ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

MEASUREMENT OF COLD WORK
IN TYPE 316 STAINLESS STEEL

by

N. J. Carson, Jr.,* Clarence Steves,
C. J. Renken, K. J. Reimann,
and Harold Berger

Materials Science Division

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

December 1972

This document is
PUBLICLY RELEASABLE
Mary R. Hale, ANL
Authorizing Official
Date 8-17-09

*Now with EBR-II Project, Argonne-West

MASTER

For Announcement in Nuclear
Science Abstracts. Distribution Limited
to Participants in the LMFBR Program.
Others request from TIC. *leg*

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

f

f

.

f

f

f

f

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	7
I. INTRODUCTION.	7
II. BEHAVIOR OF THE STEEL.	8
III. FABRICATION OF SPECIMENS	10
IV. MICROHARDNESS MEASUREMENTS.	12
V. OPTICAL STRAIN MEASUREMENTS.	15
VI. MECHANICAL TWINNING	19
A. Experimental Data.	19
B. Discussion of Results.	21
C. Conclusions	24
VII. MARTENSITE FORMATION.	24
VIII. MAGNETIC MEASUREMENT OF COLD WORK	25
IX. CRITICAL-ANGLE REFLECTIVITY MEASUREMENTS OF COLD WORK	28
X. MÖSSBAUER MEASUREMENT OF COLD WORK	33
XI. SUMMARY AND CONCLUSIONS	34
ACKNOWLEDGMENTS	35
REFERENCES.	36

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1.	Phase Diagram for Stainless Steel Weld Deposits	8
2.	Solubility of Carbon in Steel Containing 18 wt % Cr and 8 wt % Ni	9
3.	Relationship between Decrease in Density and Absolute Value of True Strain for Type 316 Stainless Steel	10
4.	Dislocation Density in Type 316 Stainless Steel as a Function of Strain	10
5.	Compositions of Type 316 Stainless Steel	11
6.	Microhardness of Type 316 Stainless Steel Strained at Room Temperature, Measured with 50-g Load	12
7.	Microhardness of Type 316 Stainless Steel Strained at Room Temperature, Measured with 500-g Load	12
8.	Microhardness of Type 316 Stainless Steel Tubes Manu- factured by CarTech	14
9.	Microhardness as a Function of Cold Work for Flat-plate Specimens Described in Fig. 5	16
10.	Variation of Dislocation Density with Microhardness for Type 316 Stainless Steel	16
11.	Grain Boundaries Intersected per Unit Length of Line Drawn on Cold-worked Rods	17
12.	Model for Optical Strain Measurement	17
13.	Optical Measurements of Strain in CarTech Round-robin Tubing	19
14.	Comparison of Strain Values Measured by Microhardness with Values Measured from Grain Shape	19
15.	Microstructure of Type 316 Stainless Steel Specimen Submitted to a True Strain of 29% at Room Temperature	19
16.	Results of Line Counts Made on Type 316 Stainless Steel Rods Elongated at Room Temperature	20
17.	Microstructure of Type 316 Stainless Steel Specimen Submitted to a True Strain of -29% at Room Temperature . . .	20
18.	Twinning Model	21
19.	Elements of Twinning System in fcc Crystals	22

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
20.	Resolution of Forces in Crystal	22
21.	Percent Reduction and Field Normal to the Rod as a Function of Distance along the Rod	26
22.	Steps in Magnetic Retentivity Measurement of Cold Work in Stainless Steel Tubing	26
23.	Retentivity Measurements on Cold-worked Tubing from Different Manufacturers	27
24.	Retentivity vs Cold Work for Flat-rolled Specimens of Fig. 5	28
25.	Test Arrangement for Rayleigh Critical-angle Reflection Measurements.	29
26.	Reflection Amplitude as a Function of Critical Angle	30
27.	Rayleigh Critical Angle as a Function of Cold-work Percentages	31
28.	Critical Angle as a Function of Microhardness	31
29.	Critical Angle as a Function of Rolling Direction of Cold-worked Type 316 Stainless Steel	31
30.	Rayleigh Critical Angle as a Function of Cold Work at Two Frequencies	32

LIST OF TABLES

<u>No.</u>	<u>Title</u>	<u>Page</u>
I.	Analysis of CarTech HT V87210.	8
II.	Microhardness of One Cold-worked Tube with Calculated Error.	13
III.	Average Centerline Microhardness of Round-robin Tubes. . .	14
IV.	Calculations for Optical Strain Measurement	18
V.	Schmid Factors for Twinning Systems in fcc Crystals Pulled along [111] Direction	23
VI.	Magnetic Field Retained by Four Specimens of Strained Type 316 Stainless Steel.	24
VII.	Microhardness and Retentivity Measurements of Round-robin Tubes	27

MEASUREMENT OF COLD WORK IN TYPE 316 STAINLESS STEEL

by

N. J. Carson, Jr., Clarence Steves,
C. J. Renken, K. J. Reimann,
and Harold Berger

ABSTRACT

Changes in the structure of Type 316 stainless steel as it is cold-worked are discussed. Various destructive and nondestructive methods of measuring cold work are presented. The destructive methods applied were microhardness and optical strain. The nondestructive methods were based on magnetic retentivity, ultrasonic critical-angle, and Mössbauer techniques. The advantages and limitations of each method are presented, together with comparable measurements on several sets of test specimens.

I. INTRODUCTION

Type 316 stainless steel possesses qualities that make it a potential candidate for a fast-breeder cladding material. Its in-reactor life appears to be greatest when it is used in a cold-worked condition rather than in a solution-annealed condition. Thus cold work is a property of the material that must be specified before purchase and verified on receipt.

This program was directed toward (1) the development of a method with the capability of nondestructive measurement of cold work in Type 316 stainless steel tubing; (2) the development of a secondary test, either destructive or nondestructive, which can be used in calibrating the standards required in most nondestructive tests; and (3) the accumulation of additional data pertaining to changes that occur in the structure of the stainless steel as it is cold-worked to more clearly define the meaning of the term cold work. It was realized at the outset of the program that the last two objectives were, to some extent, preconditions for the attainment of the first objective. For that reason, the research on the three objectives will be discussed in reverse order, culminating in a discussion of the development of nondestructive-test methods. Due to termination of the program, this final phase was not fully completed.

II. BEHAVIOR OF THE STEEL

Most of the Type 316 stainless steel used was from heat (HT) V87210 manufactured by the Carpenter Technology Corporation (CarTech).^{*} However, late in the study, Type 316 stainless steel specimens that incorporated a range of chemical compositions allowable under the then-existent Fast Flux Test Facility (FFTF) specifications were produced at ANL to check the possible effect of varying compositions on the nondestructive test methods. The analysis of HT V87210 is listed in Table I. The chromium and nickel "equivalents" for this heat are 19.5 and 16.1, respectively. When cooled rapidly from a temperature at which the carbon is in solution, this steel is a supersaturated solution of carbon in austenite (Fig. 1). It should be free of ferrite when homogeneous; however, failure to achieve homogeneity in the casting can result in areas of ferrite that are usually high in chromium.

TABLE I. Analysis of CarTech HT V87210

Element	Percent by Weight	Element	Percent by Weight
Carbon	0.053	Nickel	13.55
Manganese	1.88	Molybdenum	2.43
Silicon	0.47	Copper	0.08
Phosphorus	0.013	Boron	<0.001
Sulfur	0.008	Nitrogen	0.007
Chromium	16.37	Cobalt	0.07

Fig. 1

Phase Diagram for Stainless Steel
Weld Deposits (from Schaeffler¹).
Neg. No. MSD-56953.

When the Type 316 stainless steel was heated at 400-800°C, precipitates were produced. The kinetics of precipitation has been studied by Weiss and Stickler.² The precipitation scheme they suggest is as follows:

^{*}Santa Barbara, California.

(1) The carbides precipitate quickly because of the high diffusion rate of the carbon. This results in a carbon-depleted matrix from which chi phase $[(\text{Cr}, \text{Fe}, \text{Mo})_{18}\text{C}]$ and eta phase (Fe_2Mo) can precipitate. (2) The precipitation of chi and eta phases depletes the matrix in chromium and molybdenum and increases its solubility for carbon. (3) Some of the original carbides dissolve and leave metal concentrations from which sigma phase $[(\text{Cr}, \text{Mo})(\text{Fe}, \text{Ni})]$ can form. In their experiments, the carbide particles appeared quickly, whereas the other phases required more than 100 hr at 816°C .

By far the largest fraction of precipitate is carbide. The size of the carbide precipitates and their distribution are determined by the condition of the metal and the mobility of the carbon atoms. Boundaries and defects are preferred nucleation sites at all temperatures. Solution-annealed material heat-treated at high temperatures will have coarse precipitate particles located largely in the grain boundaries, whereas the same material heat-treated at lower temperatures will have smaller particles, many distributed within the grains. Cold-worked material will have finer particles distributed more uniformly throughout the grains after all sensitization heat treatments. To dissolve the carbide precipitates, the steel must be heated to 900°C or higher (Fig. 2).

Fig. 2

Solubility of Carbon in Steel Containing 18 wt % Cr and 8 wt % Ni (from Colombier and Hochmann³). Neg. No. MSD-56558.

The effect of cold work on the substructure of stainless steel is clouded by the formation of martensite. There is general agreement on several points: (1) The carbides formed when cold-worked stainless steel is heat-treated are nucleated on the defects produced in cold working, and (2) the temperature at which martensite starts to form (martensite-start temperature) is raised by cold work. Much of the research on stainless steel can be summarized in the words of Embury *et al.*,⁴ who found that "when mechanical working results in the production of both stable substructural barriers and deformation-induced transformation ... no simple correlation exists between the substructure and the flow stress." They performed work-hardening experiments on copper, iron, and some iron alloys, including Type 304 stainless steel, and

found a linear relationship between flow stress and the square root of the dislocation subcell diameter for all these materials except stainless steel.

Garofalo and Wriedt⁵ measured the density of Type 316 stainless steel as a function of prestrain in both tension and compression. They found two linear relationships between density decrease and true strain: one for tensile and one for compressive loading (Fig. 3).

Fig. 3

Relationship between Decrease in Density and Absolute Value of True Strain for Type 316 Stainless Steel. Neg. No. MSD-56954.

In 1965, Mazza⁶ identified deformation twins in Type 316 stainless steel that had been cold-worked more than 12%. These correlate well with the "deformation bands" of Garofalo *et al.*⁷ and the "stacking-fault clusters" of Weiss and Stickler.² Twins were formed when solution-annealed steel was elongated at room temperature. Twins were not observed when solution-annealed material was cold-worked at 400°C or when the cold work was preceded by a precipitation heat treatment. All investigators report these twins as excellent nucleation sites for precipitates.

Recently, Stickler and Booker⁸ measured dislocation densities in Type 316 stainless steel as a function of tensile strain. Their results, which cover the range of 0-20% elongation, appear in Fig. 4.

Fig. 4

Dislocation Density in Type 316 Stainless Steel as a Function of Strain (calculated from data of Stickler and Booker⁸). Neg. No. MSD-56560.

III. FABRICATION OF SPECIMENS

Cold work is generally defined in terms of the percent reduction in cross-sectional area. This terminology implies a state function. However, the amount and distribution of cold work over the cross section is largely dependent on the process and the operating conditions. Light reductions by rolling or drawing generally cold-work the outside surfaces more than the

center portions. Grinding produces highly strained surface layers, but has little effect on the interior. A given reduction in area accomplished in a series of light reductions will result in a cold-work distribution quite different from the same reduction accomplished in a single pass. Percent reduction in area is, like most cold-work functions, primarily a path function and has little meaning when the path is not specified.

The process by which tubing is made involves a high degree of redundant cold work and was judged too complex to provide material for a reference or ground state. Instead, the first specimens were cold-worked by simple tensile elongation. (Tensile rods were elongated, their end sections were removed, the working lengths were centerless ground, and their heavily cold-worked surfaces were removed by etching.) The degree of cold work was calculated from diameter measurements taken at marked locations along the tensile rods before both elongation and grinding. The mechanically worked layer produced by grinding extended to a depth of ~ 0.0254 mm (0.001 in.) from the surface. Approximately 0.076 mm (0.003 in.) was removed in etching.

Additional cold-worked specimens were prepared by compression. Right cylinders, 0.95 cm ($3/8$ in.) in diameter and 1.9 cm ($3/4$ in.) long, were pressed between carbide anvils mounted in a set of die shoes. Greased lead foils were used to lubricate the work-anvil interfaces and were replaced after each 10% reduction in height. The compressive load was applied in a conventional tensile testing machine.

The third series of specimens were tubes made available as part of a cold-work-measurement round robin that included three laboratories engaged in AEC-funded research on cold work in Type 316 stainless steel.

A fourth series of specimens was fabricated to test the effect of compositional changes on the nondestructive test methods. Final reduction on these specimens was accomplished by rolling and covered a range of 0-50%. Before the final reduction, the dimensions of each specimen were calculated so that a flat plate of ~ 1.6 mm was formed after rolling. The plates were trimmed to a finished size of ~ 50 by 25 mm, and the edges were etched. All specimens were cold-rolled $\sim 50\%$ before the final anneal. The composition of the specimens is shown in Fig. 5.

Melt No. Ext. No.	AM143 803	AM145 803	AM153 807	AM147 806	AM155 808	AM149 806	AM157 808	AM151 807
Cr	18	18	18	18	16	16	16	16
Ni	14	14	12	12	14	14	12	12
Mo	3	2	3	2	3	2	3	2
Fe	64	65	66	67	66	67	68	69

Fig. 5

Compositions of Type 316 Stainless Steel. 1 wt % Mn in all Melts. Neg. No. MSD-55760.

IV. MICROHARDNESS MEASUREMENTS

One of the first destructive techniques explored for the calibration of nondestructive test standards was hardness measurements. All mechanical work, both useful and redundant, contributes to work hardening. The measurements are easily obtainable and require only conventional equipment. The major shortcoming of hardness measurements is their response to factors other than cold work. They are not used routinely with most stainless steels because they are affected by the presence of martensite formed during cold working. When X-ray diffraction showed no trace of martensite in the cold-worked Type 316 samples, hardness measurements were pursued as an indicator of cold work.

The curves of microhardness versus true strain for HT V87210 are shown in Figs. 6 and 7. These values were obtained with a Leitz Miniload hardness tester. The surfaces tested were all perpendicular to the applied tensile or compressive load. The readings in Fig. 6 were obtained with a 50-g load, and those in Fig. 7 with a 500-g load. The 50-g load produced an indentation far smaller than a single grain, whereas the 500-g load

Fig. 6. Microhardness of Type 316 Stainless Steel Strained at Room Temperature, Measured with 50-g Load. Neg. No. MSD-53279.

Fig. 7. Microhardness of Type 316 Stainless Steel Strained at Room Temperature, Measured with 500-g Load. Neg. No. MSD-53278.

produced an indentation slightly larger than a single grain. The magnitude of the readings varied with the applied load (for all loads between 50 g and 5 kg); however, the range of readings did not change significantly with load. The readings taken with the 50-g load were adopted as standard inasmuch as the size of the indentation produced was acceptable for use on thin-wall tubing.

For the curve shown in Fig. 6, the standard error of estimate, which is the standard deviation of the data points about the regression line, is just under 1.6% true strain. To this must be added the error* incurred when test measurements are made. However, the error of the sample average depends on the number of readings averaged. For the 25 readings shown in Table II, this error is ~0.6% true strain. This sample average error combines with the 1.6% standard error of estimate to yield a total error of ~1.7%. Twice this, or 3.4%, would be a confidence level of 95%. Thus, microhardness appears to have the capability of indicating true strain in these samples of $\pm 3.4\%$ with a confidence of 95%.

TABLE II. Microhardness of One Cold-worked Tube^a with Calculated Error^b

Measurement Number, n	DPH (50 g), X	Computations		Measurement Number, n	DPH (50 g), X	Computations	
		X - 277	(X - 277) ²			X - 277	(X - 277) ²
1	277	0	0	14	265	-12	144
2	268	-9	81	15	265	-12	144
3	271	-6	36	16	296	19	361
4	280	3	9	17	277	0	0
5	280	3	9	18	293	16	256
6	274	-3	9	19	283	6	36
7	280	3	9	20	268	-9	81
8	283	6	36	21	265	-12	144
9	271	-6	36	22	268	-9	81
10	265	-12	144	23	268	-9	81
11	293	16	256	24	271	-6	36
12	280	3	9	25	286	9	81
13	296	19	361				

^a Tube 213-20, CarTech HT V87210

^b The sum of the hardness values $\Sigma X = 6923$. The average hardness value $\bar{X} = 277$, which is $\approx 27\%$ true strain. The deviation of the hardness from the average value is given by $(X - 277)$. The sum of the squares of the deviational values is $\Sigma(X - 277)^2 = 2440$. The square of the standard deviation of the previous value is $\sigma_X^2 = \Sigma[(X - 277)^2]/(n - 1) = 2440/24 = 102$. The standard deviation of the individual measurements is $\sigma_X = 10.1$ DPH. The standard deviation of the mean of the measurements is $\sigma_{\bar{X}} = \sigma_X/\sqrt{n} = 10.1/5 = 2.02$ DPH $\approx 0.58\%$ true strain

The results of microhardness measurements made on FFTF-size tubes appear in Fig. 8. All the tubes are fabricated from Type 316 stainless steel (HT V87210) as was the bar stock from which the tensile and compression specimens were fabricated. Each centerline reading is an average of 25 measurements, whereas each edge measurement is an average of 10 measurements. The data show that, for tubes from the same supplier, microhardness is an excellent indicator of the degree of cold work.

*Error is defined as the standard deviation of a measured average microhardness about the true average microhardness and is expressed in units of true strain.

Fig. 8

Microhardness of Type 316 Stainless Steel Tubes Manufactured by CarTech, ANL Neg. No. 306-1174 Rev. 1.

Microhardness measurements on tubes from different suppliers, but with comparable degrees of cold work, are shown in Table III. All figures are averages of 25 measurements made along the centerline of the tube wall.

TABLE III. Average Centerline Microhardness of Round-robin Tubes (DPH, 50 g)

Condition	Supplier				
	CarTech	Fine	Sandveik	Superior	Wolverine
Solution-annealed	$\bar{X} = 149$	$\bar{X} = 147$	$\bar{X} = 149$	$\bar{X} = 145$	$\bar{X} = 147$
	$\sigma_{\bar{X}} = 0.500$	$\sigma_{\bar{X}} = 0.788$	$\sigma_{\bar{X}} = 0.615$	$\sigma_{\bar{X}} = 0.432$	$\sigma_{\bar{X}} = 0.601$
20% cold-worked	$\bar{X} = 310$	$\bar{X} = 303$	$\bar{X} = 300$	$\bar{X} = 297$	$\bar{X} = 302$
	$\sigma_{\bar{X}} = 1.01$	$\sigma_{\bar{X}} = 7.94$	$\sigma_{\bar{X}} = 1.70$	$\sigma_{\bar{X}} = 2.52$	$\sigma_{\bar{X}} = 1.74$

Inasmuch as various fabrication methods and different heats of steel were used to make these tubes, the difference in microhardness for a given nominal amount of cold work may be due to either the processing or the composition of the steel. Microhardness as an indicator of cold work is dependent to some extent on the chemical composition of the particular heat of steel under test. An idea of the extent of this dependence can be gained by reference to Fig. 9, which shows hardness versus cold work for the flat-plate specimens of Fig. 5. A study on the effect of variations of the proportions of the various constituents on microhardness for different values of cold work was underway when the program was terminated.

The good agreement of the microhardness curves from HT V87210 is undoubtedly due to the low amount of martensite generated on cold working. The fact that the curves hold for elongated as well as compressed

specimens indicates a low sensitivity to the type of cold-working process used. However, the hardness of the cold-worked specimens is a function of their condition before cold working, and it is imperative that the final anneal be closely controlled to make the dissolution of precipitates as complete as possible. Changes in precipitate distribution can change the work-hardening rate and, consequently, the hardness of the cold-worked piece. This dependence on starting state is not considered a serious limitation because it applies to many other physical properties.

When the microhardness data from Fig. 6 are combined with the dislocation densities of Fig. 4, the plot in Fig. 10 is obtained. It appears from this plot that microhardness is a good indicator of dislocation density to 20% elongation.

A second vertical scale added on the right of Fig. 8 shows the degree of true strain necessary to produce the hardness values plotted on the left-hand scale. These values were obtained from Fig. 6. Note the large difference. A reduction of almost 40% by tensile elongation is required to produce the same hardness that was produced by a drawing reduction of 20%. Since the cold work in tensile elongation is 100% useful cold work by definition, it would appear that the redundant cold work in tube drawing is almost equal to the useful cold work. Therein lies the fallacy of using reduction in area to describe the degree of cold work.

V. OPTICAL STRAIN MEASUREMENTS

Plastic strain, in some situations, can be measured optically. The basic assumption involved is that the deformation of the individual grains in the workpiece is similar to that of the workpiece. If the workpiece is elongated, the individual grains are elongated proportionately. Thus, the degree of deformation can be calculated from the grain dimensions measured on either a plane of polish or a photograph both before and after the deformation.

Optical methods were first applied to the tensile and compression specimens without success. The number of grain boundaries intercepted per unit length of two lines was counted, one drawn parallel to and one drawn perpendicular to the direction of elongation or compression. The grain boundaries in the compression specimens tended to "wash out" at the higher degrees of cold work. Although this was not true of the tensile specimens, the counts obtained did not correlate well with the degree of deformation. Later this same technique was applied to rods reduced by rolling at room temperature. The counts made are shown in Fig. 11. The correlation with percent reduction in area is obvious.

Fig. 9

Microhardness as a Function of Cold Work for Flat-plate Specimens Described in Fig. 5. The vertical spread in values represents variations due to the chemistry changes outlined in Fig. 5. Neg. No. MSD-57526.

Fig. 10
Variation of Dislocation Density with Microhardness for Type 316 Stainless Steel. Neg. No. MSD-56559.

Fig. 11. Grain Boundaries Intersected per Unit Length of Line Drawn on Cold-worked Rods. Neg. No. MSD-56557.

Success with the rolled rods prompted application of the more sophisticated method of Flinn and Philofsky⁹ to samples of tubing manufactured for use in the FFTF. The model on which the technique is based is shown in Fig. 12, and the calculations required appear in Table IV.

The model assumes that the grains in fully annealed steel are approximately spherical and become ellipsoidal after cold working. The grains are elongated in the c direction and are compressed equally in the a and b directions. The data required are the number of grain boundaries intersected per unit length of two lines, one drawn parallel to and one perpendicular to the c axis (or direction of elongation).

The model requires the measurement of grain shape before and after cold working. The true

strain in a given axial direction is then the natural logarithm of the length of the axis after cold working divided by the length of the same axis before cold working. The model can be used to measure the difference in strain between two cold-worked states. If the dimensions before cold working are unavailable, constancy of volume may be assumed and an equivalent radius calculated and used as the starting axial dimension. This may or may not be correct. The calculations do hold for the situation in which both the annealed and cold-worked materials are referenced to an equivalent radius. In this case, the observed strain of the annealed sample is simply subtracted from that of the cold-worked sample.

Optical strain measurements were made on pieces of cold-worked tubing obtained as part of the round-robin measurement of cold work. There were four tubes with, respectively, 5, 10, 20, and 30% reduction in cross-sectional area. These were the same tubes on which microhardness

Fig. 12

Model for Optical Strain Measurement.
Neg. No. MSD-56561.

TABLE IV. Calculations for Optical Strain Measurement

Basic Data	
$N_L(1) \equiv$ Number of grain boundaries intercepted by unit length of line drawn in c direction.	
$N_L(2) \equiv$ Number of grain boundaries intercepted by unit length of line drawn in a or b direction.	
$F_0 = \frac{N_L(1) + N_L(2)}{2}$	(1)
$F_2 = \frac{3}{4}[N_L(1) - N_L(2)]$	(2)
$\Psi = \frac{F_2}{F_0}$	(3)
$\frac{a}{c} = 0.994 - 1.15\Psi + 0.250\Psi^2$	(4)
$e = 1 - \left(\frac{a}{c}\right)^2 \approx k'^2 G_0$ from Riegels, ¹¹	(5)
and	
$c = 1.621 \left(\frac{k'^2 G_0}{F_0} \right)$	(6)
The term a is found by substituting in Eq. 4 as follows:	
$e_c = \ln \left(\frac{C}{C_0} \right) \approx \ln \left(\frac{c}{r_0} \right)$	(7)
with	
$r_0 = (a^2 c)^{1/3}$	(8)

measurements had been made. All were FFTF-type tubes made by CarTech; however, the 30% cold-worked tube was from a separate heat. An annealed tube was also available, but was obviously not indicative of the structure before cold working and thus was not used.

The results of strain measurements made on these tubes appear in Fig. 13. Every point is based on two line counts of 50-100 intercepts each, which is not a large number. The error can undoubtedly be reduced by higher counts, but the trend is obvious. The fact that the curve does not go through the origin is probably due to some asymmetry in the annealed grains, which could have been compensated for had the actual starting material been available. However, it does appear that the shape of grains in drawn tubes can be correlated with the degree of strain produced in drawing, with proper selection of specimens.

Figure 14 compares the strain values obtained through microhardness with those obtained optically. The slope of the curve is 0.92. If the

slope were exactly 1.0, both measurements would have the same sensitivity to strain. Although this is not entirely true, a slope of 0.92 does show that a high degree of correlation between the two measurements can be attained.

Fig. 13. Optical Measurements of Strain in CarTech Round-robin Tubing. Neg. No. MSD-56563.

Fig. 14. Comparison of Strain Values Measured by Microhardness with Values Measured from Grain Shape. Neg. No. MSD-56562.

VI. MECHANICAL TWINNING

A. Experimental Data

The microstructures of both the tensile specimens and the compression specimens were closely examined for features that might be correlated with degree of strain. The outstanding feature of the tensile specimens, as shown in Fig. 15, was the presence of deformation twins. The density of deformation twins was measured and is plotted against the degree of true strain in Fig. 16. Although the error was fairly large, excellent correlation exists between the twin density and the degree of true strain for strains up to 30%.

Fig. 15. Microstructure of Type 316 Stainless Steel Specimen Submitted to a True Strain of 29% at Room Temperature. Mag. 325X. Neg. No. MSD-56581.

The compression specimens showed microstructures comparable to that in Fig. 17. This structure was produced by a strain of the same magnitude as the structure in Fig. 15. Only the sign of the strain is different.

It was obvious that the degree of twinning produced by tensile deformation was much higher than that produced by compressive deformation.

Fig. 16

Results of Line Counts Made on Type 316 Stainless Steel Rods Elongated at Room Temperature. Neg. No. MSD-53280.

Fig. 17

Microstructure of Type 316 Stainless Steel Specimen Submitted to a True Strain of $\sim 29\%$ at Room Temperature. Mag. 325X. Neg. No. MSD-56580.

The X-ray examination of the Type 316 stainless steel rods, performed initially to detect martensite, showed that the starting material had a $\langle 111 \rangle$ fiber texture. This means that the individual grains tend to have a $\langle 111 \rangle$ direction parallel to the axis of the bar. This was confirmed by metallographic examination of the cross section of bars deformed in tension. The twins in a grain frequently intersected one another at $\sim 60^\circ$, thereby constructing a pattern of equilateral triangles. This threefold symmetry is characteristic when viewing along a $\langle 111 \rangle$ direction. This texture is customary in a face-centered-cubic (fcc) alloy that has been rod-rolled.

B. Discussion of Results

The marked difference between the effects of tension and compression on the deformation of polycrystalline metals has been reported previously in niobium¹² and iron.¹³ In these cases, both the flow stress and the extent of twinning differed from tension to compression, with deformation in compression accompanied by more twinning at a lower flow stress than that in tension. It was shown that this behavior can be ascribed to the presence of a preferred grain orientation in the material. As we shall see, mechanical twinning is more sensitive to the crystal orientation than is slip.

With the help of the ball model of Fig. 18, we can visualize the geometry of twinning. The lattice is composed of a stack of $\{111\}$ planes

Fig. 18. Twinning Model. Neg. No. MSD-56588.

be created. However, Jaswon and Dove¹⁵ have stated the principle that when alternative geometric choices of creating a twin are available, nature prefers the method of smallest shear. Since no convincing exceptions to this principle have been observed, we may regard this as a law. Therefore, one direction of shear will be preferred to its reverse, and there will be an "allowed" and a "forbidden" shear for twinning.

Twinning shear in fcc crystals occurs along $\langle 112 \rangle$ directions with the sense of the allowed shear determined from the following argument. In Fig. 19, a hemisphere is made to undergo a simple shear and becomes

with atom centers located at sites A, B, and C on successive planes. Twinning occurs in the fcc system by simple movement of $\{111\}$ planes along $\langle 112 \rangle$ directions;¹⁴ i.e., the plane above A can be displaced to move its atom centers from position B to position C or vice versa. However, only one of these movements is possible. As can be seen in Fig. 18, if the planes are stacked in an A, B, C sequence and if a shear is imposed so that $A \rightarrow B \rightarrow C \rightarrow A$, a twin will be generated. If the shear is applied in the reverse direction so that $A \rightarrow C \rightarrow B \rightarrow A$, a twin will not be created; in fact, the fcc arrangement is lost. Since this offends thermodynamics, it is unlikely. If the shear strain is doubled in the reverse direction, a twin will

Fig. 19

Elements of Twinning System in
fcc Crystals. Neg. No. MSD-56587.

an ellipsoid. The plane K_1 is common to both the hemisphere and ellipsoid and is totally unaffected by the shear. This is the first undistorted plane, or twinning plane. A second undistorted plane, K_2' , is common to both hemisphere and ellipsoid. Before the shear occurred, this plane was K_2 of the hemisphere. The sense of the shear is indicated by the direction n_1 . The material above K_1 is moved along n_1 relative to the material below K_1 . The terms K_1 , K_2 , and n_1 are called the twinning elements of the twinning system. The upward normal to the K_1 plane, N_{K_1} , and n_1 must be chosen so that the upward normal to the K_2 plane, N_{K_2} , lies between them on a stereographic projection. The correct choice indicates the "allowed" sense of the twinning shear. In the fcc system, K_1 and K_2 are of the type (111) and $(\bar{1}\bar{1}1)$, respectively, and n_1 is of the type $[\bar{2}11]$.

With the correct senses for the allowed twinning shears known, the Schmid Factors for the various twinning systems may be calculated. In Fig. 20, if a normal tensile stress σ is applied along the axis of the single crystal bar, a shear stress τ is set up on the inclined plane where

$$\tau = \sigma \cos \phi \cos \lambda.$$

The shear stress is trying to displace the material above the inclined plane in the direction B relative to the remainder of the crystal. By resolving forces, it is obvious that, if the applied stress σ is reversed (a compressive stress), the direction in which τ acts is also reversed.

Suppose now that the inclined plane is a twinning plane. If, in tension, τ acts in the sense of the "allowed" twinning shear and if the orientation factor $\cos \phi \cos \lambda$ (also called the Schmid Factor) is large (approaching 0.5), then a large shear stress is set up in the correct sense, and twinning is a possibility. At the same time, in compression, τ must act in the "forbidden" sense, and twinning on this plane is not possible. Twinning would also be unlikely if $\cos \phi \cos \lambda$ were small, because the shear stress that motivates the process would be small.

Suppose a crystal is stressed along the direction $[pqr]$, either in tension or compression. The shear stress set up on any twinning system is $\sigma \cos \phi \cos \lambda$. If N_{K_1} is $[HKL]$, and n_1 is $[hkl]$, then

an ellipsoid. The plane K_1 is common to both the hemisphere and ellipsoid and is totally unaffected by the shear. This is the first undistorted plane, or twinning plane. A second undistorted plane, K_2' , is common to both hemisphere and ellipsoid. Before the shear occurred, this plane was K_2 of the hemisphere. The sense of the shear is indicated by the direction n_1 . The material above K_1 is moved along n_1 relative to the material below K_1 .

Fig. 20

Resolution of Forces in Crystal.
Neg. No. MSD-56589.

$$\cos \varphi = \frac{pH + qK + rL}{\sqrt{p^2 + q^2 + r^2} \sqrt{H^2 + K^2 + L^2}},$$

and

$$\cos \lambda = \frac{ph + qk + rl}{\sqrt{p^2 + q^2 + r^2} \sqrt{h^2 + k^2 + l^2}}.$$

Taking $[pqr]$ to be $[111]$, the direction of stressing in the textured steel, the values of $\cos \varphi \cos \lambda$ given in Table V are calculated for each twinning system.

TABLE V. Schmid Factors for Twinning Systems in fcc Crystals
Pulled along $[111]$ Direction

System Number	Normal to Twinning Plane, N_{k_1}	Allowed Twinning Direction, M_1	Schmid Factor, $\cos \varphi \cos \lambda$
1	$[111]$	$[\bar{2}11]$	0
2	$[111]$	$[11\bar{2}]$	0
3	$[111]$	$[1\bar{2}1]$	0
4	$[\bar{1}11]$	$[211]$	+0.314
5	$[\bar{1}11]$	$[\bar{1}1\bar{2}]$	-0.157
6	$[\bar{1}11]$	$[\bar{1}\bar{2}1]$	-0.157
7	$[\bar{1}\bar{1}1]$	$[1\bar{1}\bar{2}]$	-0.157
8	$[\bar{1}\bar{1}1]$	$[\bar{2}\bar{1}1]$	-0.157
9	$[\bar{1}\bar{1}1]$	$[121]$	+0.314
10	$[\bar{1}1\bar{1}]$	$[1\bar{2}\bar{1}]$	-0.157
11	$[\bar{1}1\bar{1}]$	$[\bar{2}1\bar{1}]$	-0.157
12	$[\bar{1}1\bar{1}]$	$[112]$	+0.314

The shear stress τ is $\sigma \cos \varphi \cos \lambda$ and should be positive to be of the correct sign to cause twinning. Calling σ positive when the loading is tensile and negative when it is compressive, we see that, in tension, only twinning systems 4, 9, and 12 are highly stressed for twinning, with $\tau = +0.314\sigma$. In compression, these systems are stressed incorrectly for twinning. Instead, the systems 5-8, 10, and 11 are stressed correctly, but with only a small shear stress of $\tau = +0.157\sigma$.

On the other hand, since there is no "allowed" or "forbidden" sense of the shear in slip, the shear stress set up on the $\{111\} \langle 110 \rangle$ slip systems will be exactly the same in tension and compression and will have a value of 0.272σ on the most favorably oriented slip systems. Therefore, in tension, twinning systems 4, 9, and 12 are more highly stressed than the slip systems and, accordingly, twinning occurs. In compression, however, the correctly stressed twinning systems are stressed less than the slip systems, and so slip alone occurs.

C. Conclusions

Type 316 stainless steel develops a texture during cold working that is not removed by ordinary annealing. This texture and the process by which the steel is cold-worked interact to influence the extent to which mechanical twins are produced in the steel. The mechanical twins are preferred sites for carbide nucleation during reactor operation. Although dislocations also act as nucleation sites, the twin boundaries have a higher thermal stability than dislocations and should make for a more stable precipitate distribution. Theoretically, the presence of precipitates can reduce swelling during irradiation, provided the number density of the precipitates is high.

Twinning is also the first step in martensite formation. This is of interest because the presence of martensite can be detected with a magnetometer or a low-frequency eddy-current device and its amount correlated with the degree of cold work by a particular process. The detection of martensite by a magnetic method is discussed in Sec. VIII.

VII. MARTENSITE FORMATION

According to Bogers and Burgers,¹⁶ the initial step in the formation of body-centered-cubic (bcc) martensite from an fcc structure is the formation of a twin. It follows that if twinning is affected by texture, the amount of martensite formed on cold working should also be dependent on the interaction of texture and the cold-working process.

To test this hypothesis, two cylindrical samples were prepared. One was given a true strain of 29% by tensile elongation, and the other a true strain of -29% by compression. After cold working, the two rods were cut in half and one-half of each rod was sensitized at 650°C for 24 hr. All four samples were magnetized, and their retained magnetic field was measured with a magnetometer. The measurements appear in Table VI.

TABLE VI. Magnetic Field Retained by Four Specimens of Strained Type 316 Stainless Steel^a

Condition	Percent True Strain	
	$\epsilon = 29$	$\epsilon = -29$
As-strained	0.32	0.09
Strained and sensitized (24 hr at 650°C)	4.17	0.12

^aValues are in milligauss per gram.

The magnetic field retained by the tensile specimen was more than three times that retained by the compressed specimen. Furthermore, the field retained by the sensitized tensile specimen was more than 30 times that of the sensitized compression specimen.

It can be concluded from this simple experiment that (1) the amount of martensite formed on cold-working Type 316 stainless steel is influenced by the texture of the steel and the process by which the steel is cold-worked, and (2) the tendency to form martensite on subsequent heat treatment is also influenced by the texture and loading. A tensile force applied along a $\langle 111 \rangle$ direction of the crystal appears to produce a material high in martensite.

VIII. MAGNETIC MEASUREMENT OF COLD WORK

Austenitic stainless steels such as Types 316 and 304 are weakly magnetic in the solution-annealed condition. Their initial relative permeabilities usually fall in the range below 1.01, but their magnetic properties change when they are cold-worked--drastically for Types 302 and 304, but only slightly for Types 310 and 316.¹⁷ The weak magnetic properties of Type 316 both before and after cold working considerably complicated the application of any magnetic measurement as a nondestructive indication of cold work. Nevertheless, since the number of possible nondestructive methods applicable to this problem was limited, it was decided to devote some effort to the development of a method of detecting and measuring the magnetic characteristics of Type 316 stainless steel.

A number of magnetic properties may be selected individually or in combination to indicate the presence of martensite in the steel. These include permeability at some particular value of magnetic intensity, coercive force, hysteresis loss, and retentivity. This latter property, also called remanence, or residual induction, was selected as the easiest to detect and measure. Retentivity is a measure of the flux density (B) when the magnetic intensity (H) is zero.

Retentivity measurements were first made on the solid-rod specimens produced by simple tensile elongation. These rods contained a sufficient mass of material to produce a retained field after magnetization that could be detected by a high-sensitivity (1 mOe full scale) magnetometer. The specimens were magnetized across their diameters. The results showed a high degree of correlation between retentivity and reduction in rod area for reductions over 20%. Below this value, the magnetometer did not possess sufficient sensitivity to make meaningful measurements. Figure 21 shows the results of measurements on a rod that contains 29-39% reduction in area.

Fig. 21

Percent Reduction and Field Normal to the Rod as a Function of Distance along the Rod. Results from tensile rod 3. Neg. No. MSD-52165.

These results were sufficiently encouraging to justify the search for some method of increasing the sensitivity of the retentivity method. Greatly enhanced sensitivity was required if any measurements on thin-walled tubing were to be attempted. Tubing has a much lower mass per unit length available to develop a magnetic field compared with solid-rod specimens of comparable diameter. The diagram of Fig. 22 explains the method finally developed. The unfiltered output of the magnetometer at maximum sensitivity presents a signal that contains large components of 60-Hz hum, wideband noise, low-frequency drift, and random variations from small changes in the local magnetic field. Hidden in this signal is the mechanically modulated 10-Hz signal produced by the weak magnetism of the specimen rotating at 10 rps. This 10-Hz signal is preferentially amplified by the narrow-band filter and demodulated to produce a signal that can be displayed on a meter or pen recorder.

Fig. 22

Steps in Magnetic Retentivity Measurement of Cold Work in Stainless Steel Tubing. In Step 1, the tube is demagnetized by rotation through a strong dc field and translation through an ac field to remove all residual radial and axial fields. In Step 2, the tube is linearly translated through a strong, unidirectional magnetic field so as to be magnetized along a diameter. In Step 3, the specimen is rotated at a constant rate past the magnetometer probe. For a nearly linear read-out, the modulator could be designed to be sensitive to the log of the output of the filter. Neg. No. MSD-57850.

Some experimental results from this equipment are shown in Fig. 23. The retentivity appears to build exponentially as a function of the cold work.

Fig. 23

Retentivity Measurements on Cold-worked Tubing from Different Manufacturers.

Curve A represents material from CarTech HT V87210, as-received; curve A' represents the same material after a light etch to remove surface cold-worked material. Curves B and B' are similar, but represent material from Superior HT 172783. Neg. No. MSD-57851.

For this reason, a highly worked but thin layer of material on the surface of the tube has an important influence on the output of the test system. As can be seen from Fig. 23, a light etch of the tube surface (<0.0025 -mm or 0.1-mil reduction on the diameter) has a significant effect on the measured values of retentivity. Light buffing of the surface of a tube with fine emery cloth produces a large increase in the measured retentivity.

The mechanically modulated retentivity method was also used to test the tubing described in Table III. Part of the information in Table III and measured retentivity values for each tube are reproduced in Table VII. It is clear that there is a wide variation in measured retentivity and, presumably, in the amount of martensite in these tubes when compared with the relatively narrow spread in microhardness. Unfortunately, some of the tubes had been belt-sanded and others had not, but even after etching, considerable spread in the measured retentivity values was still observed. These data are not surprising, since it had already been concluded that the amount of martensite formed during cold working of Type 316 stainless steel is influenced by the texture of the steel and the process by which the steel is cold-worked.

TABLE VII. Microhardness^a and Retentivity Measurements of Round-robin Tubes

Condition	Type of Measurement	Supplier				
		CarTech	Fine	Sandveik	Superior	Wolverine
Solution-annealed	Hardness	$\bar{X} = 149$	$\bar{X} = 147$	$\bar{X} = 149$	$\bar{X} = 145$	$\bar{X} = 147$
	Relative retentivity	0	0	0	0	0
20% cold-worked	Hardness	$\bar{X} = 310$	$\bar{X} = 303$	$\bar{X} = 300$	$\bar{X} = 297$	$\bar{X} = 302$
	Relative retentivity	0.53	0.58	0.91	1.04	1.20

^aSee Table III.

Tests on the flat-rolled specimens of various compositions described earlier (Fig. 5) were undertaken to assess the effect of chemical composition on the magnetic retentivity of the steel. For these measurements, a turntable that rotated at 10 rps was constructed. The specimens were magnetized parallel to their longest dimensions and placed on the turntable under the magnetometer probe. Rotation of the turntable produced a pulsed

Fig. 24. Retentivity vs Cold Work for Flat-rolled Specimens of Fig. 5. Neg. No. MSD-56890.

variation of high harmonic content in the magnetometer output. The spectrum of this pulse had a peak near 30 Hz; therefore, a 30-Hz filter was used to enhance the output signal-to-noise ratio of the magnetometer. This filter, together with the relatively greater mass of the rolled plates, resulted in a system output signal with low noise. The results of the measurements are shown in Fig. 24. The sensitivity of the retentivity and presumably the martensite concentration to chemical composition appear to be established by these data, but unfortunately there is a complicating factor. During the metallographic preparation necessary for microhardness measurements, it was discovered that many of the flat-plate specimens contained large amounts of carbide precipitates along the grain boundaries and, in some cases, even stringers of free carbon. Since this condition is bound to affect martensite formation, the results shown in Fig. 24 are almost certain to have been

influenced by the condition of the specimens. However, the microhardness values of the flat-plate specimens given in Fig. 9 are probably essentially correct, because microhardness measurements are made within individual grains. It is possible that the dip in the curves of Fig. 24 for values of reduction in the range of 10-20% is associated with the faulty structure of the specimens, although no thesis can be offered that might explain this dip. Research on the program was terminated before this point could be clarified.

IX. CRITICAL-ANGLE REFLECTIVITY MEASUREMENTS OF COLD WORK

A beam of ultrasonic energy incident at an angle on a liquid-solid interface will be reflected at the same angle. The amplitude of the reflected energy remains almost constant over the entire range of incident angles, except at the Rayleigh critical angle, where a sharp reduction

occurs. At this angle, a large part of the incident energy is converted into surface waves on the solid at the liquid-solid interface. The classical mathematical model fails to predict the behavior of the reflected beam at the critical angle. Becker and Richardson^{18,19} developed a model that takes into account damping of vibrations in solids and hysteresis between stress and strain. This model accurately describes the amplitude and phase relation of the reflected beam around the critical angle. The dependence of the critical-angle position or phase transition on material properties makes it an effective method of detecting changes of these properties in the reflecting medium. The values obtained are the result of average variations in a layer one wavelength deep, the penetration depth of Rayleigh surface waves. Variation of frequency causes variation of penetration depth and enables the evaluation of properties at different thicknesses or the homogeneity of properties across a cross section.

A special instrument was built to measure the effect of cold work on the position of the critical-angle reflection minimum. The minimum-position method was chosen, rather than the phase-transition measurement, because of simplicity and unsophisticated equipment. The apparatus (Fig. 25) consists of a double goniometer, centrally motor driven and connected to a linear potentiometer, which supplies the voltage reference for the angular position. The 10-MHz ultrasonic transmitter is mounted on one arm of the goniometer, and the other arm carries the 10-MHz receiver. A gated high-frequency voltage is applied to the transmitter from a gated power

Fig. 25. Test Arrangement for Rayleigh Critical-angle Reflection Measurements. ANL Neg. No. 306-1428.

oscillator. The receiver signal is fed through a preamplifier and gated amplifier to a peak detector. The position reference voltage is applied to the x axis of an x-y recorder, and the output of the peak detector is fed into the y-input terminal. An angular sweep around the critical angle generates a curve that shows the relationship between reflected-energy amplitude and angular position. The transmitter and receiver are mounted on small goniometers that are attached to x-y micropositioners for fine alignment. The angular displacement is continuously monitored by an indicator gauge that measures the vertical displacement of the driver disk. From the known geometry, the angle can be accurately computed, and nonlinearities of the reference potentiometer or voltage variations eliminated. With a 5° sweep that resulted in a 300-mm trace length, the accuracy was within 1 mm or 1 min of arc.

Initial alignment of transmitter and receiver is the most time-consuming portion of the procedure. It was achieved by applying a pulse-echo method separately to each transducer. A small metal sphere, placed in the pivot plane of the double goniometer, served as the reflector. An angular sweep with the sphere as the reflector resulted in a uniform output with proper alignment. The use of bounded beams results in a lateral beam displacement that necessitates a lateral transducer translation at the critical angle to the point of minimum amplitude.

The position of the Rayleigh critical angle was plotted for each cold-work group, which yielded a family of curves as shown in Fig. 26. The measurements were made in a direction perpendicular to the rolling direction of the specimen, because of greater changes in the position of the critical angle. The critical-angle positions for each percentage of cold work were averaged and plotted as shown in Fig. 27.

Fig. 26. Reflection Amplitude as a Function of Critical Angle. Neg. No. MSD-57517.

Fig. 27. Rayleigh Critical Angle as a Function of Cold-work Percentages. Neg. No. MSD-56621.

Fig. 28. Critical Angle as a Function of Microhardness. Neg. No. MSD-57515.

The results show that a correlation exists between the percentage of cold work and the critical-angle position. The effect of chemistry on the critical angle is slight although, to assess it statistically, a large number of samples would have to be measured and evaluated. To compare the microhardness results with critical-angle measurements, a curve that relates these values is shown in Fig. 28.

It was noted during the measurements that a greater change in the critical angle occurs in the direction perpendicular, rather than parallel, to the rolling operation of the specimen. To determine if these changes are gradual for angles in between, a polar plot (Fig. 29) of the critical angle as a function of rolling direction was obtained.

Fig. 29. Critical Angle as a Function of Rolling Direction of Cold-worked Type 316 Stainless Steel. Neg. No. MSD-56971.

The polar plot of the critical angle as a function of the rolling direction for different cold-work percentages suggests that the texture contributes the most to the shift in critical-angle position. To verify this assumption, one set of samples with different cold-work percentages was completely annealed to remove the work hardening. The critical angle after annealing was the same as that obtained before annealing for all but the 50% cold-work sample. It appears that annealing the samples after 50% cold work caused sufficient alteration of the texture of the steel to be detectable by critical-angle measurements, but 30% or less cold work did not.

To estimate the relationship between position shift of the critical angle and grain deformation due to work hardening, grain boundaries per unit length were counted in the direction perpendicular and parallel to the deformation. From the known dependence of the critical angle on the velocity change, the following correlation between the angle variation and grain elongation was established:

$$\frac{\Delta\theta}{\theta} = 0.063 \frac{\Delta n}{n} = 0.063 \frac{\Delta l}{l},$$

where θ is the critical angle, n is the number of grain boundaries per unit length, and l is the grain diameter. The proportionality constant will vary with material and texture, and the given relation is only valid for the curves displayed in Fig. 29.

Fig. 30. Rayleigh Critical Angle as a Function of Cold Work at Two Frequencies. Neg. No. MSD-57516.

Variation of material properties with depth was studied by determining the critical angle at 10 and 2.3 MHz. The results are shown in Fig. 30.

Preparation of samples for the microhardness test revealed regions of carbon stringers in grain boundaries. These carbon stringers may have slightly influenced the microhardness measurements taken within a grain boundary (50-g load); they also altered the critical-angle reflectivity test, thereby masking the effect of chemical composition. This assumption was confirmed when the specimens were checked at a lower frequency. Lowering the ultrasonic frequency theoretically¹⁹ shifts the critical angle to a lower value. Figure 30 indicates the opposite, which suggests a decrease of ultrasonic velocities and an increase in damping with penetration depth caused by scattering at the carbon stringers. The density of these carbon stringers increased with depth in the specimen.

X. MÖSSBAUER MEASUREMENT OF COLD WORK

Mössbauer spectroscopy^{20,21} has become a useful tool for metallurgical and chemical analyses. The technique has been shown to be particularly useful for analysis of iron-based materials. For example, it has been possible to differentiate between austenite, martensite, ferrite, and cementite²²⁻²⁴ and to determine variations such as carbon content in martensite and austenite.^{25,26} Effects of cold work in nickel-iron alloys have also been observed by Mössbauer methods.²⁷

Initial Mössbauer tests in this program were made with special thin foils (0.0254 mm thick) of Type 316 stainless steel used in a conventional through-transmission manner. Included were tests of similar foils that had been only solution-annealed, or solution-annealed and then cold-worked 25%. A slightly reduced line width was observed for the cold-worked material.²⁸ However, it was recognized that through-transmission Mössbauer tests for cold work would present a number of practical difficulties. These included not only the impossibility of investigating samples thicker than ~0.05 mm, but also the variable results that could be obtained because of slight differences in sample thickness.

Emphasis shifted to backscatter Mössbauer methods^{29,30} with which tests could be made on the same samples studied in other aspects of the program. Tests were made with equipment at Northern Illinois University³¹ and Carnegie-Mellon University.³² Professor P. A. Flinn kindly furnished several backscatter Mössbauer measurements of sample AM 145 (Fig. 5) observed in the following conditions: solution-annealed and cold-worked 10, 20, 30, and 50%. The line widths observed in this series of tests did not vary significantly, nor did they present any trend toward a particular direction.³²

The same samples were later sensitized at 650°C for 20 min to induce a small amount of carbide precipitation. The samples were tested again by backscatter Mössbauer spectroscopy with results similar to those indicated above. The only consistent comparison between the results on the as-rolled and sensitized samples was that the line widths of the sensitized samples were smaller than those of the as-rolled samples.³² This indicated that Mössbauer spectroscopy could possibly be useful for observing carbide precipitates in Type 316 stainless steel. However, the investigation to check that possibility thoroughly was not completed.

Aside from the single attractive possibility that Mössbauer measurements could be used in a practical backscatter mode to differentiate between various carbide precipitate amounts in Type 316 stainless steel, the tests on this aspect of the program were negative. The limited tests indicated that Mössbauer spectroscopy was not particularly sensitive to cold work in Type 316 stainless steel nor to allowable chemistry variations within the Type 316 specification.

XI. SUMMARY AND CONCLUSIONS

For a given fabrication process performed on a fully annealed Type 316 stainless steel with an analysis comparable to that given in Table I, microhardness and grain-shape measurements are adequate indicators of percent true strain at room temperature. Both the number of deformation twins and the amount of martensite produced are functions of (1) the texture of the steel at the time it is cold-worked and (2) the cold-working process; both are high when the individual austenite crystals are submitted to a tensile load applied parallel to a $\langle 111 \rangle$ direction.

Magnetic retentivity can be used to indicate the amount of cold work in Type 316 stainless steel. A nondestructive test system can be built that will be suitable for mill operation and that will measure the weak fields produced by Type 316 stainless steel, but the system would have to be recalibrated for every change in fabrication procedure or heat of steel.

The Rayleigh critical-angle reflection method is a useful tool to evaluate work-hardening conditions if some precautions are taken:

- a. A standard sample of identical chemical composition, grain size, and texture is necessary for calibration.
- b. The sample under investigation must be checked in several directions to determine its rolling or work-hardening direction.
- c. The sample should be measured in the direction perpendicular to the rolling operation for maximum sensitivity.
- d. A calibration curve that relates cold work to critical-angle position for this material is essential to determine the actual unknown percentage of cold work.

Mössbauer spectroscopy does not appear to be a useful nondestructive test to indicate cold work in Type 316 stainless steel, because the changes observed for this property and for allowable variations in chemistry were not statistically significant. On the other hand, carbide precipitation, another characteristic of stainless steel that may be related to improved reactor performance, appears to lend itself to detection by the backscatter Mössbauer method. If this characteristic proves important, the relative insensitivity of the Mössbauer results to cold work or chemistry could lead to a nondestructive test uniquely sensitive to carbide precipitation in this stainless steel.

ACKNOWLEDGMENTS

The authors thank C. N. Reid, The Open University, Bletchley, Buckinghamshire, England, for his assistance in evaluating the effect of texture on twinning and M. H. Mueller for the X-ray diffraction work.

REFERENCES

1. A. L. Schaeffler, *Metal Prog.* 56, 680 (1949).
2. B. Weiss and R. Stickler, *Phase Instabilities During High Temperature Exposure of 316 Austenitic Stainless Steel*, Westinghouse Scientific Paper 70-1D4-STABL-P1 (July 1970).
3. L. Colombier and J. Hochmann, *Stainless and Heat Resisting Steels*, Edward Arnold Ltd., London, p. 16 (1967).
4. J. D. Embury, A. S. Keh, and R. M. Fisher, *Trans. AIME* 236, 1252-1260 (1966).
5. F. Garofalo and H. A. Wriedt, *Acta Met.* 10, 1007 (1962).
6. J. A. Mazza, *J. Iron and Steel Inst.* 204, 783 (1966).
7. F. Garofalo, F. Von Gemmingen, and F. Domis, *Trans. Am. Soc. Metals* 54, 430-443 (1961).
8. R. Stickler and G. R. Booker, "Application of Selected-area Electron Channeling Patterns Obtained in the SEM to Deformation Studies," in *Electron Microscopy and Structure of Materials*, Gareth Thomas, ed., Univ. of California Press, Berkeley, pp. 301-312 (1972).
9. J. E. Flinn and E. M. Philofsky, *Trans. Am. Soc. Metals* 61, 37-41 (1968).
10. E. M. Philofsky and J. E. Hilliard, *Quart. Appl. Math.* 27, 79-86 (1969).
11. V. F. Riegels, *Archiv. d. Math.* 2, 117-125 (1949).
12. C. N. Reid, A. Gilbert, and G. T. Hahn, *Acta Met.* 14, 975 (1966).
13. C. E. Richards and C. N. Reid, *Trans. AIME* 242, 1831 (1968).
14. J. A. Venables, *Deformation Twinning in fcc Metals*, R. E. Reed Hill et al., eds., Gordon and Breach, New York, p. 77 (1964).
15. M. A. Jaswon and D. B. Dove, *Acta Cryst.* 9, 621 (1956).
16. A. J. Bogers and W. G. Burgers, *Acta Met.* 12, 255 (1964).
17. J. K. Stanley, *Electrical and Magnetic Properties of Metals*, Am. Soc. Metals, Metals Park, Ohio, p. 309 (1963).
18. F. I. Becker, *Phase Measurements of Reflected Ultrasonic Waves Near the Rayleigh Critical Angle*, BNWL-SA-3188 (Mar 1970).
19. F. I. Becker and R. L. Richardson, "Ultrasonic Critical Angle Reflectivity," in *Research Techniques in Nondestructive Testing*, R. S. Sharpe, ed., Academic Press, New York, pp. 91-131 (1970).
20. H. Frauenfelder, *The Mössbauer Effect*, W. A. Benjamin, New York (1963).
21. G. K. Wertheim, *Mössbauer Effect: Principles and Applications*, Academic Press, New York (1964).
22. C. W. Kocher, *Phys. Letters* 14, 287-289 (1965).
23. P. A. Flinn, *Advances in Materials Research--Experimental Methods of Materials Research, Vol. 1*, Herbert Herman, ed., Interscience, New York, pp. 163-192 (1967).

REFERENCES

24. M. Ron, A. Kidron, H. Schechter, and S. Niedzwiedz, *J. Appl. Phys.* **38**, 590-594 (1967).
25. P. M. Gielen and R. Kaplow, *Acta Met.* **15**, 49-63 (1967).
26. T. Zemcik, *Phys. Letters A* **24**, 148-150 (1967).
27. Y. Tino and J. Anai, *J. Phys. Soc. (Japan)* **32**, 941-948 (1972).
28. S. L. Ruby, Argonne National Laboratory, private communication (1971).
29. J. H. Terrell and J. J. Spijkerman, *Appl. Phys. Letters* **13**, 11-13 (1968).
30. R. N. Ord, *Appl. Phys. Letters* **15**, 279-282 (1969).
31. C. W. Kimball and W. C. Harper, Northern Illinois University, De Kalb, private communication (1971).
32. P. A. Flinn, Carnegie-Mellon University, Pittsburgh, Pa., private communications (1971-1972).