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METALLURGICAL FACTORS INFLUENCING THE
MAGNETIC ANALYSIS OF SURFACE HARDENED
AND TEMPERED STEEL

by

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AND TEMPERED STEEL

ABSTRACT

The purpose of this work was to determine quantitatively the effects of residual stress and retained austenite on measurements by magnetic analysis of surface hardened (by carburizing) and tempered steel.

A quantitative relationship has been determined between magnetic measurements and surface residual stresses in carburized and hardened H-8620 steel as these stresses were progressively relaxed by tempering at 250°F and 350°F at a constant level of retained austenite.

A quantitative relationship has been determined between magnetic measurements and uniform compressive stress in hot rolled 3145 steel between the stress limits of 2550 and 12750 psi.

Preliminary investigations of varying percentages of retained austenite in H-8620 steel appear to indicate that, so long as the austenite remains untransformed, its effect is small relative to residual stress effects.

When carburized and hardened H-8620 steel is tempered 15 1/2 minutes at 500°F, a substantially greater effect is observed on magnetic measurements than can be accounted for on the basis of residual stress alone. No explanation for this effect has been found, to date.

A time dependency of magnetic measurements has been noted. Magnetic imbalance has been observed between identically carburized and hardened H-8620 steels separated only by a period of three months in time of heat treatment. There has not been sufficient experimental evidence accumulated to date to justify an explanation of this effect.

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- II. Sample data sheet and calculations of retained austenite from x-ray data.

METALLURGICAL FACTORS INFLUENCING THE
MAGNETIC ANALYSIS OF SURFACE HARDENED
AND TEMPERED STEEL

INTRODUCTION

The application of magnetic measurements to inspection of heat treated steels is an attractive means of quality control. The advantages of the method are found in the fact that it is a nondestructive test which can be made economically. Magnetic analysis, however, can be sensitive to a wide variety of both mechanical and metallurgical conditions such as mechanical discontinuities, amount of cold work, shape and distribution of phases, and even relatively small variations in analysis of certain chemical components (1) (2)¹. If, therefore, an inspection is to be totally meaningful, it is necessary to identify that specific condition within a sample which is responsible for the magnetic measurement. Zuschlag (3) states that, "The resultant distortion of the alternating magnetic field or of any voltage induced by the field must be measurable and interpretable as an indicator of the condition that caused the deviation in the magnetic characteristics."

It is the reason of the problems indicated above that the investigation, the results of which are given in this report, was undertaken. In view of the substantial variety of test sample conditions involved, this work has been limited to surface residual stress and retained austenite effects occasioned by the carburizing and hardening and subsequent tempering of a single heat of H-8620 steel. An attempt has been made to assess quantitatively the separate effects of these parameters on magnetic measurements.

¹The numbers in parentheses pertain to references appended to this report.

EXPERIMENTAL PROCEDURE

Material:

All test coupons used in this work were taken from a single bar of H-8620 steel in the annealed condition as supplied by The Springfield Armory. Preliminary metallographic examination of this steel showed banding. Magnetic measurements on as received stock revealed an orientation effect. This effect was small at low sensitivity settings of the comparator and was less with transverse sections than with longitudinal sections. The observed sensitivity was attributed to banding, but it was felt that relative to effects resulting from residual stress and austenite, the banding would not preclude the use of the steel as intended. All data related to residual stress effects reported herein are taken from test coupons cut transversely from the original bar stock, unless otherwise stated.

Equipment:

Magnetic measurements were made on a commercial Magnetic Analysis Production Comparator, Serial No. CP490 (D-7273882-A; S-61-3665) on loan to The University of Connecticut by The Springfield Armory. In order to obtain better pattern resolution and a more quantitative evaluation of observed patterns than obtainable with the oscilloscope on the commercial comparator, a Tektronix, Type 551 Dual-Beam oscilloscope with graduated scale was utilized to indicate the differential voltage from the test coils.

Residual stress and retained austenite determinations were carried out on a General Electric Co. XRD-5 x-ray diffractometer.

Sample polishing for diffraction work was done on a Buehler Electro-Polisher.

An x-ray stress constant for H-8620 steel was determined on a calibration rig designed by the authors and constructed in the Experimental Shop of the School of Engineering. This calibration rig was designed to stress two "matched" tensile specimens simultaneously. The "matched" specimens were obtained by machining a thicker sample to length, width and loading pin dimensions, slitting, hardening, and tempering the resulting specimens, finish grinding to close tolerances and stress relieving after grinding. The utilization of a "matched" pair in the calibration rig allowed SR-4 gages to be attached to one of the pair while the second similarly stressed member of the pair could be x-ray irradiated for ((211)) peak position determinations. Since loading was accomplished in this rig by forces transmitted through an Instron load cell, both SR-4 and load cell data could be used for computation of applied stresses. Stresses computed by the two methods were in excellent agreement.

Procedure:

At the outset of the investigation, it was recognized that replication of magnetic readings was of prime importance. All incidental effects of specimen size and shape, position in the test coils, variations in operations of heat treatment and specimen polishing would have to be controlled within proper limits. To this end, a number of trial treatments and processing techniques were evaluated. A standard specimen size and shape together with reproducible processing techniques were ultimately established as itemized in the following:

1. Specimens (transverse) were finish ground to ± 0.001 " tolerance on all dimensions in the form of squares $3/4$ " on a side and $1/4$ " thick.

2. Carburizing and hardening was carried out on one sample at a time by packing in a refractory crucible with a measured volume of compound and heating for 1 1/2 hours total furnace time. After carburizing, the individual sample was shaken from crucible to a wire basket (to separate compound) and oil quenched.

3. Carburized and hardened samples were then directly electro-polished for 20 seconds with no mechanical preliminary polishing. This polishing was found consistently to remove 0.0005" from the sample surface.

Excellent replication of magnetic readings was found to be obtainable by comparisons of many samples processed as outlined in the preceding. Further, it was found that subsequent tempering treatments for the temperatures and times employed in this work did not necessitate re-polishing of samples for diffraction studies.

Following the heat treatment and polishing, magnetic measurements were made by balancing any two coupons and then noting the degree of imbalance when other similarly processed coupons were substituted for one of the originally balanced pair. The majority of these measurements were taken at a current setting of 2 and a sensitivity setting of 3 1/2. One-inch I.D. coils were used with machined maple sample positioning fixtures. With the exception of one or two specimens (which were discarded) similarly processed samples were found to balance one another within such narrow limits of deviation as to be negligible. In excess of thirty specimens were processed and compared during the course of this work.

Following surface hardening, polishing and magnetic balancing of samples, x-ray measurements were taken for residual stress calculations. These data were obtained in the form of time for 10^5 counts at $\psi = 0^\circ$ and 10^6 counts at $\psi = 60^\circ$ (lying in a principal plane of the standard

sample) in $1/2^\circ$ increments of 2θ between 154° and $157 1/2^\circ$ values of 2θ for the ((211)) peak of mertensite. Chromium K-alpha radiation was employed and the average of three or more counts at each $1/2^\circ$ position of the diffractometer was recorded. No filtering* of radiation was used, but a pulse height selector was employed and adjusted to give a maximum peak to background ratio. Separate background data were recorded for this steel and extrapolated to 2θ values under the ((211)) peak for purposes of corrections in calculations of residual stress after the method of Koistinen and Marburger (4). While the Lorentz-Polarization and Flat Plate corrections after Koistinen and Marburger were utilized, attempts to use their three point peak calculation method led to inconsistent values of residual stress. To remedy this situation, a 5 point least squares calculation based on the parabola was adopted. The precision of residual stress measurements was thus improved, but it is felt that further improvements are possible in any future work by utilizing a 9 point least squares peak calculation in conjunction with an improved peak to background ratio.

The x-ray stress constant for H-8620 steel was determined in the tension calibration rig. Tensile stresses were calculated from load cell indications in the rig and checked against SR-4 strain gage measurements on precision machined "matched" pairs of hardened and tempered tensile specimens. A total of eleven determinations at stresses between 5,000 and 50,000 psi were made.

The procedure employed for determining the effect of residual stress was based on the fact that excellent replication of magnetic read-

*Recent experimentation appears to indicate that a combination of vanadium filter and the pulse height selector will permit better precision in residual stress determinations by virtue of the improved peak to background ratio.

ings was obtained in freshly quenched samples. Also, reasonably consistent values of surface residual stress were determined. Three freshly hardened samples showing magnetic balance in any combination which were also determined to have similar values of residual stress and retained austenite within the accepted limits of uncertainty were selected. Magnetic balance readings between any two as a reference pair were recorded as well as balance readings between one of the reference pair and the third sample. The third sample was then tempered at a desired temperature for a relatively short period of time. After air cooling from the tempering, the magnetic readings of the reference pair were repeated after which the tempered sample was compared magnetically with the one sample with which it had been compared prior to tempering. Any imbalance between tempered and reference sample was recorded in volts. The tempered sample was then evaluated for both residual stress and retained austenite. This sample was then re-tempered for a longer period at the given temperature and the above described measurements repeated. By this technique data showing the degree of imbalance in volts depending on the degree of stress relaxation by tempering were obtained.

In order to determine the magnitude of magnetic imbalance resulting from a uniform stress, a cylindrical bar of 3145 was stressed elastically in an Instron testing machine. This stressed bar in a test coil was compared with an unstressed bar in the second test coil. Compressional stresses in 2550 psi increments up to 12750 psi were applied and corresponding imbalance voltages recorded. It was necessary to use a current setting of 1 and a sensitivity of 1 in this work in order to obtain a satisfactory unstressed balance adjustment.

Measurements of percentage by volume of retained austenite were also made on each sample after every heat treatment. Standard techniques

involving planimetry of areas under the ((200)), ((220)) δ^1 and the ((200)), ((211)) α peaks were employed. Reasonably consistent results have been obtained when the retained austenite is calculated from all four integrated intensity ratios. It has been noted that values of retained austenite calculated from the two ratio combinations involving the ((211)) α peak in the as hardened steel tend to be slightly higher. This is attributed to the broadness of the ((211)) peak, and the fact that the high angle limit of the diffractometer does not permit a true background level to be reached on this high angle side of the peak, if high compressive stresses are present.

Preliminary work intended to assess the effect of varying amounts of retained austenite was carried on by employing refrigeration as well as liquid nitrogen quenching. In this case, the magnitudes of voltage imbalance between an oil quenched reference sample at a given level of retained austenite and a differently quenched or refrigerated sample having a different amount of retained austenite were recorded and evaluated.

RESULTS AND DISCUSSION

Retained Austenite:

The work designed to indicate the effect of retained austenite on magnetic analysis measurements was undertaken immediately following the experimental establishment of the fact that replication of magnetic readings could be obtained, using the heat treating and preparational procedures described under Experimental Procedure. In order to observe the effects which changing orientation of test samples in the coils indicated, it was necessary to use high sensitivity settings on the comparator, or high current settings, or both. It was also necessary to

use these settings when standard samples were compared with similarly carburized, oil quenched and refrigerated samples and with carburized and liquid nitrogen quenched samples.

Table I gives results which are representative of these comparisons. The data are arranged to show the orientation effects as well as effects which could reflect differences in surface percentage volume of retained austenite.

From examination of these data, it will be noted that orientation effects are more pronounced in longitudinal than in transverse samples. This difference was attributed to the banded microstructure of the steel.

Further, it will be noted that orientation effects in longitudinal samples are greater than those noted when samples having different amounts of retained austenite are compared. Similar comparisons on transverse samples do show some apparent influence which might be attributed to austenite, but these differences were reduced to a negligible amount when the comparator current setting was reduced from 7 to 2. All later results on residual stress effects were determined at current settings of 2 with a sensitivity of $3 \frac{1}{2}$. Since significant stress influences were noted at these settings, and because the effects of retained austenite were found to be negligible and for the most part less than orientation effects, it was concluded that the effects of retained austenite were minor relative to residual stress effects. While retained austenite in the amount ranging from 40 to 50% by volume would suggest a substantial magnetic effect, it should be borne in mind that these values represent surface determinations on a carburized and hardened steel. Fig. 1 shows a typical hardness-distance plot for H-8620 so treated. The low surface hardness is attributed to the high retained austenite content. Peak

hardness at a depth of 0.010" to 0.012" is presumably associated with a minimum retained austenite content at this depth. Using this plot as an indirect measure of austenite distribution, it is estimated that percentage of austenite based on the total volume of steel sample in the magnetic field is on the order of 3 percent or less.

Residual stress determinations were not made on a majority of those specimens for which data are given in Table I. Coleman and Simpson (5) in an earlier study of "Residual Stresses in Carburized Steels" stated that, "Refrigeration transforms much of the retained austenite, but does not result in a significant change in the residual stress."*

Residual Stress:

In order to calculate residual stresses from diffraction data, it was necessary to determine the x-ray stress constant for heat treated H-8620 steel. The calibration data taken on oil quenched and tempered "matched" tensile specimens are plotted in Fig. 2. These data were obtained in the rig (earlier described) using $\Delta 2\theta$ values computed from ((211)) peak determinations at $\psi = 0$ and $\psi = 45^\circ$. The least squares stress constant converted to $\Delta 2\theta$ for $\psi = 0$ and $\psi = 60^\circ$, was found to be 57,000 psi per degree of $\Delta 2\theta$. A value of 53,000 psi per degree of $\Delta 2\theta$ reported in the Fifth Monthly Progress Report dated February 13, 1963, was found to be in error when data were recalculated in preparation for this final report.

The fact that stress will affect the magnetization of ferromagnetic materials is well known and has been the subject of many investigations (6).

*More recent residual stress measurements made since the completion of the retained austenite comparisons appear to indicate some basis for questioning this conclusion.

TABLE I
Representative Data Showing Effects of Orientation and Retained Austenite on Magnetic Measurements of Surface Hardened H-8620 Steel - 2.0" I.D. Test Coils

Sample No. **	Heat Treatment	Volume Austenite-%	Comparator Settings		Magnetic Imbalance Maximum Oscilloscope Deviation - Volts Orientation	Orientation Difference Δ Volts	Austenite Difference Δ Volts
			Sens.	Curr.			
3L	Solid Carburize 1650°F, 1 1/2 hrs Oil Quench	40-45	6	7	Reference Sample	Reference Sample	Reference Sample
1L	"	40-45	6	7	2.5	3.7	---
2L	"	40-45	6	7	0.2	5.0	---
4L	"	40-45	6	7	2.1	5.8	---
5L	Solid Carburize 1650°F, 1 1/2 hrs Liq. Nitrogen Quench	20	6	7	1.2	3.7	1L-5L, 1.3, 1.3 2L-5L, 1.0, 0.3 4L-5L, 0.9, 3.0
1L	Solid Carburize 1650°F, 1 1/2 hrs Oil Quench	43	3 1/2	7	Reference Sample	Reference Sample	Reference Sample
10	"	45 1/2	3 1/2	7	0.07	0.51	---
8	"	40	3 1/2	7	0.33	0.29	---
9	Solid Carburize 1650°F, 1 1/2 hrs Oil Quench Refrig'n -100°F 1 hr	25	3 1/2	7	1.55	0.55	10-9, 1.48, 0.42 8-9, 1.22, 0.38

* Orientation differences are by a 90° rotation about an axis perpendicular to the cylindrical axis of the test coil.
 ** The letter L indicates a sample cut longitudinally from original bar of H-8620.

Whether such an effect could be observed under the stress conditions existent in the standard samples selected for this study remained to be determined.

For purposes of assessing the magnitude of the stress influence on magnetic measurements, compression loads were applied to a one-inch bar of A.I.S.I. 3145 steel in an Instron testing machine. The experiment which is described under Experimental Procedure permitted magnetic comparisons with a mating unstressed bar of steel. The results of this series of tests are plotted in Fig. 3. The data represent the mean imbalance voltage at each stress level of repeated loadings and unloadings. Excellent replication of imbalance readings was noted in repeated loadings, and the effects of stresses well below 2000 psi could be detected magnetically.

With the knowledge that the effects of relatively small unidirectional stress could be detected by the instrument, relaxation of residually stressed samples by low temperature tempering was initiated. It was hoped that, despite the complexity of the residual stress distribution in the hardened coupons, an "overall" stress effect might be detected.

The results of these experiments are summarized in plot form in Fig. 4. The procedure followed in obtaining these results has been outlined. In all of this work, the highly stressed as quenched specimens were used as the reference state. The measured values of surface retained austenite are also indicated. It can be seen that this parameter was virtually constant within the limits of uncertainty of the measurement. The scatter of the volts vs. residual stress data is attributed to two factors. The x-ray method of residual stress measurement at best does not yield high precision, and this fact is undoubtedly reflected in these

data. A small orientation effect was also noted in making the magnetic measurements. There is no question, however, of the fact that the measurement is sensitive to variation in surface residual stress. While it is convenient to measure surface stress in one principal direction of the specimen, the symmetry of the specimen suggests that a biaxial equi-stress condition must obtain. In a few specimens, measurements were made at ninety degrees to one another thus representing both principal directions in the sample. These measurements were found to confirm the anticipated biaxiality. In view of this, the surface stress measurement in one principal direction becomes a convenient quantitative index of the mechanical state of the sample. It is possible that stored strain energy would be a more basic parameter to use for representing the state of the steel.

Comparisons of the data show that stresses were not so extensively relaxed by tempering at 250°F. The maximum tempering time at 250°F was 315 minutes, and the stress was reduced from 46,000 psi to 29,000 psi.

Some tempering experiments at 212°F for times up to 120 minutes failed to yield significant changes in magnetic measurements, and the residual stress was not significantly altered.

When a similar series of experiments was run at a tempering temperature of 500°F, a substantially greater magnetic effect was observed. The maximum magnetic deviation resulting from tempering at 350°F for 120 minutes was 0.84 volts. A similarly hardened sample having essentially the same initial residual surface stress and retained austenite when tempered for 15 1/2 minutes at 500°F resulted in a deviation voltage of 1.33. In this case, the retained austenite was lowered from 35% to 27% and the broadness of the ((211)) peak was reduced by comparison with those

found in all samples tempered at lower temperatures. It is evident from these observations that metallurgical changes in addition to stress relaxation are in process during tempering at 500°F. Time did not allow a detailed study of these changes and identification of that condition which was responsible for the large magnetic change.

Aging Effect:

During the course of this investigation, a change in magnetic state of some of the earlier heat treated samples was observed. This effect was reported in the Third Progress Report dated December 5, 1963. Magnetic measurements made on 5 freshly hardened samples showed good magnetic similarity when compared with one another, but all showed deviations when compared with an intended reference pair which had been identically hardened some three months earlier. To date, it has not been possible to investigate the cause of this apparent aging.

CONCLUSIONS

The results of this investigation are summarized in the following:

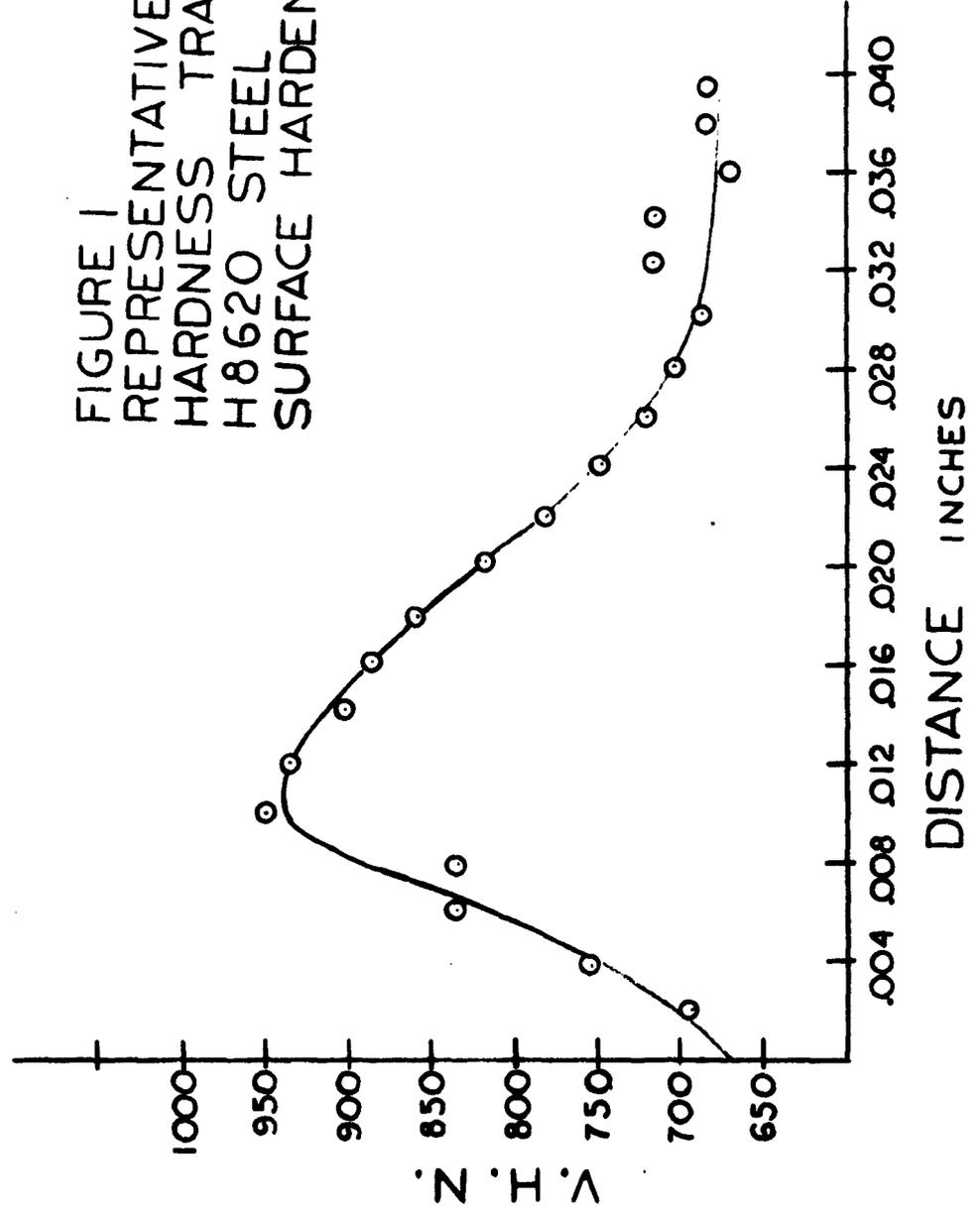
1. The residual stresses in surface hardened and tempered H-8620 steel have a significant effect on the magnetic behavior of this steel as measured by magnetic analysis.
2. The effect of untransformed, surface retained austenite in surface hardened H-8620 steel over the range from 20 to 50 per cent by volume is minor relative to residual stress effects and is of the order of orientation effects.
3. Surface residual stresses in surface hardened H-8620 can be relaxed by tempering at temperatures of 250°F and 350°F for times up to 300 minutes without significant change in the surface volume content of

retained austenite.

4. Tempering of surface hardened H-8620 steel at 212°F for times up to 120 minutes does not significantly reduce surface residual stress or significantly affect magnetic analysis measurements.

5. Tempering of surface hardened H-8620 steel at 500°F for 15 1/2 minutes results in a greater magnetic effect than can be accounted for on the basis of residual stress relaxation. The volume content of surface retained austenite is reduced by this tempering treatment.

FIGURE 1
REPRESENTATIVE VHN
HARDNESS TRAVERSE
H8620 STEEL
SURFACE HARDENED



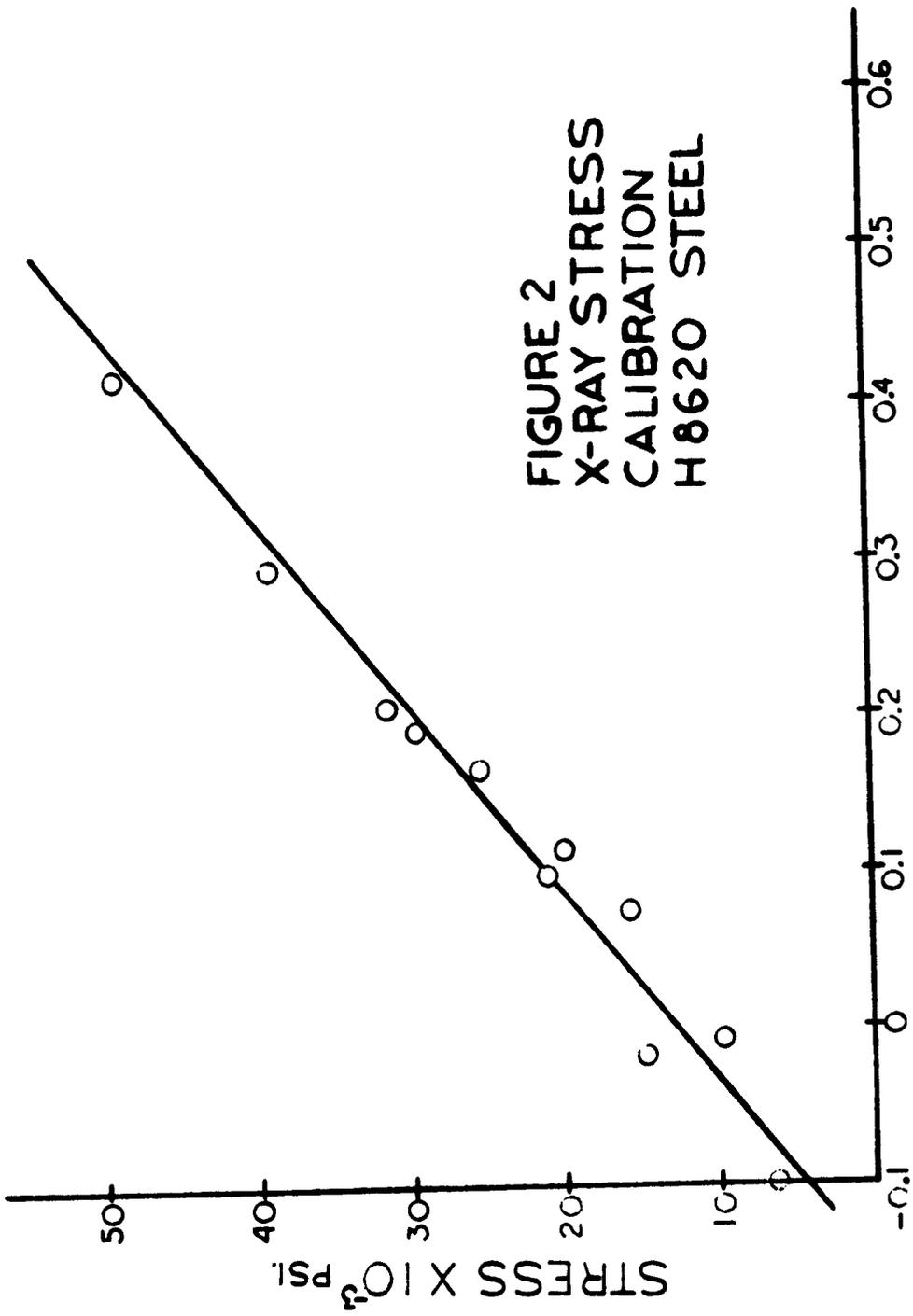
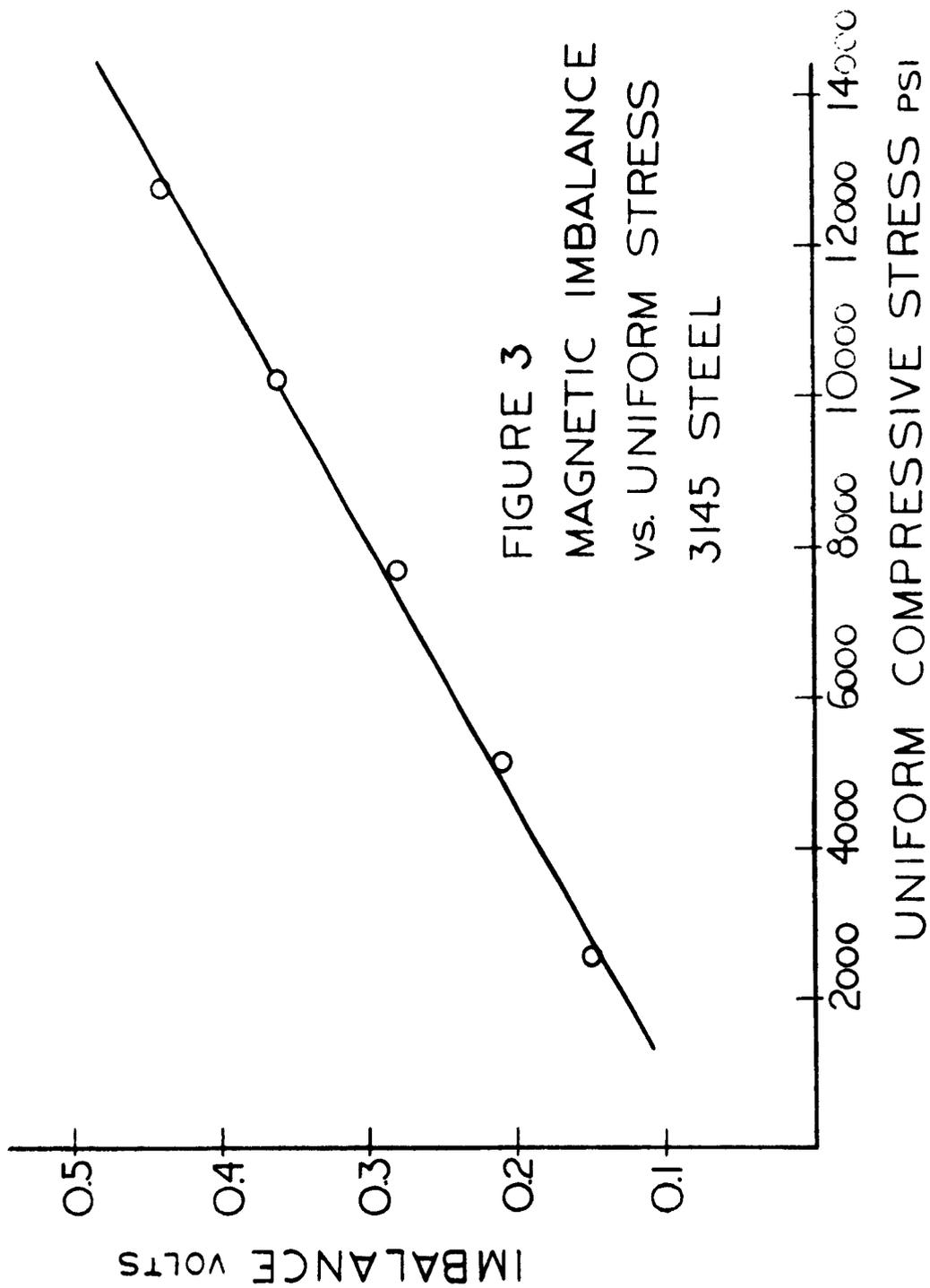
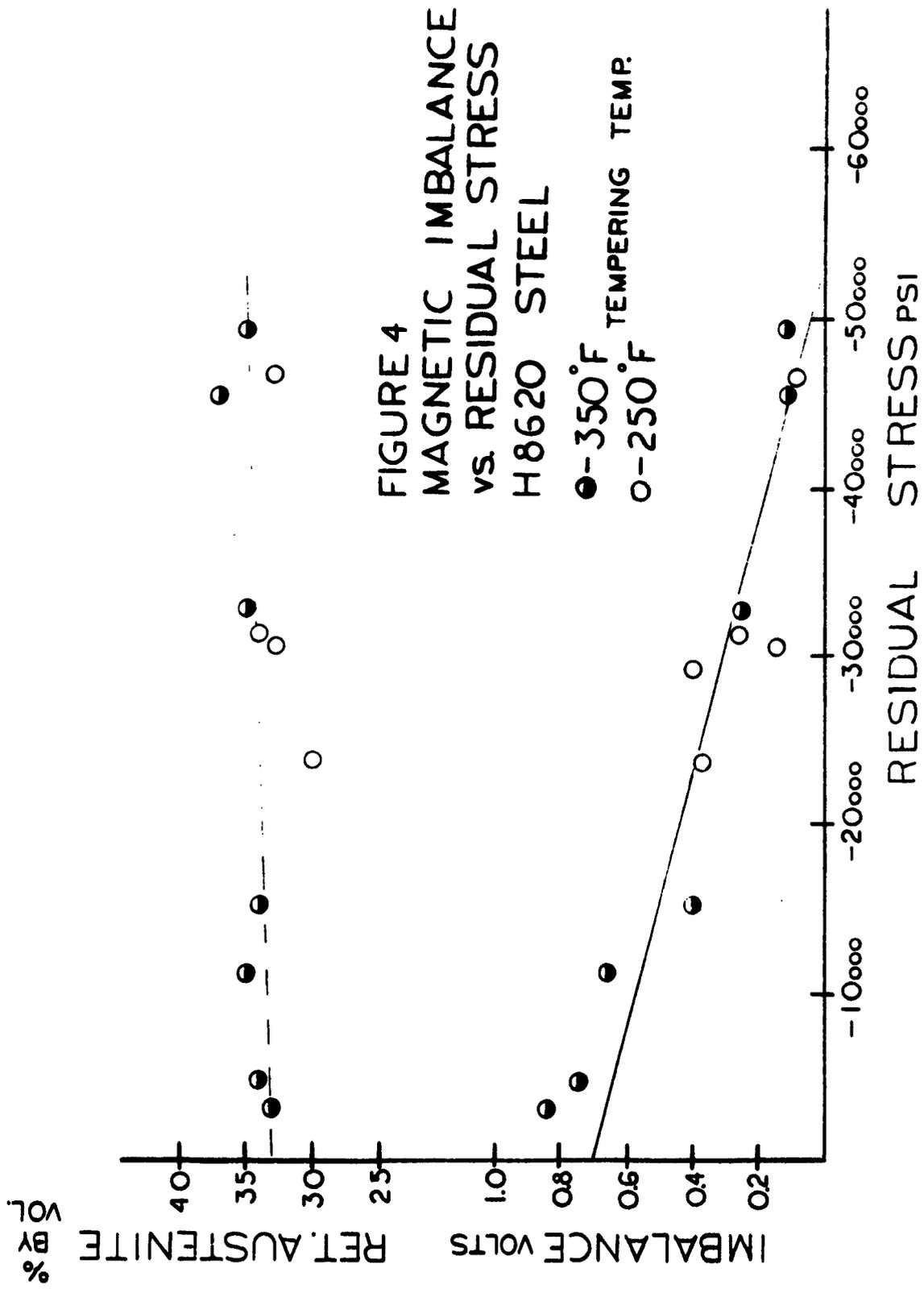


FIGURE 2
 X-RAY STRESS
 CALIBRATION
 H8620 STEEL

$$\Delta 2\theta = 2\theta_N - 2\theta_{45} \text{ DEG.}$$





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- (6) A. Cochardt "Magnetomechanical Damping", Chap. 11, p. 251 in the book, "Magnetic Properties of Metals and Alloys", 1959, American Society for Metals.

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APPENDIX I
Sample Data Sheet and Calculation of Residual Stress
from X-ray Data
Sample No. S.R.P. 32

$\psi = 0^\circ$					$\psi = 60^\circ$				
time(secs.) for 10^5 counts					time(secs.) for 10^6 counts				
2 θ	run 1	run 2	run 3	Ave.	2 θ	run 1	run 2	run 3	Ave.
153	14.94	14.93	14.96	14.943	154	59.95	59.93	59.93	59.937
153.5	14.74	14.77	14.74	14.750	154.5	58.75	58.69	58.72	58.720
154	14.59	14.62	14.55	14.587	155	57.75	57.75	57.71	57.737
154.5	14.41	14.38	14.38	14.390	155.5	56.99	56.97	57.05	57.003
155	14.41	14.34	14.35	14.367	156	56.60	56.56	56.53	56.563
155.5	14.43	14.46	14.49	14.460	156.5	56.29	56.37	56.33	56.330
156	14.58	14.62	14.57	14.590	157	56.28	56.36	56.34	56.327
					157.5	56.48	56.42	56.49	56.463

Flat Plate - Lorentz Polarization and Background Corrections

$\psi = 0^\circ$					$\psi = 60^\circ$				
2 θ	time	CPS	Bkg.	Corr'd.	2 θ	time	CPS	Bkg.	Corr'd.
153.5	14.750	6779.7	5347	172.8	154	59.937	16684.2	13998	528.8
154	14.587	6855.4	5353	177.5	154.5	58.720	17030.0	14100	557.5
154.5	14.390	6949.3	5359	184.0	155	57.737	17320.0	14200	573.6
155	14.367	6960.4	5366	180.6	155.5	57.003	17542.9	14300	576.2
155.5	14.460	6915.6	5372	171.1	156	56.563	17679.4	14400	562.7

Parabolic Least Squares Peak Determination

$\psi = 0^\circ$					$\psi = 60^\circ$				
2 θ	X	Y	XY	X ² Y	2 θ	X	Y	XY	X ² Y
153.5	0	172.8			154	0	528.8		
154	1	177.5			154.5	1	557.5		
154.5	2	184.0			155	2	573.6		
155	3	180.6			155.5	3	576.2		
155.5	4	171.1			156	4	562.7		
Sum		886.0	1771.7	5276.5	Sum		2798.8	5684.1	17040.9

Least Squares Coefficients

$\psi = 0$	$\psi = 60$
$a = 2.736$	$a = 6.993$
$b = 10.913$	$b = 36.622$
Peak 2 $\theta = 154.497$	Peak 2 $\theta = 155.309$

$\Delta 2\theta = 0.812$

For Stress $K = 57,000 \text{ psi}/\Delta 2\theta^\circ$; $C = 46,000 \text{ psi}$.

APPENDIX II
 Sample Data Sheet and Calculations of
 Retained Austenite from X-ray Data
 Sample No. S.R.P. 32

Mean Values of Planimeted Peak Areas Above Background
 and Calculated Constants

Peak	Area	R calc'd
200 γ'	1.000	28.15
200 α	1.333	15.98
220 γ'	1.380	40.10
211 α	9.195	148.49

Retained Austenite Calculations

$$C_{\gamma'} = \frac{1}{1 + \frac{R_{\gamma'} I_{\alpha}}{R_{\alpha} I_{\gamma'}}$$

$$\frac{R_{200 \gamma'} I_{200 \alpha}}{R_{200 \alpha} I_{200 \gamma'}} = (1.762)(1.333) = 2.349 ; \frac{1}{2.349} = 0.2985$$

$$\frac{R_{220 \gamma'} I_{200 \alpha}}{R_{200 \alpha} I_{220 \gamma'}} = (2.509)(0.9659) = 2.423 ; \frac{1}{2.423} = 0.2921$$

$$\frac{R_{200 \gamma'} I_{211 \alpha}}{R_{211 \alpha} I_{200 \gamma'}} = (0.1896)(9.195) = 1.743 ; \frac{1}{1.743} = 0.3645$$

$$\frac{R_{220 \gamma'} I_{211 \alpha}}{R_{211 \alpha} I_{220 \gamma'}} = (0.2701)(6.663) = 1.800 ; \frac{1}{1.800} = 0.3571$$

Unclassified

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R. R. Biederman
Date: April 5, 1963

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A nondestructive evaluation of the effects of surface residual stress and untransformed austenite on the magnetic analysis of steel shows a quantitative relationship between magnetic imbalance and surface residual stress in carburized and hardened H-8620 steel. Stresses were progressively relaxed by low temperature tempering holding retained austenite constant.

Effects of retained austenite on magnetic imbalance were found to be small and of the order of magnitude of orientational effects attributed to banding.

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