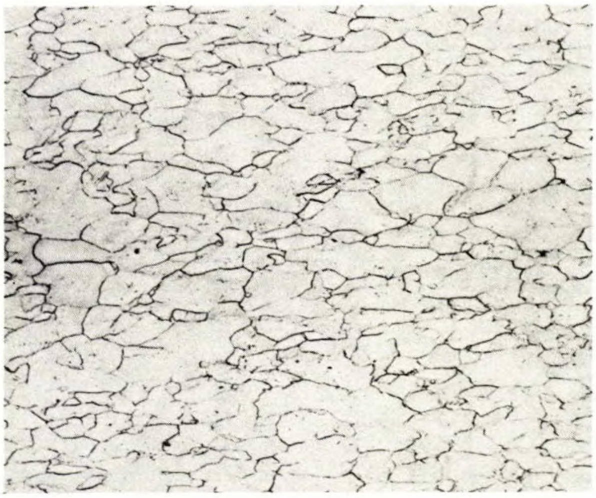
X 100

Figure 3. 0.051-In. Gauge, Nickel Strip, Series A, 30% CR, X-Plane, ASTM Grain Size-7.5.



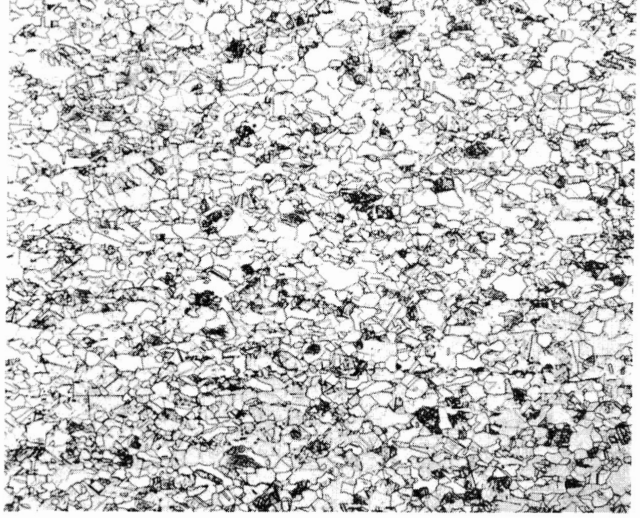
X 100

Figure 4. 0.051-In. Gauge, Nickel Strip, Series A, 40% CR, X-Plane, ASTM Grain Size-7.



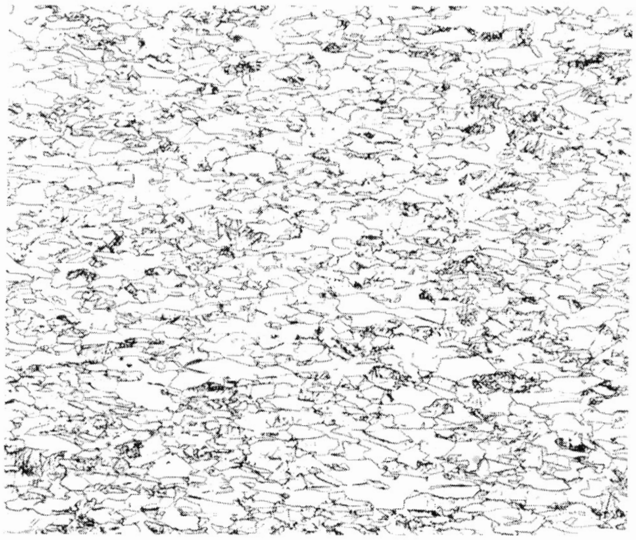
X 100

Figure 5. 0.051-In. Gauge, Nickel Strip, Series A, 50% CR, X-Plane, ASTM Grain Size-6.5.



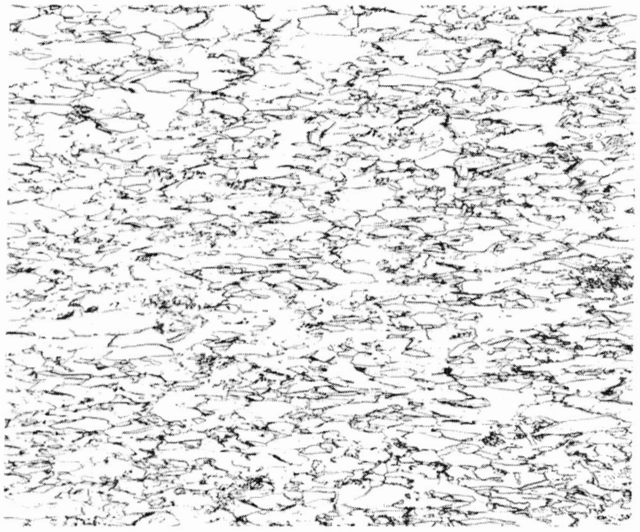
X 100

Figure 6. 0.051-In. Gauge, Nickel Strip, Series A, Ann, Y-Plane, ASTM Grain Size-7.5.



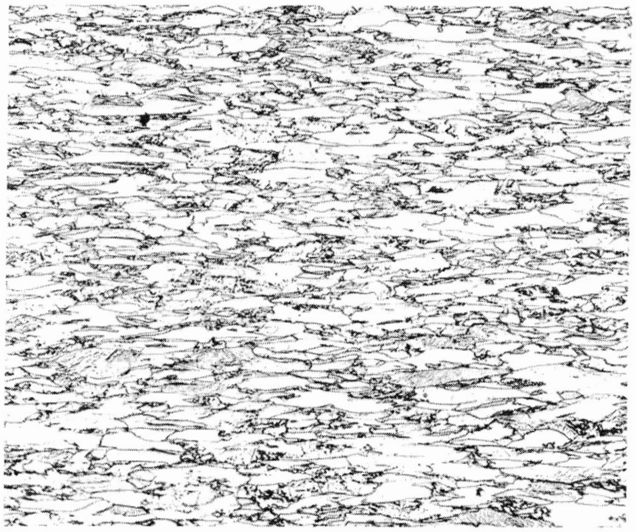
X 100

Figure 7. 0.051-In. Gauge, Nickel Strip, Series A, 30% CR, Y-Plane.



X 100

Figure 8. 0.051-In. Gauge, Nickel Strip, Series A, 40% CR, Y-Plane.



X 100

Figure 9. 0.051-In. Gauge, Nickel Strip, Series A, 50% CR, Y-Plane.

X 100

Figure 10. 0.051-In. Gauge, Nickel Strip, Series A, Ann, Z-Plane, ASTM Grain Size-7.5

X 100

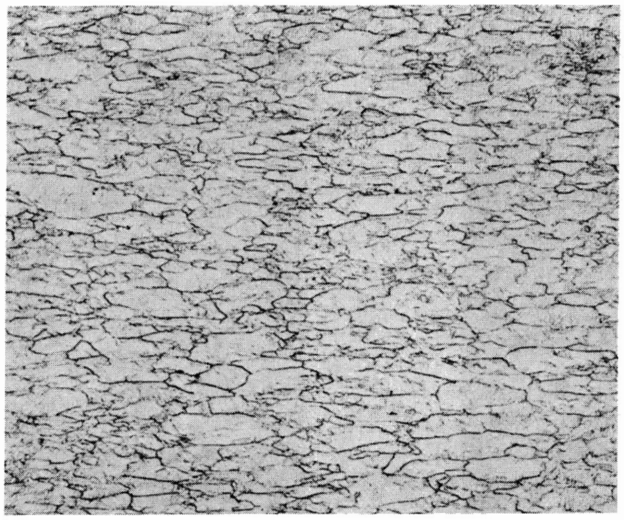
Figure 11. 0.051-In. Gauge, Nickel Strip, Series A, 30% CR, Z-Plane, ASTM Grain Size-8.

X 100

Figure 12. 0.051-In. Gauge, Nickel Strip, Series A, 40% CR, Z-Plane.

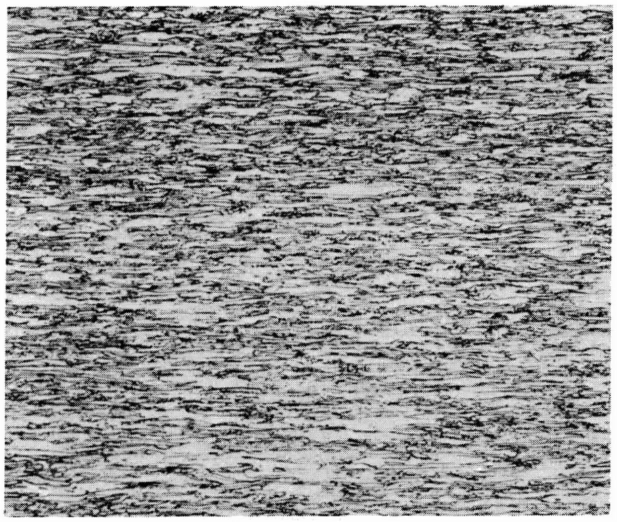
X 100

Figure 13. 0.051-In. Gauge, Nickel Strip, Series A, 50% CR, Z-Plane.



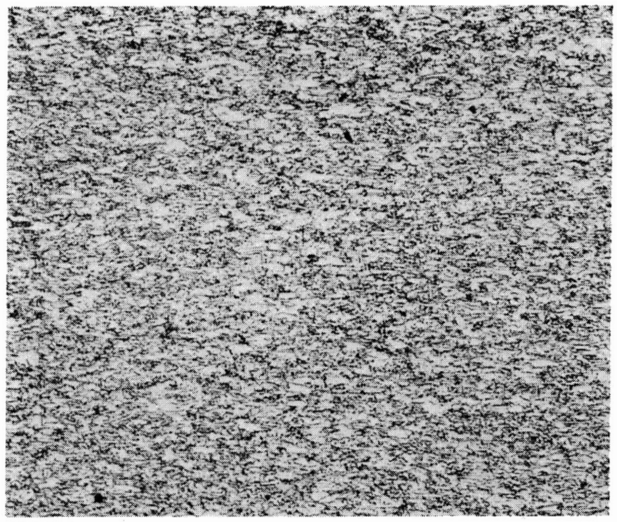
X 100

Figure 14. 0.051-In. Gauge, Nickel Strip, Series B, 71.7% CR, X-Plane.



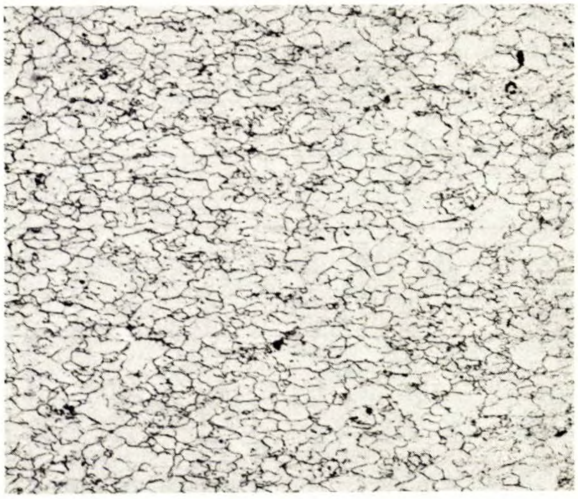
X 100

Figure 15. 0.051-In. Gauge, Nickel Strip, Series B, 71.7% CR, Y-Plane.



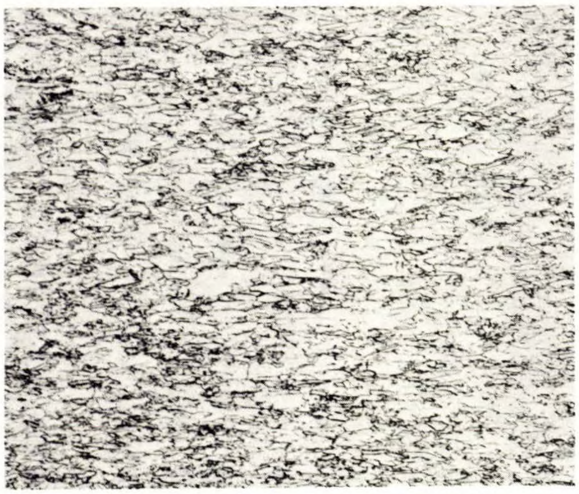
X 100

Figure 16. 0.051-In. Gauge, Nickel Strip, Series B, 71.7% CR, Z-Plane.



X 100

Figure 17. 0.051-In. Gauge, Nickel Strip, Series C, 43.3% CR, X-Plane, ASTM Grain Size-7.5



X 100

Figure 18. 0.051-In. Gauge, Nickel Strip, Series C, 43.3% CR, Y-Plane.

X 100

Figure 19. 0.051-In. Gauge, Nickel Strip, Series C, 43.3% CR, Z-Plane.

TABLE 2

ASTM Grain Size and Degree of Elongation
in 0.051-in. Nickel Strip

Identification	X-Plane		Y-Plane		Z-Plane	
	Grain Size	Degree of Elongation	Grain Size	Degree of Elongation	Grain Size	Degree of Elongation
Series A Ann	8	1:1	7.5	1.2:1	7.5	1.1:1
10% CR	7.5	1.1:1	8	1.1:1	8	1.1:1
20% CR	8	1.2:1	7.5	1.2:1	8	1.1:1
30% CR	7.5	1.4:1	-	1.5:1	8	1.1:1
40% CR	7	1.3:1	-	1.5:1	-	1.2:1
50% CR	6.5	1.3:1	-	1.6:1	-	1.2:1
Series B 71.7% CR	-	1.8:1	-	-	-	-
Series C 43.3% CR	7.5	1.2:1	-	-	-	-

Grain Structure of 0.036-In. Gauge, Nickel Strip

There is no change in the microstructure of the 0.036-in. strip until the material is reduced by 40% (Series A). Structures in the three planes of annealed specimens shown in Figures 20, 21, and 22 (X-, Y-, and Z-planes, respectively) are typical of both annealed and 30% CR material.

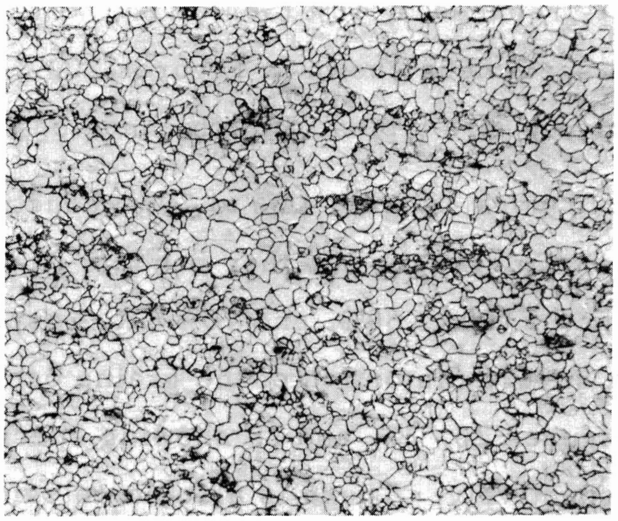
After cold working from 40% to 70% reduction, the size of the grains in the X-plane increases and they become elongated as is shown in Figures 23, 24, and 25 (50%, 60%, and 70% CR, respectively). As in the 0.051-in. strip, a severe distortion of the grains occurs in the Y-plane and is illustrated in Figures 26, 27, and 28 showing specimens cold rolled by 50%, 60%, and 70%, respectively. There is also some evidence of cold work in the structures of 40% to 70% CR specimens in the Z-plane.

In Series B, there are elongated grains in the X-plane, and the Y- and Z-planes have cold worked structures.

The grains in the X-plane of specimens in Series C (Figure 29) are not as large as those in the X-plane of specimens in Series A with the same percentage of cold work (60%, Figure 24). This is possibly due to a difference in grain size in the strip before cold reduction because specimens in Series C were not subjected to an intermediate anneal. The Y- and Z-planes in this series contain worked structures.

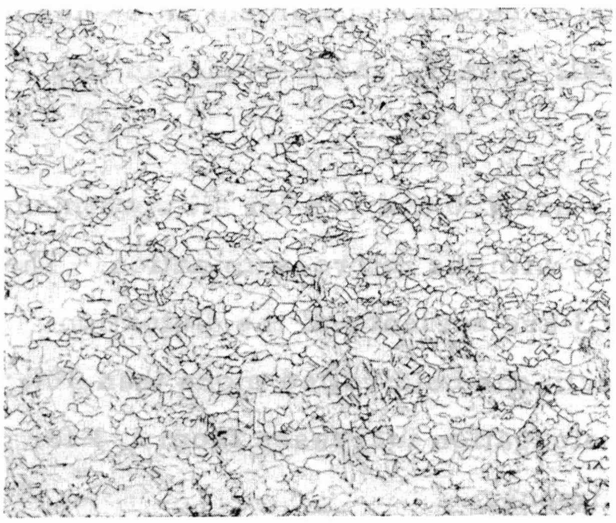
The ASTM grain sizes in Table 3 are 7.0 and 7.5 for all specimens cold rolled up to 30%. This is in agreement with the microexamination. The degrees of grain elongation listed in Table 3 are evidence of the distortion of the grains in the X- and Y-planes in specimens cold rolled more than 30% (Series A). In the Y-plane, the degree of elongation increases from 1.3 for 30% CR specimens to 3.2 for 70% CR specimens.

As in the 0.051-in. strip, the degree of elongation for grains in the Y- and Z-planes of specimens in Series B and C could not be determined because of their worked structure.



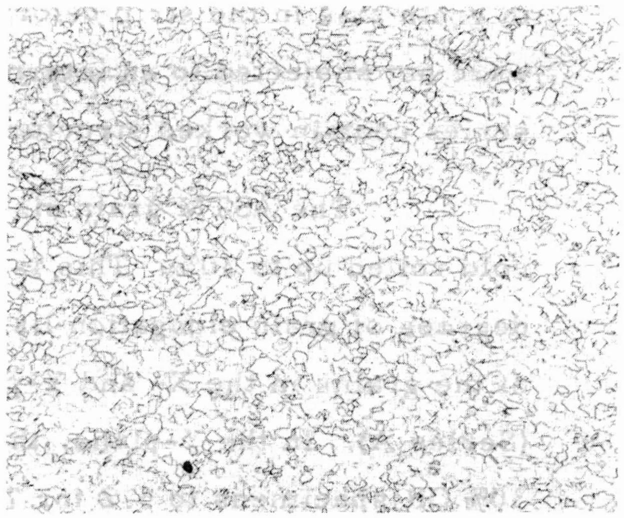
X 100

Figure 20. 0.036-In. Gauge, Nickel Strip, Series A, Ann, X-Plane, ASTM Grain Size-7.5.



X100

Figure 21. 0.036-In. Gauge,Nickel Strip, Series A, Ann, Y-Plane, ASTM Grain Size-7.0.

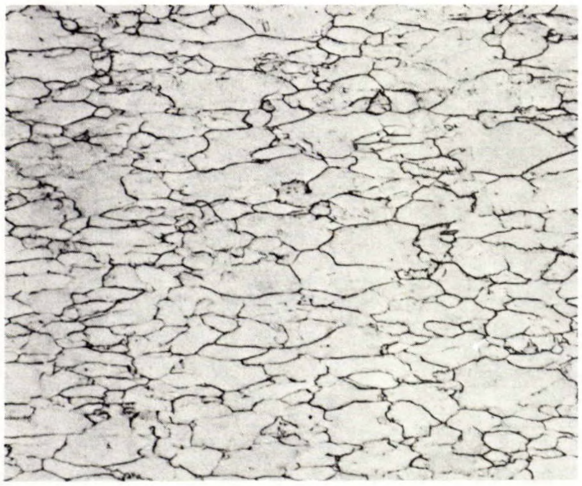


X 100

Figure 22. 0.036-In. Gauge,Nickel Strip, Series A, Ann, Z-Plane, ASTM Grain Size-7.0.

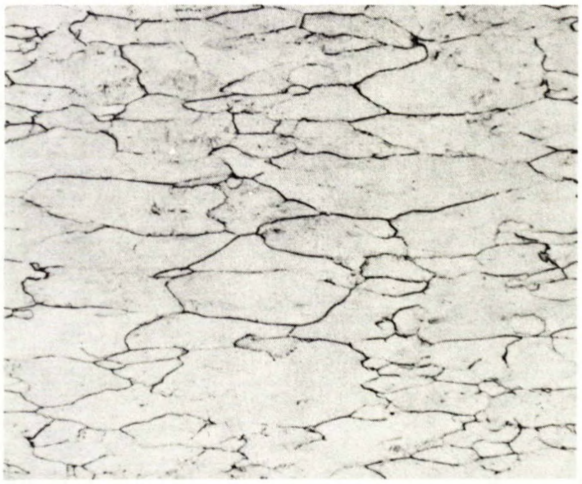
X 100

Figure 23. 0.036-In. Gauge,Nickel Strip, Series A, 50% CR, X-Plane.



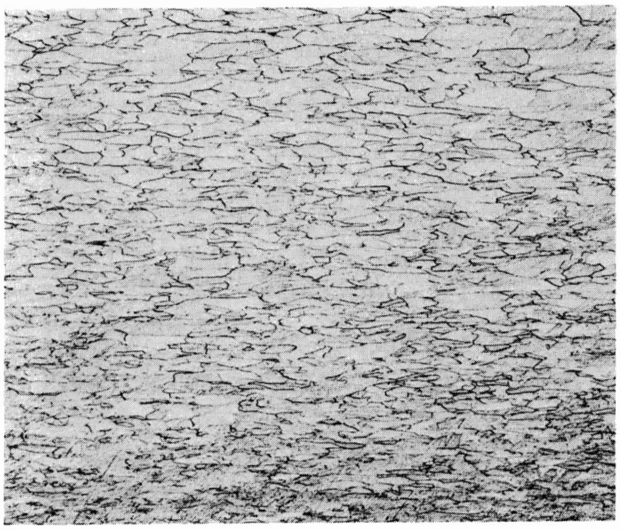
X 100

Figure 24. 0.036-In. Gauge, Nickel Strip, Series A, 60% CR, X-Plane.



X 100

Figure 25. 0.036-In. Gauge, Nickel Strip, Series A, 70% CR, X-Plane.

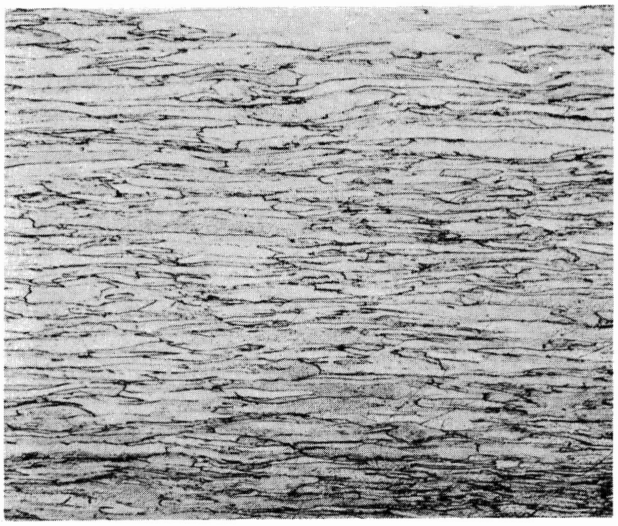


X 100

Figure 26. 0.036-In. Gauge, Nickel Strip, Series A, 50% CR, Y-Plane.

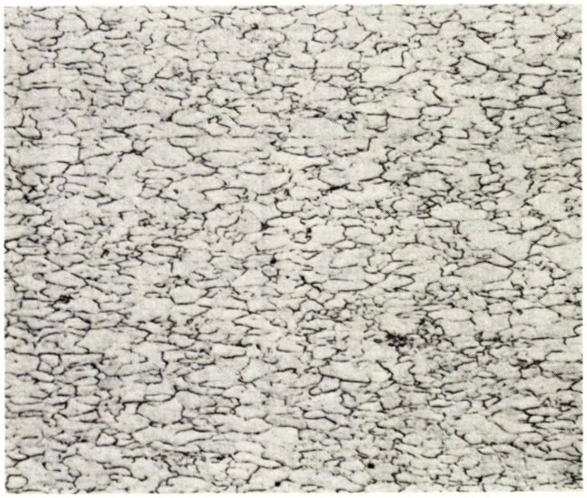
X 100

Figure 27. 0.036-In. Gauge, Nickel Strip, Series A, 60% CR, Y-Plane.



X 100

Figure 28. 0.036-In. Gauge, Nickel Strip, Series A, 70% CR, Y-Plane.



X 100

Figure 29. 0.036-In. Gauge, Nickel Strip, Series C, 60% CR, X-Plane.

TABLE 3

ASTM Grain Size and Degree of Elongation in 0.036-In. Nickel Strip

Identification	X-Plane		Y-Plane		Z-Plane	
	Grain Size	Degree of Elongation	Grain Size	Degree of Elongation	Grain Size	Degree of Elongation
Series A Ann	7.5	1:1	7.0	1.2:1	7.0	1.1:1
10% CR	7.0	1.3:1	7.0	1.2:1	7.0	1.2:1
20% CR	7.5	1.2:1	7.5	1.2:1	7.5	1.1:1
30% CR	7.5	1.2:1	7.0	1.3:1	7.0	1.2:1
40% CR	-	1.6:1	-	1.7:1	-	1.1:1
50% CR	-	1.5:1	-	1.5:1	-	1.4:1
60% CR	-	1.9:1	-	1.7:1	-	-
70% CR	-	1.2:1	-	3.2:1	-	1.5:1
Series B 80% CR	-	1.4:1	-	-	-	-
Series C 60% CR	-	1.4:1	-	-	-	-

In summary, cold rolling evidently deforms the originally equiaxed grains to a flattened pancake-like form. This results in an apparent increase in grain size in the plane of rolling but the grain volume remains constant.

It should be appreciated that materials examined in this report were received in the annealed and cold-rolled conditions. Grain size at the intermediate anneal gauge was accepted as being the same in all cases.

Superficial Hardness of 0.051-In. Gauge, Nickel Strip

Results of superficial hardness tests for the 0.051-in. strip are listed in Table 4. All tests were first carried out on mounted specimens but erratic results for the R_{30T} tests on the X-plane suggested that the 0.051-in. strip was too thin for this test on flat-mounted specimens. Therefore, the R_{30T} hardness results listed for the X-plane are those carried out on unmounted nickel strip. Vickers hardness tests on the X-plane were done on both the mounted and unmounted strip; the hardness values are similar.

Vickers hardness values for the three planes are reasonably consistent for each increment of cold reduction; however, in the results of the R_{30T} and Tukon tests there is some scatter for the values among the planes. Average values for the X-, Y-, and Z-planes, in each specimen for each method of testing, were plotted against the per cent of cold reduction as is shown in Figure 30 (Series A). The sharpest increase in hardness is between the annealed specimens and the 20% CR specimens.

Specimens in Series B were cold rolled 71.7% and had a correspondingly high hardness as is shown in Table 4.

The hardness of specimens in Series C that were cold rolled 43.3% was higher than for specimens in Series A with the same amount of cold reduction which may be due to the difference in starting thickness of the two strips or to the difference in grain size and hardness of starting materials (Table 1).

TABLE 4

Superficial Hardness Tests of 0.051-In. Nickel Strip

Series	Plane	% CR	Hardness			
			VHN 5-Kg Load	*VHN 5-Kg Load	Knoop 0.5-Kg Load	R_{30T}
A	X	Ann	78	85	95	30*
	Y	"	89		103	30
	Z	"	84		91	30
A	X	10% CR	131	133	154	70*
	Y	"	134		143	56
	Z	"	136		155	59
A	X	20% CR	164	165	179	74*
	Y	"	172		174	66
	Z	"	175		188	71
A	X	30% CR	170	172	188	77*
	Y	"	175		207	69
	Z	"	175		203	64
A	X	40% CR	175	180	178	77*
	Y	"	179		196	71
	Z	"	178		219	60
A	X	50% CR	189	188	216	81*
	Y	"	199		235	76
	Z	"	198		226	70
B	X	71.7% CR	211	210	256	83*
	Y	"	214		254	76
	Z	"	215		229	78
C	X	43.3% CR	197	198	222	80*
	Y	"	195		236	76
	Z	"	195		212	75

*Unmounted Specimens

Superficial Hardness of 0.036-In. Gauge, Nickel Strip

Table 5 lists the results of superficial hardness tests that were carried out on the 0.036-in. strip. Vickers and Tukon hardness tests were carried out on all the mounted specimens. Vickers hardness tests were also done on the X-plane of unmounted specimens and show close agreement with tests on mounted specimens. The 0.036-in. strip was too thin to obtain reliable R_{30T} hardness values on either the mounted or unmounted specimens.

Both the Vickers and Knoop hardness values for the X-plane of the annealed specimens were considerably lower than the values of the Y- and Z-planes. The reason for this is not understood, and this difference is not as marked in the cold-rolled specimens.

In Figure 30, average hardnesses of the X-, Y-, and Z-planes in each specimen, for both methods of testing, are plotted against the per cent of cold reduction and can be compared with similar plots for the 0.051-in. strip also shown in Figure 30. The plots for the Vickers and Knoop hardnesses for both gauges of nickel strip are similar but values for the 0.036-in. strip are higher. Because the annealed 0.036-in. strip was harder (Tables 1, 4, and 5), higher values for the cold rolled specimens would also be expected.

Specimens in Series B were cold rolled 80% and, as is shown in Table 5, the hardnesses are appropriately higher than the 70% CR specimens in Series A.

The hardnesses for specimens in Series C (60% CR) were higher than for specimens with the same cold reduction in Series A and these compare with the 80% CR specimens in Series B. Previous results show that the 0.051-in. gauge specimens in Series C are harder than those in Series A with the same per cent of cold reduction. The reason for this is not clear because the starting material for Series C is harder than for Series A

TABLE 5

Superficial Hardness Tests of 0.036-In. Nickel Strip

Series	Plane	% CR	Hardness		
			VHN 5-Kg Load	*VHN 5-Kg Load	Knoop 0.5-Kg Load
A	X	Ann	82	80.2	90
	Y	"	122		156
	Z	"	118		144
A	X	10%	151	156	157
	Y	"	158		166
	Z	"	157		164
A	X	20%	190	192	208
	Y	"	187		226
	Z	"	183		220
A	X	30%	185	185	211
	Y	"	190		215
	Z	"	187		211
A	X	40%	187	190	206
	Y	"	192		218
	Z	"	192		227
A	X	50%	195	199	220
	Y	"	206		227
	Z	"	203		225
A	X	60%	206	216	232
	Y	"	216		246
	Z	"	219		249
A	X	70%	212	216	246
	Y	"	223		255
	Z	"	216		258
B	X	80%	221	226	255
	Y	"	227		271
	Z	"	223		271
C	X	60%	223	232	271
	Y	"	226		268
	Z	"	224		271

* Unmounted Specimens.

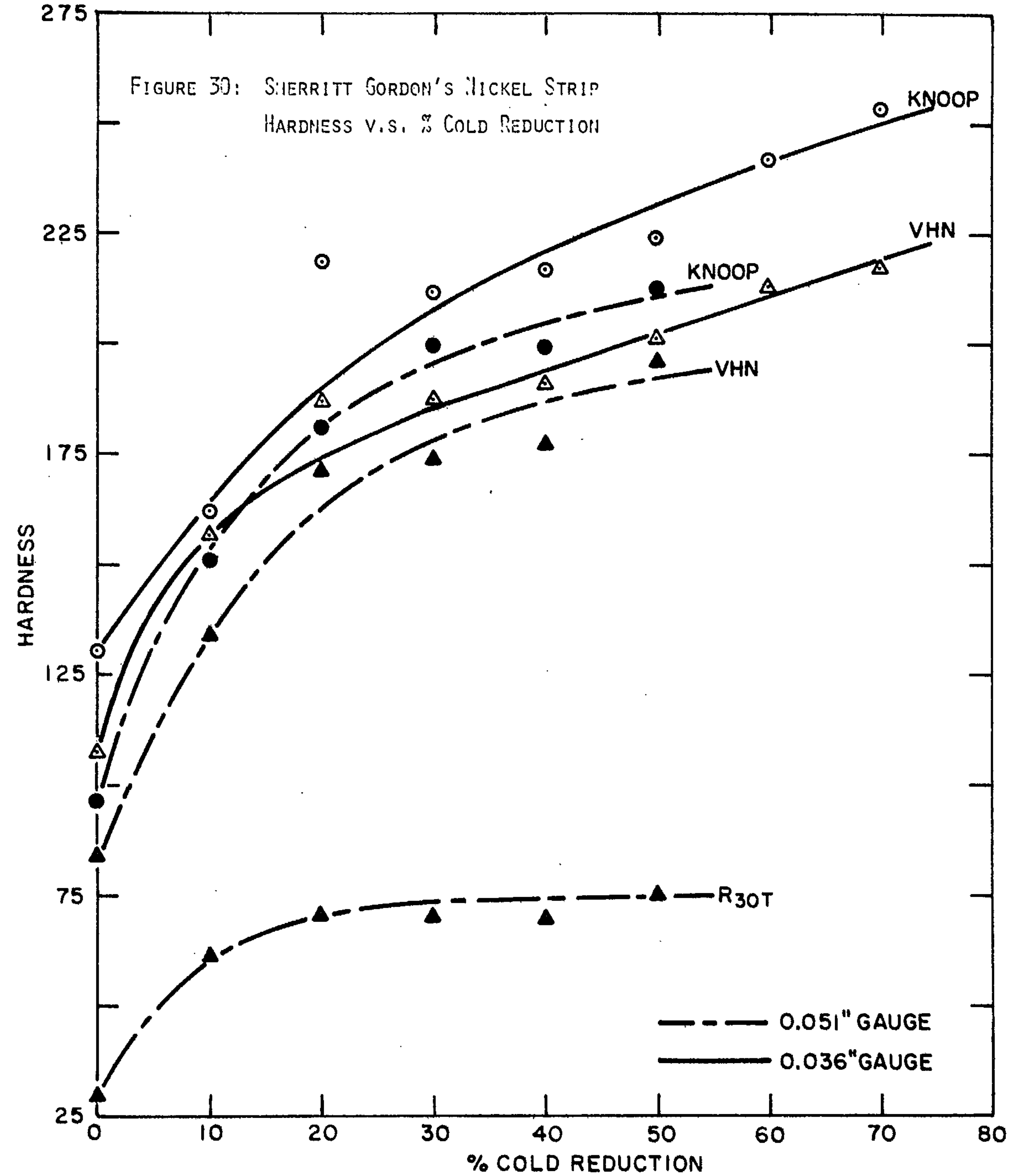


FIGURE 30: SHERRITT GORDON'S NICKEL STRIP
HARDNESS V.S. % COLD REDUCTION
KNOOP
VHN
KNOOP
VHN
R30T
0.051" GAUGE
0.036" GAUGE
HARDNESS
% COLD REDUCTION
275
225
175
125
75
25
0
10
20
30
40
50
60
70
80

in the 0.051-in. strip and softer in the 0.036-in. strip (Table 1). It is possible that the thickness of the starting material, 0.180 in. for Series A and 0.090 in. for Series C, has some influence on the hardness after cold reduction.

Magnetic Properties

Magne Gage readings obtained on the specimens of 0.051-in. and 0.036-in. strips in Series A are related to per cent cold reduction in Figure 31. The magnetic permeability of both nickel strips decreases with increased per cent of cold reduction; the greatest decrease occurs in the first 20%. Because the two strips have separate plots there must be factors other than the per cent of cold reduction that affect their magnetic property.

The thickness of the strips had no noticeable influence on the Magne Gage readings. Also, microexamination shows that the structure does not affect the magnetic property of the strips because the grains in both strips remain unaffected with up to 20% cold reduction and it is within this range that there is the greatest change in the magnetic permeability (Figure 31) which corresponds inversely to rising hardness (Figure 30).

To determine the relationship between hardness and magnetic permeability, Vickers hardness values for specimens in Series A for both nickel strips were plotted against their Magne Gage readings (Figure 32). The best straight line through the points shows that the magnetic permeability decreases as the hardness increases. There is no distinction between the strips, and though there is some scatter, it is evident that the magnetic permeability of both the 0.051-in. and 0.036-in. strips is inversely proportional to strip hardness.

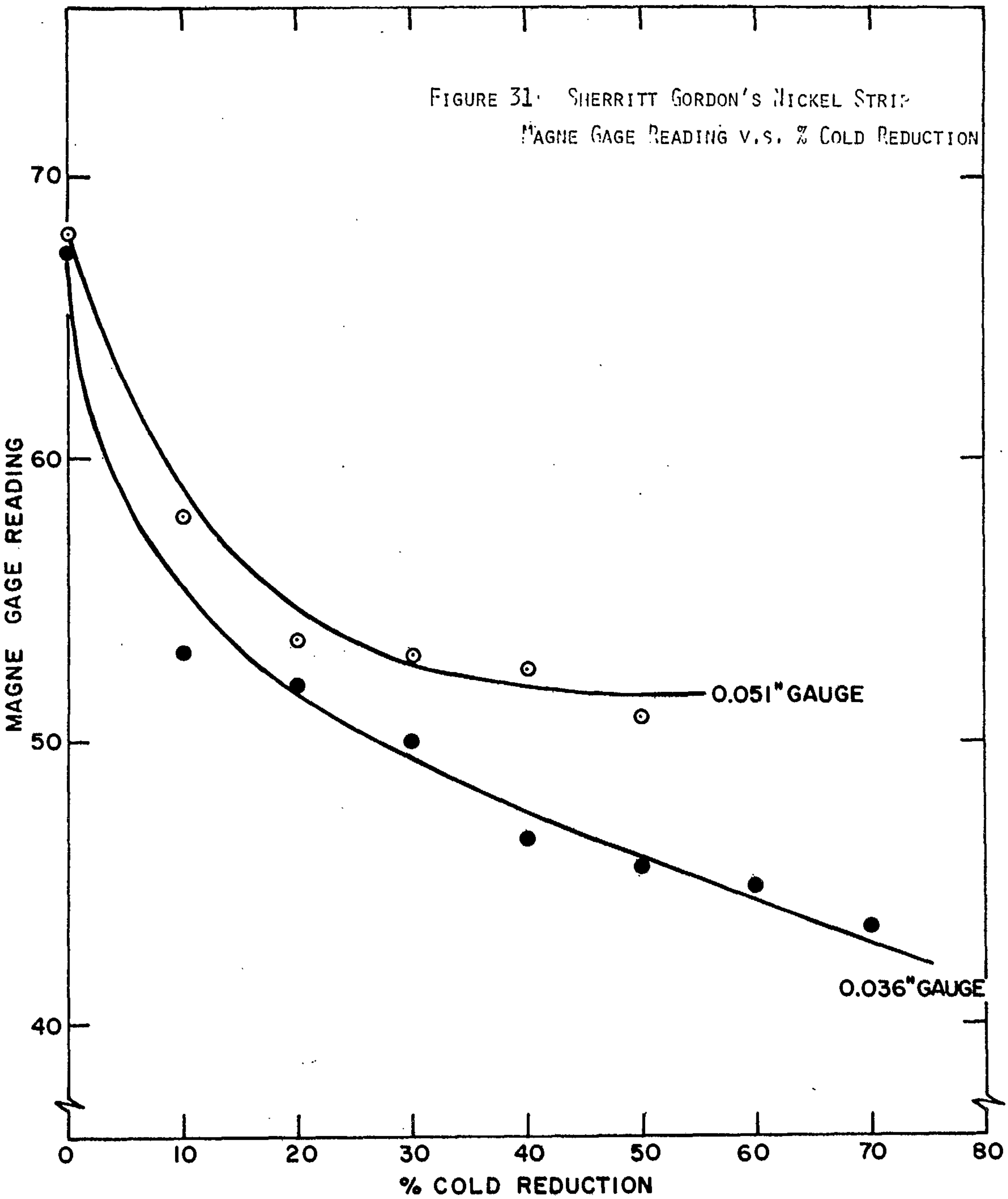


FIGURE 31· SHERRITT GORDON'S NICKEL STRIP
MAGNE GAGE READING V.S. % COLD REDUCTION
MAGNE GAGE READING
70
60
50
40
0.051" GAUGE
0.036" GAUGE
0
10
20
30
40
50
60
70
80
% COLD REDUCTION

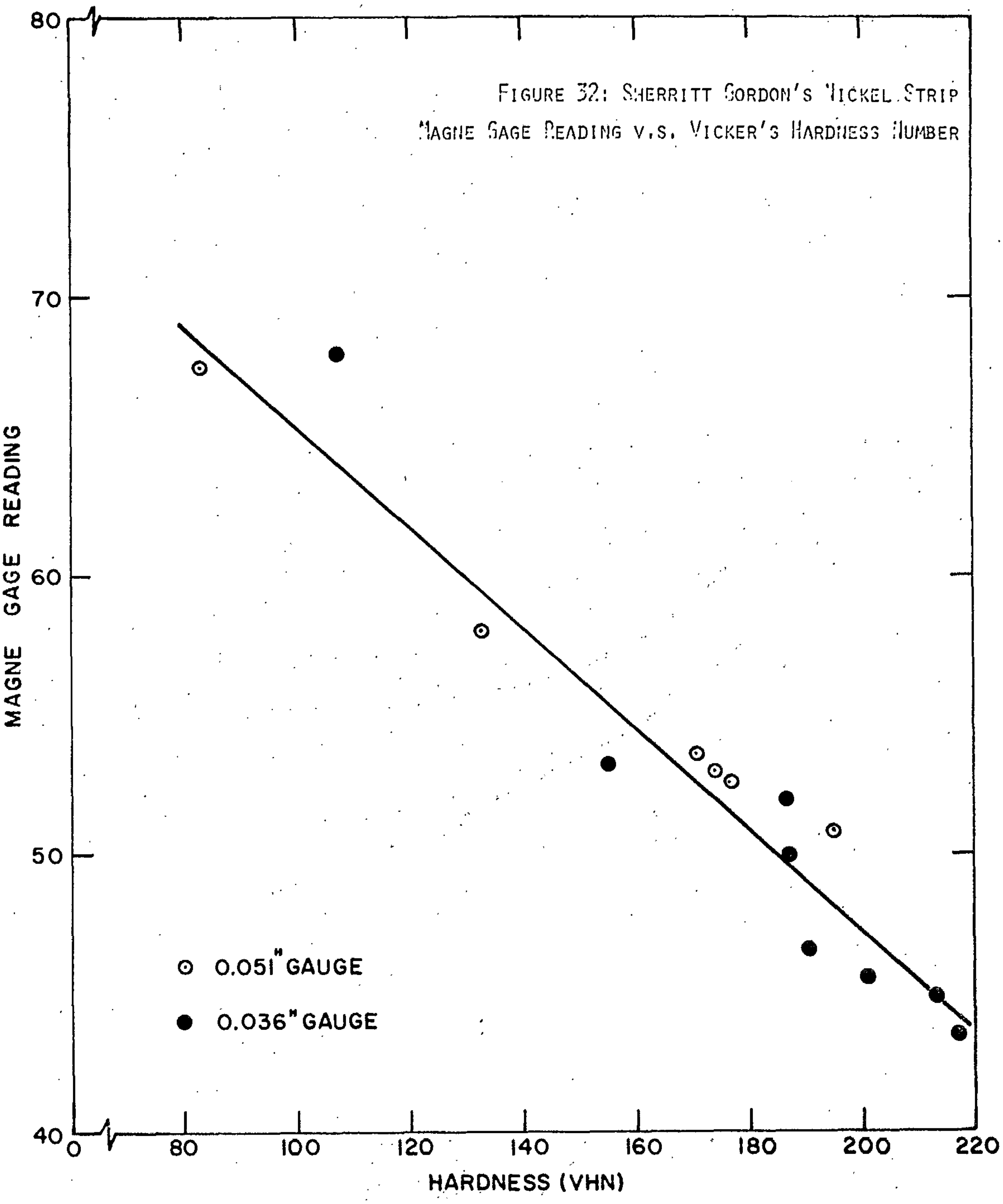


FIGURE 32: SHERRITT GORDON'S NICKEL STRIP
MAGNE GAGE READING V.S. VICKER'S HARDNESS NUMBER

CONCLUSIONS

1. There is no change in the equiaxed grain structure in 0.051-in. or 0.036-in. strip until the cold reduction exceeds 20 and 30%, respectively. Grains in specimens with additional cold work are flattened to a pancake-like shape.

2. The greatest increase in hardness occurs in the first 20% of cold reduction for both nickel strips.

3. Hardness values for the 0.036-in. strip are higher than those for the 0.051-in. strip with the same per cent of cold reduction.

4. For the same per cent of cold reduction, material cold rolled from 0.090-in. strip is harder than that cold rolled from 0.180-in. strip.

5. The magnetic permeability is higher for the 0.051-in. strip than for the 0.036-in. gauge for the same cold reduction.

6. The magnetic permeability of both nickel strips is inversely proportional to strip hardness.

ADDITIONAL WORK

Because the highest magnetic permeability and hardness change was observed to be between 0 and 20% cold reduction, additional work has been done on nickel strip within this range of cold work and will be reported elsewhere.