THE EFFECTS OF NITROGEN CONCENTRATION BETWEEN 0.27 PER CENT AND 1.30 PER CENT ON INTERNAL FRICTION PEAKS IN 304L STAINLESS STEEL

by

Richard Crockett Nickols, Jr.

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INTRODUCTION

The purpose of this investigation was to determine the effect of nitrogen concentration on the internal friction peaks associated with nitrogen in AISI 304L stainless steel.

Internal friction peaks occur in metals as a result of the vibrational energy of the metals being converted into other forms of energy. The stress-induced preferential distribution of an interstitial atom is one example of this energy conversion. This effect occurs when the strain in a metal lags behind the application of the stress and is caused by the interstitial atoms changing place in the lattice. The measurement of this strain lag can be used to calculate the diffusion rates, activation energies, and solid solubilities of the interstitial atoms being investigated.

There has been relatively little work done on internal friction peaks associated with interstitial atoms in face-center cubic lattices until recently, because there was no satisfactory mechanism to explain the phenomenon. With the introduction by Cheng and Chang[1] of a satisfactory explanation of the mechanism, the investigation of this phenomenon has increased. Most of the previous work in this area was concerned with the effect of carbon on its associated internal friction peaks in austenitic stainless steel. Cofer[2] and Manning[3] found that an internal friction peak resulting from interstitial nitrogen was also present in austenitic stainless steel.

This investigation will describe the effect of the nitrogen con-

centration on the internal friction peaks found in austenitic stainless steel with nitrogen contents between 0.27 and 1.30.

A torsional pendulum incorporating a photoelectric measuring device was used to determine the internal friction spectrum. Where it was feasible, data were processed by using an IBM 1620 Data Processing System.

REVIEW OF LITERATURE

Zener[4] defines anelasticity or elastic aftereffect as the property of a solid in virtue of which stress and strain are not uniquely related in the preplastic range. One area of the anelasticity phenomenon, the damping capacity or internal friction of metals, has received the most attention in the solution of metallurgical problems. Entwhistle[5] states that damping capacity implies that the metal itself is able to convert vibrational energy into other forms of energy. Quantitatively, damping capacity is measured by the amount of energy transformed within the solid during one cycle of vibration. Difficulties arise principally because of the wide range of damping capacity which may be encountered and in determining what source of internal friction is being observed.

Four sources of internal friction in metals are the thermo-elastic effect, magneto-elastic effect, mechanical behavior of grain boundaries and stress-induced preferential distribution of interstitial solute atoms. This thesis will deal with the latter of these effects, the stress-induced preferential distribution of interstitial solute atoms.

Snoek[6], while investigating the magnetic aftereffect in alpha iron, discovered that these magnetic aftereffects depended upon the presence of either carbon or nitrogen in the iron. Further observation showed that the carbon and nitrogen were only active while in solution. Richter[7] discovered that this mechanism was found to be true also for another new phenomenon, that of elastic aftereffect,

and was able to show that both effects had the same strong dependence on temperature. Snoek[8] further investigated the elastic aftereffect of alpha iron loaded with nitrogen by making it part of a freely oscillating system and measuring the logarithmic decrement of the system. This was the first application of the torsional pendulum to measure the stress-induced preferential distribution of interstitial solute atoms. As a result of these investigations, Snoek[9] postulated that the interstitial atoms were distributed equally in the x, y, and z lattice directions and, if the lattice were stretched in the z direction, there would be a preference for y positions and an elastic aftereffect would be the result. This mechanism will be more completely described later. Through these observations Snoek[10] was the first to state the mechanisms by which the stress-induced preferential distribution of interstitial solute atoms caused an elastic aftereffect and, as a result, this phenomenon is referred to as the Snoek effect.

Measurements of the Snoek effect have been used in metallurgy to determine diffusion coefficients, solid solubilities and activation energies for various metal systems, and can be made because of the following relationship.. The interstitial atoms occupy octahedral interstitial positions in alpha iron and produce a localised tetragonal distortion. There will be a random distribution of the interstitial atoms in the unstressed lattice, but application of a stress along a $[\bar{1}00]$ direction favors a distribution by jumps of half the lattice parameter into sites having a tetragonal axis parallel to that of the

applied stress. This migration of atoms resulting from the application of the stress produces a strain which varies with time at a rate determined by the migration rates of the atoms from one interstitial site to another.

An interstitial atom in a face-centered cubic lattice will occupy the octahedral position at the center of the unit cell and the six face-centered nearest-neighbor atoms will be equally displaced; therefore, a stress will produce a spherical distortion and result in a uniform distribution of interstitial atoms along the x, y, and z axes. Because of this there can be no Snoek effect found in a face-centered cubic lattice from a geometrical standpoint. Internal friction studies by Rozin and Finkelshtein[11], Cofer[12] and Manning[13] indicate the presence of an internal friction peak caused by interstitial atoms in austenitic stainless steels.

Obviously, there is another mechanism by which the interstitial atom migrates to produce this observed elastic aftereffect. Entwhistle[14] suggests that one of the face-centered atoms is replaced by a substitutional atom of different size producing a distortion around the substitutional-interstitial atom pair which will be ellipsoidal and when under stress will preferentially orient itself so that the greatest tensile distortion is closest to the direction of applied tensile strain. Investigations by Cheng and Chang[15] led them to postulate that the internal friction peaks in face-centered cubic materials occurred through the preferential rotation of the axes of interstitial pairs, similar

to the mechanism[16] of solute pairs in substitutional solid solutions. They theorized that an interstitial atom may fill up a lattice vacancy thus becoming a substitutional atom. In the meantime, another interstitial atom jumps to the neighborhood of this substitutional atom forming a stable atom pair, one interstitial, one substitutional, through the release of thermal energy.

Wu and Wang[17] give more detailed explanation of this mechanism and also state the mechanism by which the internal friction peak is formed. Figure 1 shows a model of a face-centered cubic lattice with a vacancy at ($\frac{1}{2}$, $\frac{1}{2}$, 0). As an interstitial atom falls into the vacancy, another interstitial moves into z_1, forming an atom pair whose axis is parallel to the z axis. As a compressive stress is applied along the z-direction, the interstitial atom at z_1 tends to jump to one of the x or y interstices to relieve the lattice strain. Conversely, if a tensile stress is applied along the z-direction, an interstitial atom in the x or y interstices tends to jump into z_1 or z_2. Therefore, with a compressive stress, the atom pair rotates until its axis is parallel to axis of the tensile stress. This rotation of the atom pair requires that one atom jump from one position to another with the application stress producing a strain which varies with time at a rate determined by the migration rate of the atom jumping from one site to another. This mechanism is identical to the mechanism by which interstitials travel in a body-centered cubic lattice except for the distance traveled. In a face-centered cubic lattice the interstitial

jumps a mean distance of $\frac{\sqrt{2}}{2}$ a_o along ⟨110⟩, while in a body-centered cubic lattice the interstitial jumps a mean distance of ½ a_o along the ⟨100⟩, a_o being the lattice parameter in both cases. Because of the close similarity between these two internal friction mechanisms, the formulae applicable to the body-centered cubic mechanism can also be applied to the face-centered cubic mechanism.

This migration of interstitial atoms resulting from the application of a stress produces a strain which varies with time at a rate determined by the migration rates of the atoms from one interstitial site to another. Figure 2 shows a graphical representation of the strains involved. The anelastic strain ϵ_a stems from the interstitial atoms changing place. Measurements of the rate of decay of ϵ_a can be used to calculate the diffusion rates, activation energies, and solid solubilities of the system being investigated. A detailed discussion of these measurements and calculations will follow.

To determine the internal friction spectrum for a metal, all that is necessary is to measure the logarithmic decrement, δ as a function of temperature for a constant angular frequency. The logarithmic decrement is the logarithm of the ratio of successive amplitudes and is expressed as

$$\delta = LN \frac{A_N}{A_{N+1}} \qquad (1)$$

where A_n and A_{n+1} are the amplitude of successive oscillations of the torsional pendulum. δ can also be expressed for N oscillations as

$$\delta = \frac{1}{N} LN \frac{A}{A'} \qquad (2)$$

where A is the initial amplitude and A' is the amplitude after N oscillations. Once δ has been determined, the lag angle, α, can be found from the expression

$$\delta = \pi \alpha \qquad (3)$$

where α is the angular measurement of the strain lag behind the applied stress; a condition necessary for internal friction to occur. Dieter[18] states that for a standard linear solid (any material exhibiting anelasticity) there is only a single relaxation time, τ. τ is related to δ by the expression

$$\delta = E_S \pi \frac{\omega \tau}{1 + \omega^2 \tau^2} \qquad (4)$$

where E_S, the relaxation strength, is a constant of the metal, and ω is the angular frequency of vibration. Equation 4 is symmetrical in both ω and τ and has a maximum value when

$$\omega \tau = 1 \qquad (5)$$

In metals, τ varies exponentially with temperature so that

$$\tau = \tau_o \, EXP - \frac{\Delta H}{R T} \qquad (6)$$

For a given frequency (f_1) the relaxation time is expressed by

$$\tau = \frac{1}{\omega} \qquad (7)$$

and the peak occurs at a temperature (T_1). At another value of frequency (f_2) the relaxation peak will occur at a temperature (T_2). From the temperature dependence of relaxation time (Equation 6) the activation energy ΔH can be determined.

$$\Delta H = R \frac{LN(f_2/f_1)}{1/T_1 - 1/T_2} \qquad (8)$$

For a given relaxation time the diffusion coefficient of the interstitial atom is given by

$$D = \frac{a_o^2}{b\tau} \qquad (9)$$

where a_o is the lattice parameter and b is a constant depending upon the lattice structure.

Ke and Wang[19] observed internal friction peaks associated with the stress-induced diffusion of carbon in an austenitic stainless steel. They state that the height of the peak increased and the position of the peak shifted to a lower temperature with an increase in the carbon content. When they reduced the amount of carbon in solid solution by tempering, the height of the peak decreased and the peak shifted to a higher temperature. Since this peak was associated with a relaxation process, they postulated that the observed peak was associated with the stress-induced diffusion of the carbon in the specimen.

These observations and Wu and Wang's[20] proposed mechanism indicate that it is possible for stress-induced diffusion to occur in a face-centered cubic lattice.

Previous work at Virginia Polytechnic Institute by Cofer[21] and Manning[22] show that an internal friction peak resulting from the stress-induced diffusion of nitrogen in an austenitic stainless steel (AISI 304L) is also present. A survey of the literature revealed that no work has been done concerning the effect of the nitrogen concentration on the internal friction peaks associated with nitrogen in AISI 304L stainless steel.

INVESTIGATION

A. Object of Investigation:

It was indicated by previous work that it is possible to detect internal friction peaks caused by the movement of interstitial atoms in a contaminated face-centered cubic lattice. This investigation was concerned with the movement of interstitial nitrogen in an 18-8 stainless steel lattice. The concentration of the nitrogen was varied from 0.27 per cent to 1.30 per cent. The effects of the varying concentration on the internal friction peak associated with the nitrogen interstitial were investigated. Particular interest was focused on the activation energy for diffusion of the nitrogen atom at various levels of concentration. A simple quantitative method for nitrogen analysis in 18-8 stainless steel was also investigated.

The specimens were subjected to a frequency of one cycle per second over a temperature range from ambient to 300 degrees centigrade. These parameters were selected because previous investigations showed the internal friction peak of nitrogen interstitials to fall in this range of frequency and temperature. To permit the determination of activation energies at various levels of concentration, several specimens were subjected to a lower frequency of 0.48 cycles per second.

B. Method of Procedure:

The internal friction tests were made with a torsional pendulum apparatus similar to the one designed by Ke[23]. The torsional pendulum was mounted in a Marshall-split-type seven zone furnace which provided

uniform temperature along the length of the sample by utilizing variable transformers connected to separate zones of the furnace. A uniform temperature along the length of the sample is one of the most important test parameters of the torsional pendulum, and the above arrangement produced a temperature variation of no more than $\pm$ 1.5 degrees centigrade along the sample length. The furnace was mounted vertically and was supported by a drill press stand mounted on a heavy wooden base. This mounting arrangement provided a rigid mount and also abosrbed shocks from outside sources. A Honeywell "Electronic 17" temperature controller used in conjunction with an iron-constantan thermocouple was used to regulate the furnace temperature. The temperature range of the furnace was from 0 degrees to 1200 degrees centigrade.

Test specimens were hung in the furnace vertically with the top held firmly by a pin vise which was connected rigidly to the furnace top. The lower end of the specimen was connected to a pin vise which was attached to the pendulum. The pendulum consisted of a horizontally mounted ¼ inch rod with two adjustable weights. The frequency of the pendulum could be changed by moving the adjustable weights of the pendulum. Attached to the pendulum center was a one-inch-square mirror. A ¼ inch rod was attached to the bottom of the pendulum center and was immersed in a container of oil. This arrangement quickly dampened out any lateral movement of the pendulum. The torsional pendulum is shown in Figure 3. The total mass of the pendulum produced a longitudinal stress of 855 pounds per square inch in the specimen. Wert[24]

states that the longitudinal stress on the specimen is of no importance for the type of material used in this investigation if kept below 1000 pounds per square inch. The pendulum was set in motion by two electromagnets which were positioned to attract the adjustable pendulum weights. When the electromagnets were de-energized the pendulum was set in motion. This minimized initial lateral motion.

Internal friction was determined with the torsional pendulum by measuring respectively the logarithmic decrement and the frequency of the free torsional vibration of the wire specimen. These two parameters were measured by focusing the light beam from a small light source onto the mirror attached to the pendulum center. The reflected light beam was focused on a screen, Figure 4, positioned nine feet opposite the mirror. The screen contained five photoelectric cells positioned on a natural logarithmic scale. The photoelectric cells were connected to a multi-channel switching circuit, which is shown in Figure 5. The reflected light beam, as it passed each photoelectric cell, triggered a 110-volt relay in the switching circuit. The relay energized a pen on a multi-channel Esterline-Angus Model AW recorder which produced a permanent record on a strip chart, Figure 6, from which the logarithmic decrement and frequency of the pendulum could be calculated. These calculations were performed using an IBM 1620 Data Processing System. The use of the photoelectric measuring device reduced measuring error and simplified data reduction, and the use of the computer reduced calculation time and increased accuracy.

A value for the logarithmic decrement was obtained at eleven degree centigrade intervals from ambient temperature to 310 degrees centigrade for each specimen, and from these values the internal friction spectrum was plotted. The specimens were 8 ½ inches long, and care was taken when placing the specimen between the pin vises to insure that the specimen was located at the same position in the furnace for each test.

The wire being investigated was cold-drawn AISI 304L 0.025 inch diameter wire. The heat analysis of the wire is shown in Table 1. All specimens were prepared in the following manner. McDanel AP 35 alumina four-hole tubing with an inside hole diameter of 1/32 inch was used to support the wire during preparation. The tubing insured that the specimens were straight after preparation. The wire was cleaned in acetone to remove surface contamination before it was inserted into the tubing. All specimens were given a twelve-hour anneal at 1025 degrees centigrade in a vacuum to insure that the wire was fully austenitic before nitriding. The nitriding was accomplished by holding the wires at a temperature of 530 degrees centigrade and passing ammonia gas through the tubing containing the wires. At this temperature the ammonia dissociated, sending monoatomic nitrogen into the metal lattice. After nitriding the wires were given a homogenizing treatment to distribute the nitrogen evenly through the specimen. The homogenizing treatment consisted of holding the wires at 1000 degrees centigrade while ammonia gas was being passed through the tubing. The ammonia dissociated,

providing an equilibrium condition on the outer surface of the wires which kept the nitrogen in the wires from escaping. The nitrogen concentration of the specimens was varied by varying the exposure time of the specimens to the nitriding treatment. All specimens were quenched after homogenizing to insure that a sufficient number of vacancies existed in the lattice to accommodate the internal friction mechanism. The nitrogen content of the samples was determined by a phosphoric and perchloric acid distillation method.

C. Results:

Four groups of test specimens were prepared. The specimens in group one were annealed as-received wire and contained 0.04 per cent nitrogen. The specimens in group two were annealed, nitrided for six hours, homogenized for 24 hours, and quenched. The group two specimens contained 0.27 per cent nitrogen. Thespecimens in group three were annealed, nitrided for 48 hours, homogenized for 72 hours, and quenched. The group three specimens contained 0.60 per cent nitrogen. The specimens in group four were annealed, nitrided for 96 hours, homogenized for 96 hours, and quenched. The group four specimens contained 1.30 per cent nitrogen. A summary of the specimen preparation is shown in Table 2. One specimen of group four was divided into four equal segments along an eight inch length, and each segment was analyzed separately to determine if the nitrogen distribution was equal along the sample. These analyses showed that there was no significant difference in the nitrogen content along the length of the test specimen.

The amplitudes of internal friction values were plotted as a function of temperature over the range from ambient to 325 degrees centigrade. The internal friction spectra that resulted from this investigation are shown in Figures 7, 8, and 9. The spectra of specimens from groups one, two, three, and four run at 1.1 cycles per second are shown in Figure 7. The spectrums of specimens from groups two and four which were run at 0.48 cycles per second are shown in Figure 8. The internal friction peaks shifted to lower temperatures when run at a lower frequency allowing the activation energies for the different peaks to be calculated using Equation 8.

A metallographic investigation was made to aid in the interpretation of the internal friction measurements. Photomicrographs from this investigation are shown in Figures 11 and 12. The specimens containing 0.27, 0.60, and 1.30 per cent nitrogen were observed to be slightly magnetic before testing with the degree of magnetism increasing with increasing nitrogen content. Each specimen also became more magnetic after testing.

DISCUSSION OF RESULTS

Internal friction measurements by Ke and Yang[25] of carbon in gamma iron showed that the internal friction peaks associated with the carbon rise with an increase in the carbon content. A similar effect would be expected in the case of nitrogen in gamma iron.

The internal friction of the annealed as-received wire containing 0.04 per cent nitrogen is shown in Figure 7. The frequency was 1.1 cycles per second, and the data showed that there was no peak in the spectrum below 325 degrees centigrade. The internal friction spectrums of the specimens containing 0.27 per cent, 0.60 per cent, and 1.30 per cent nitrogen are also shown in Figure 7. The frequency for these specimens was 1.1 cycles per second, and the data showed that there was a peak common to each spectrum at about 126 degrees centigrade. This peak increases in height with an increase in nitrogen content and is assumed to be associated with the nitrogen present in the specimens. Shewmon[26] states that the magnitude of the internal friction peak associated with stress-induced diffusion at its maximum is proportional to the number of atoms or atom pairs which give rise to the peak in question. Thus the variation of the magnitude of the peak due to interstitial pairs gives the variation in the concentration of such pairs. A plot of peak heights as a function of nitrogen concentration is shown in Figure 10. This plot shows that the height of the peak is linear with the nitrogen concentration. This linear relationship intercepts the ordinate at a Q^{-1} value of 0.001 which corresponds

favorably with the value of the internal friction spectrum of the specimen containing 0.04 per cent nitrogen. This linear relationship could be used as a basis for the quantitative analysis of nitrogen in stainless steel. The lower limit of detection would appear to be around 0.25 per cent nitrogen because of the low magnitudes of the internal friction peaks in this range of concentration. The analysis made above shows that the proposed "interstitial pair" model of stress-induced preferential distribution of interstitial atoms in a face-centered cubic lattice is essentially correct.

The internal friction spectra of the specimens containing 0.27 per cent and 1.30 per cent nitrogen run at 0.48 cycles per second are shown in Figure 8. The internal friction peaks associated with the nitrogen have shifted to lower temperatures as expected. The use of two different frequencies produced internal friction peaks at two different temperatures which provided sufficient data to calculate the activation energy using Equation 8. The activation energy for diffusion at 0.27 per cent nitrogen was found to be about 40,000 calories per mole which is in general agreement with the values found by Ke and Wang[27] for carbon diffusion in an 18-8 type stainless steel. Ke and Wang also observed that the activation energy was lowered with an increase in carbon content. The activation energy for diffusion at 1.30 per cent nitrogen was found to be 9,400 calories per mole. This value was much less than expected but was in agreement with the activation energy calculated from half width measurements by Eckel and

Manning[28] for a 2.20 per cent nitrogen concentration in austenitic stainless steel. These observations showed that there was a decrease in the activation energy with an increase in nitrogen content.

Another internal friction peak was observed in the spectrum of the specimen containing 1.30 per cent nitrogen. This peak occurred at about 162 degrees centigrade for a frequency of 1.1 cycles per second and is shown in Figure 7. The peak also occurred in the spectrum for 1.30 per cent nitrogen run at 0.48 cycles per second but at a lower temperature of 152 degrees centigrade. This spectrum is shown in Figure 8. It was postulated by Eckel and Manning[29] that there could be two relaxation times in the internal friction peak associated with nitrogen in austenitic stainless steel that was observed by Manning[30]. They also calculated that the activation energy for the peak at the higher temperatures was 32,000 calories per mole. The calculated activation energy for the similar peak in this investigation was 34,000 calories per mole. Manning's measurements of logrithmic decay[31] were made by the visual observations of a reflected light beam on an engraved scale, and it is possible that this method of measurement could have prevented the resolution of the two peaks. It is probable that these two peaks, one postulated and one observed, are the same.

The question now is what causes the second peak in the spectrum. One possible answer is the interaction of nitrogen with an alloying element in the stainless steel. Darken and Gurry[32] state that the addition to steels of elements which form relatively stable nitrides

usually give rise to the precipitation of one of these nitrides rather than an iron nitride. One of these more stable nitrides is formed when nitrogen interacts with chromium. Dulis and Smith[33] have previously reported the formation of chromium nitrides in 304L stainless steel. They also stated that the depletion of chromium in the austenite by the formation of chromium nitride unbalanced the matrix composition so that some of the austenite transformed to ferrite. This would explain the change in magnetic properties of the specimens after testing. The specimen containing 1.30 per cent nitrogen run at 1.1 cycles per second was rerun at the same frequency, and this spectrum is shown in Figure 9. The first peak attributed to interstitial nitrogen is not observable, and the second peak has grown in magnitude. This would indicate that some of the nitrogen atoms that contributed to the stress-induced diffusion in the first run are not available to contribute to the stress-induced diffusion during the second run. Some of the nitrogen atoms probably diffused to some structural site favorable for the formation of chromium nitrides, and this could be one possible explanation for the disappearance of the first peak.

Dulis and Smith[34] observed that the chromium nitride precipitate in 304L stainless steel occurred both at the grain boundaries and within the grains; and that the latter precipitate had in some cases the appearance of pearlite, and in others was distinctly Widmanstatten. The structure described is very similar to Figure 11 which is the structure of the specimen containing 1.30 per cent nitrogen before

testing. Figure 12 shows the same specimen after testing. The structure of the specimen has changed with many more spheroid-shaped precipitates present and the pearlite and Widmanstatten structure having completely disappeared. The disappearance during the second run of the internal friction peak associated with the stress-induced diffusion of nitrogen and the change in the structure of the specimen indicate that the second peak probably occurs as a result of chromium nitride formation, chromium nitride transformation, or a combination of both.

CONCLUSIONS

The following conclusions can be made from this investigation concerning the effect of nitrogen concentration upon the internal friction peak associated with nitrogen in austenitic stainless steel.

(1) The amplitude of the internal friction peak associated with the stress-induced diffusion of the nitrogen interstitial increases in height with an increase in nitrogen content.

(2) The linear relationship between the amplitude of the internal friction peak and the nitrogen content could be used as a basis for a quantitative analysis of nitrogen in austenitic stainless steel.

(3) There is a decrease in the activation energy of diffusion with an increase in nitrogen content.

(4) A second internal friction peak located at a higher temperature than the stress-induced internal friction peak was observed, and was thought to result from a chromium-nitrogen interaction.

(5) The observed change in the magnetic properties of the specimens during testing probably occurred as a result of the depletion of chromium in the austenite by the formation of chromium nitrides which unbalanced the matrix composition causing the austenite to transform to ferrite.

SUMMARY

The internal friction spectra for AISI 304L stainless steel containing 0.04 per cent, 0.27 per cent, 0.60 per cent and 1.30 per cent nitrogen were determined using a torsional pendulum. The magnitude of the internal friction spectrum increased as a linear junction of nitrogen concentration and could be used as a basis for a quantitative analytical method of nitrogen determination in austenitic stainless steel. The activation energy of diffusion decreased with an increase in nitrogen content and ranged from 40,000 calories per mole for 0.27 per cent nitrogen to 9,400 calories per mole for 1.30 per cent nitrogen.

The presence of another internal friction peak was detected and was thought to occur from a chromium-nitrogen interaction. This premise was supported by the three following observations:

(1) The disappearance of the internal friction peak associated with the stress-induced diffusion of nitrogen and the increase in magnitude of the peak associated with the chromium-nitrogen interaction during the rerunning of a test specimen

(2) The increase in the magnetism of the specimens during testing because of an austenite-ferrite transformation

(3) The observed change in the metallographic structure of the specimen after testing from a pearlite type structure to one containing many small precipitates.

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TABLE 1

CHEMICAL ANALYSIS OF THE AISI TYPE 304L STAINLESS STEEL WIRE USED IN THE INVESTIGATION

Element	Per Cent
Carbon	0.070
Chromium	18.46
Nickel	10.28
Silicon	0.51
Sulfur	0.026
Manganese	1.02
Phosphorus	0.020

TABLE 2

SUMMARY OF SPECIMEN PREPARATION

GROUP	NITRIDING TIME HOURS AT 530 °C	HOMOGENIZING TIME HOURS AT 1000 °C	NITROGEN CONTENT WEIGHT PER CENT
1	0	0	0.04
2	6	24	0.27
3	48	72	0.60
4	96	96	1.30

All specimens were annealed for 12 hours at 1025 degrees centigrade before nitriding.

TABLE 3

INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 0.04 PER CENT NITROGEN

Temperature, °C	Logarithmic Decrement, Q^{-1}
29.4	.00131
40.6	.00134
48.9	.00135
60.0	.00125
71.1	.00127
82.2	.00117
93.3	.00122
104.4	.00120
126.7	.00119
136.7	.00116
148.9	.00114
160.0	.00115
171.1	.00115
182.2	.00115
192.2	.00112
202.8	.00116
213.9	.00111
226.7	.00110
236.7	.00112
248.9	.00111
259.4	.00111
271.1	.00111
282.2	.00110
292.2	.00110

Frequency of pendulum was 1.1 cycles per second.

TABLE 4

INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 0.27 PER CENT NITROGEN

Temperature, °C	Logarithmic Decrement, Q^{-1}
26.7	.00281
37.8	.00283
48.9	.00295
60.0	.00308
71.1	.00304
82.2	.00295
93.3	.00309
115.6	.00286
125.0	.00295
137.8	.00273
148.9	.00271
160.0	.00263
173.9	.00246
183.3	.00237
192.2	.00231
203.9	.00224
215.6	.00218
227.7	.00219
236.7	.00230
248.9	.00219
260.0	.00231
271.1	.00239
282.2	.00228
294.4	.00231
304.4	.00232
315.6	.00241

Frequency of pendulum was 1.1 cycles per second.

TABLE 5

INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 0.60 PER CENT NITROGEN

Temperature, °C	Logarithmic Decrement, Q^{-1}
28.3	.00234
37.8	.00262
49.4	.00267
60.0	.00280
70.0	.00291
82.2	.00315
93.3	.00326
104.4	.00378
115.6	.00357
126.7	.00382
137.8	.00379
148.9	.00360
160.0	.00355
171.1	.00326
182.2	.00311
192.2	.00292
204.4	.00280
215.6	.00265
226.7	.00263
236.1	.00251
247.8	.00241
260.0	.00242
270.0	.00261
282.2	.00238
290.6	.00240
301.7	.00246
312.8	.00258

Frequency of pendulum was 1.1 cycles per second.

TABLE 6

INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 1.30 PER CENT NITROGEN

Temperature, °C	Logarithmic Decrement, Q^{-1}
30.6	.00652
37.8	.00691
48.9	.00652
60.0	.00706
71.1	.00706
82.2	.00792
93.3	.00816
104.4	.00846
115.6	.00846
126.7	.00978
137.8	.00902
148.9	.01016
160.0	.01098
171.1	.01009
182.2	.00939
191.7	.00873
204.4	.00770
214.4	.00745
225.6	.00701
237.2	.00648
248.9	.00573
260.0	.00588
270.0	.00562
279.4	.00575
292.2	.00631
302.8	.00648
313.9	.00648

Frequency of pendulum was 1.1 cycles per second.

Specimen 1, Run 1.

TABLE 7

INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 0.27 PER CENT NITROGEN

Temperature, °C	Logarithmic Decrement, Q^{-1}
29.4	.00343
37.8	.00344
48.9	.00329
60.0	.00341
71.1	.00365
82.2	.00370
93.3	.00336
104.4	.00359
115.6	.00364
126.7	.00364
137.8	.00353
148.9	.00342
160.0	.00347
171.1	.00378
181.1	.00350
192.2	.00328
204.4	.00316
215.6	.00305
226.7	.00300
237.8	.00293
248.9	.00266
260.0	.00272
271.1	.00272
282.2	.00271
293.3	.00266
304.4	.00272
313.3	.00272

Frequency of pendulum was 0.48 cycles per second.

TABLE 8

INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 1.30 PER CENT NITROGEN

Temperature, ^{o}C	Logarithmic Decrement, Q^{-1}
26.7	.00616
40.6	.00619
48.9	.00650
60.0	.00676
71.1	.00759
82.2	.00834
93.3	.00891
104.4	.00859
115.6	.00828
126.7	.00731
137.8	.00754
150.0	.01254
160.0	.01190
171.1	.00774
182.2	.00684
193.3	.00606
204.4	.00626
215.6	.00626
226.7	.00653
237.8	.00650
248.9	.00672
260.0	.00672
268.3	.00668
282.2	.00700
293.3	.00688
303.3	.00726

Frequency of pendulum was 0.48 cycles per second.

TABLE 9

INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 1.30 PER CENT NITROGEN

Temperature, °C	Logarithmic Decrement, Q^{-1}
32.2	.00210
37.8	.00222
48.9	.00255
60.0	.00274
71.1	.00301
82.2	.00330
93.3	.00387
104.4	.00406
115.6	.00422
126.7	.00535
137.8	.00865
148.9	.01307
160.0	.01365
171.7	.01131
182.2	.00919
193.3	.00795
204.4	.00692
215.6	.00726
226.7	.00629
237.8	.00629
248.9	.00629
260.0	.00639
271.1	.00661
281.1	.00676
293.3	.00735
303.3	.00726

Frequency of pendulum was 1.1 cycles per second.

Specimen 1, Run 2.

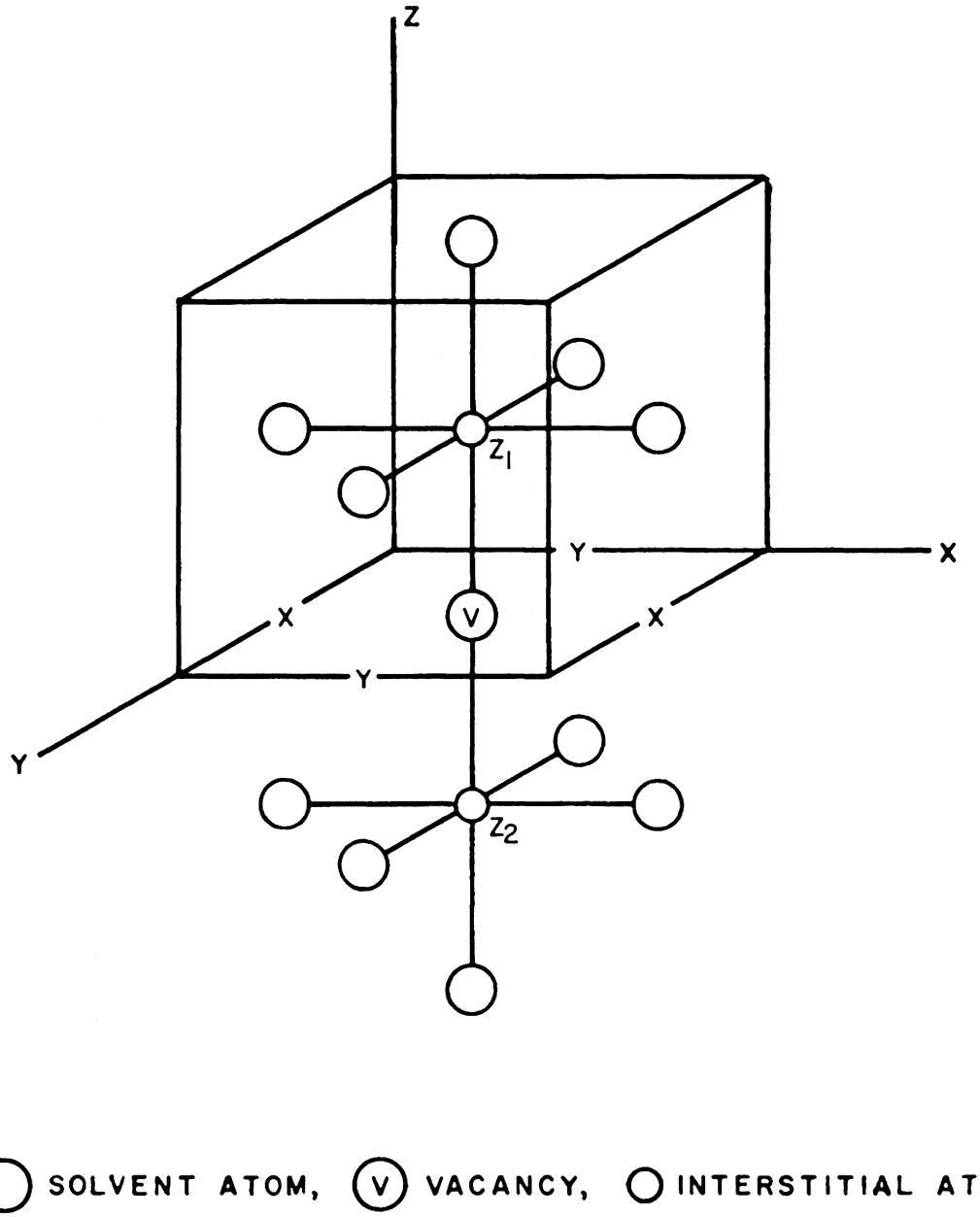

FIGURE 1. A FACE-CENTERED CUBIC LATTICE SHOWING THE PAIR ROTATIONAL MECHANISM.

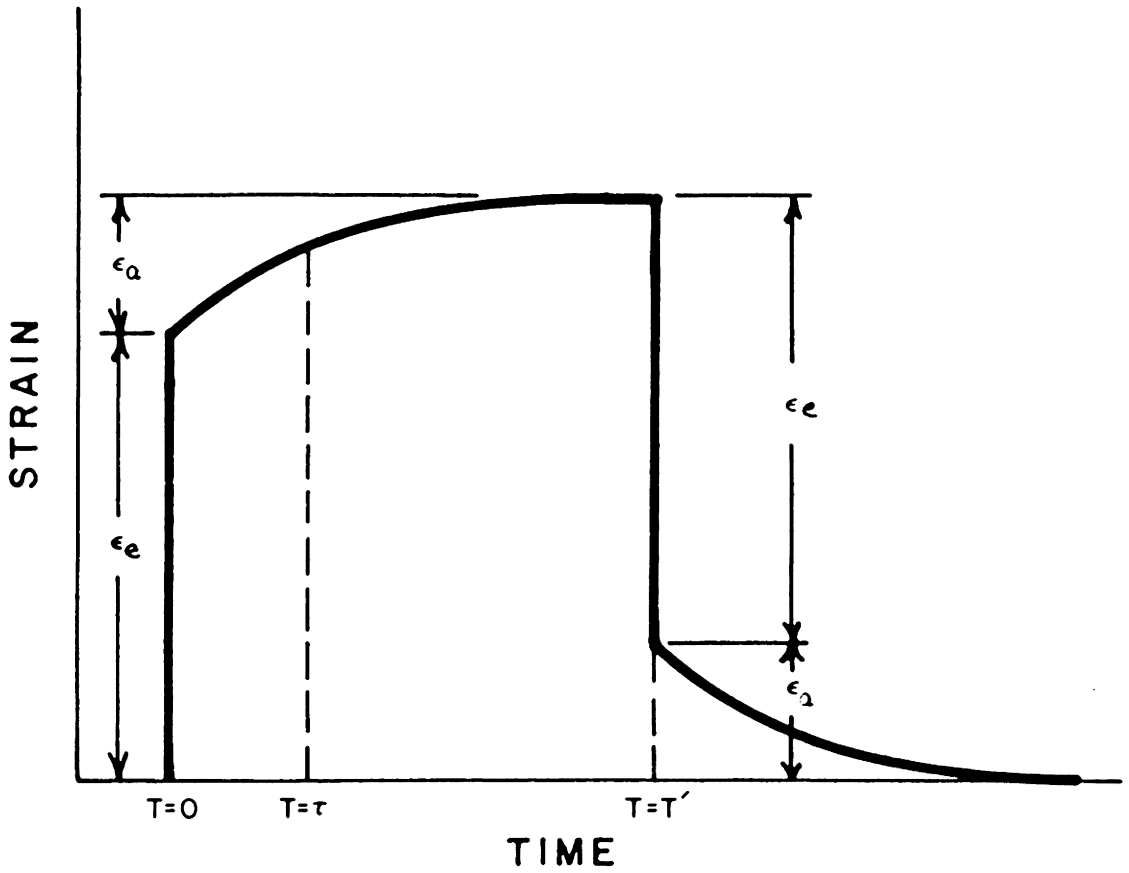

FIGURE 2. STRAIN VERSUS TIME FOR AN ANELASTIC SPECIMEN WHEN A LOAD IS APPLIED AT T=0 AND REMOVED AT T=T'.

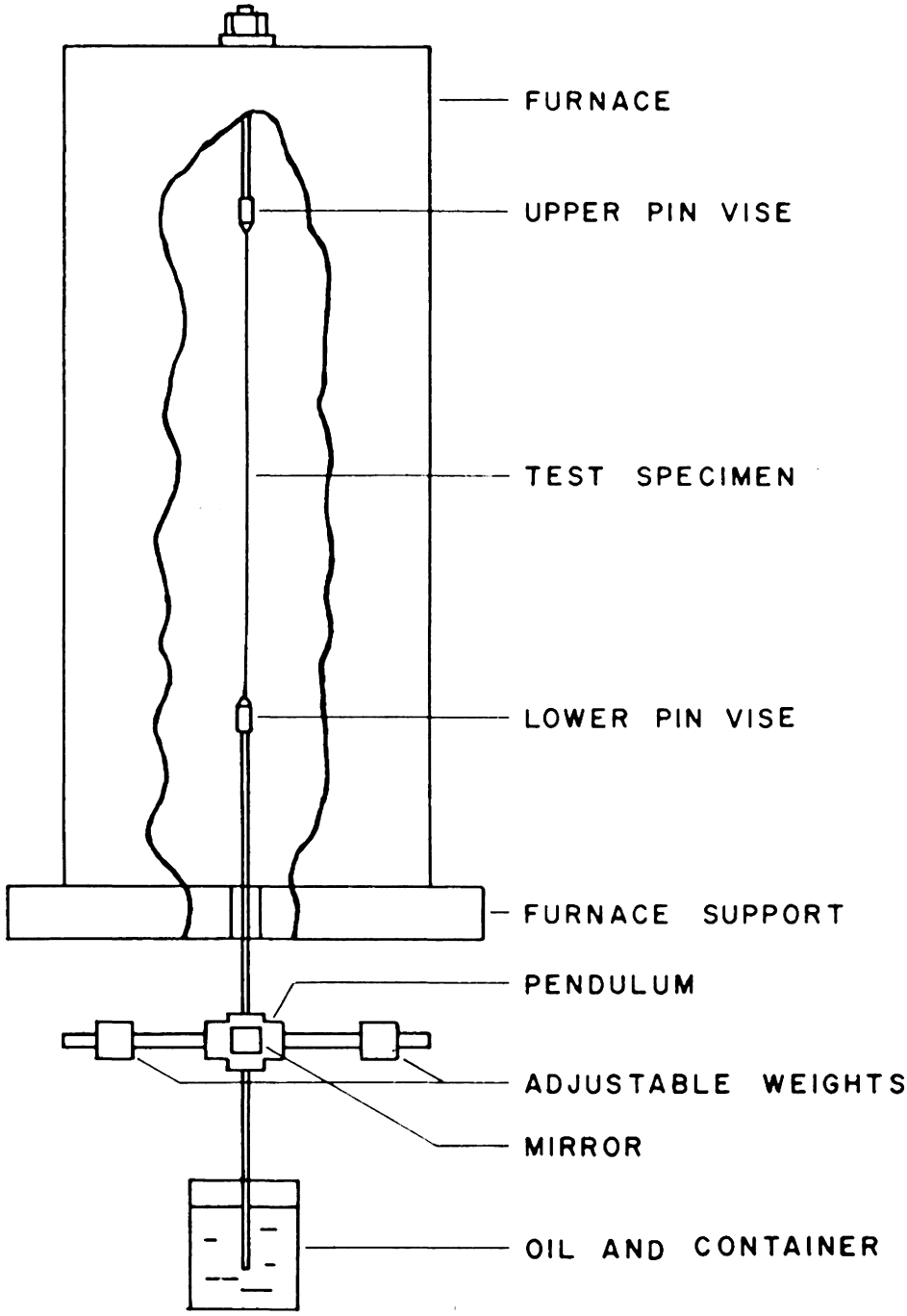

FIGURE 3. TORSIONAL PENDULUM USED IN THE INVESTIGATION.

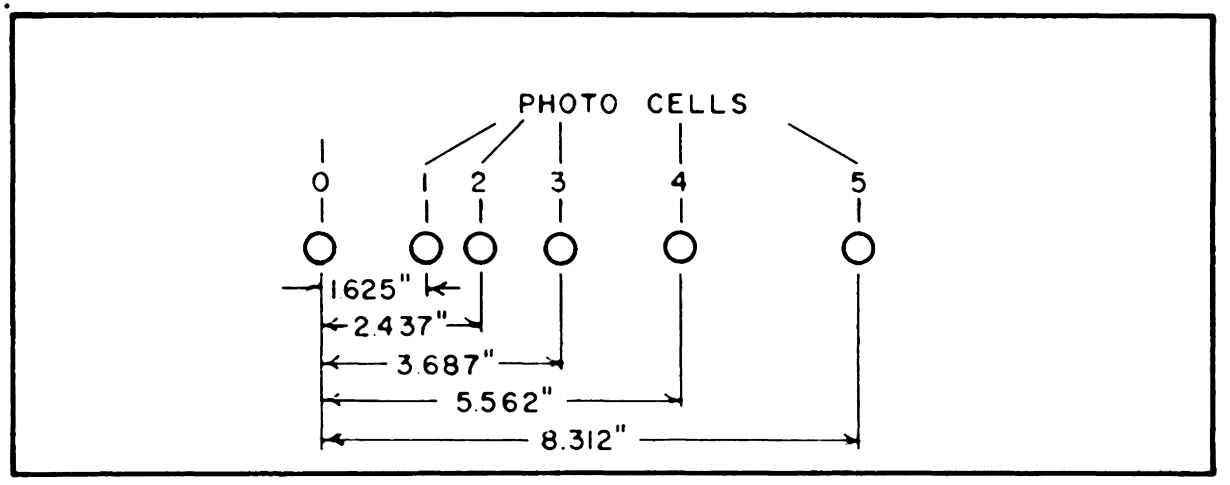

FIGURE 4. ARRANGEMENT OF PHOTOELECTRIC CELLS FOR MEASUREMENT OF LOGARITHMIC DECREMENT.

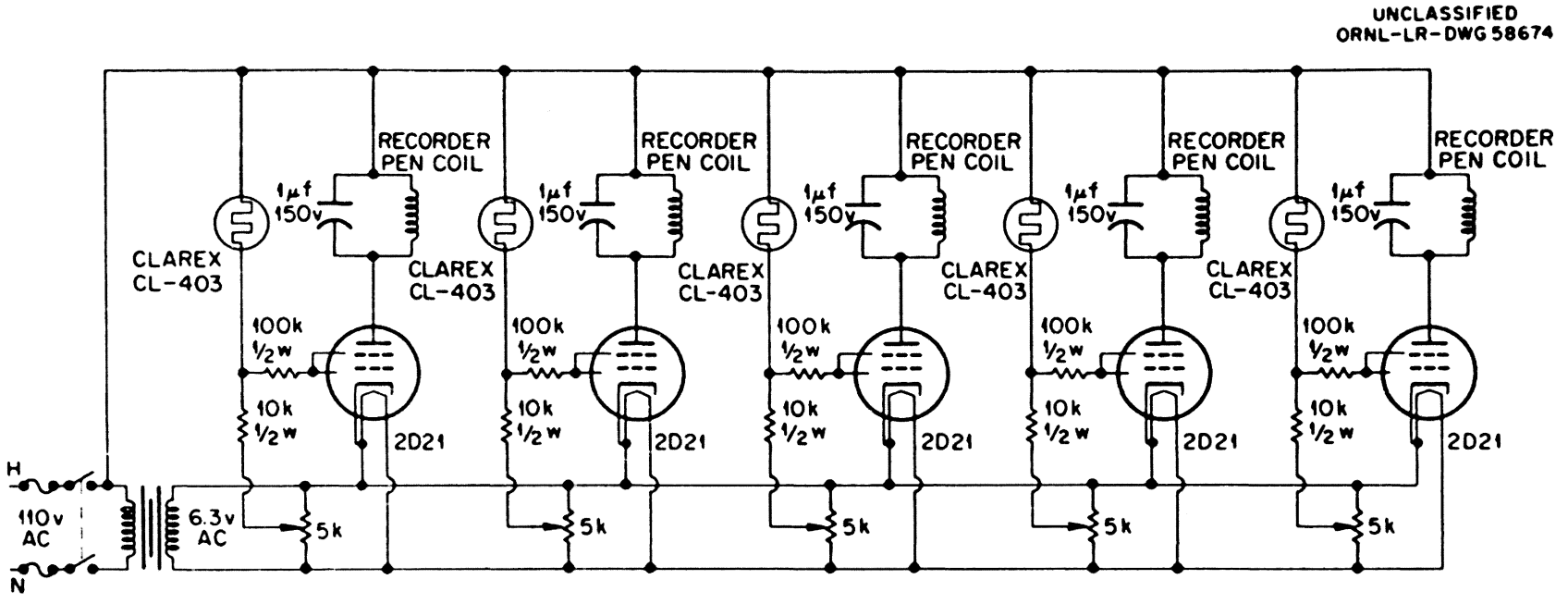

FIGURE 5. Multichannel Switching Circuit for Photoconductive Cells.

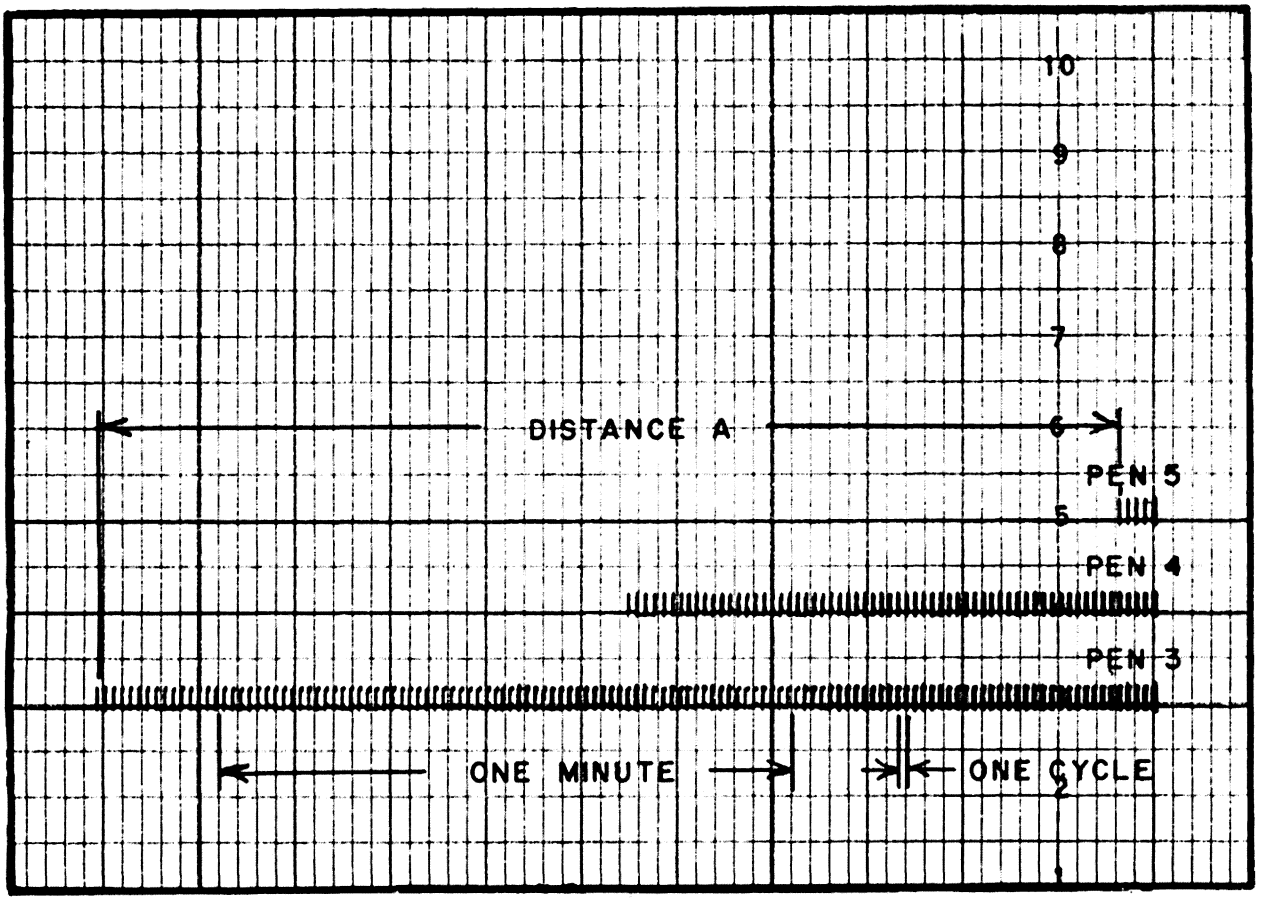

FIGURE 6. STRIP CHART RECORDING OF MEASUREMENT OF LOGARITHMIC DECAY.

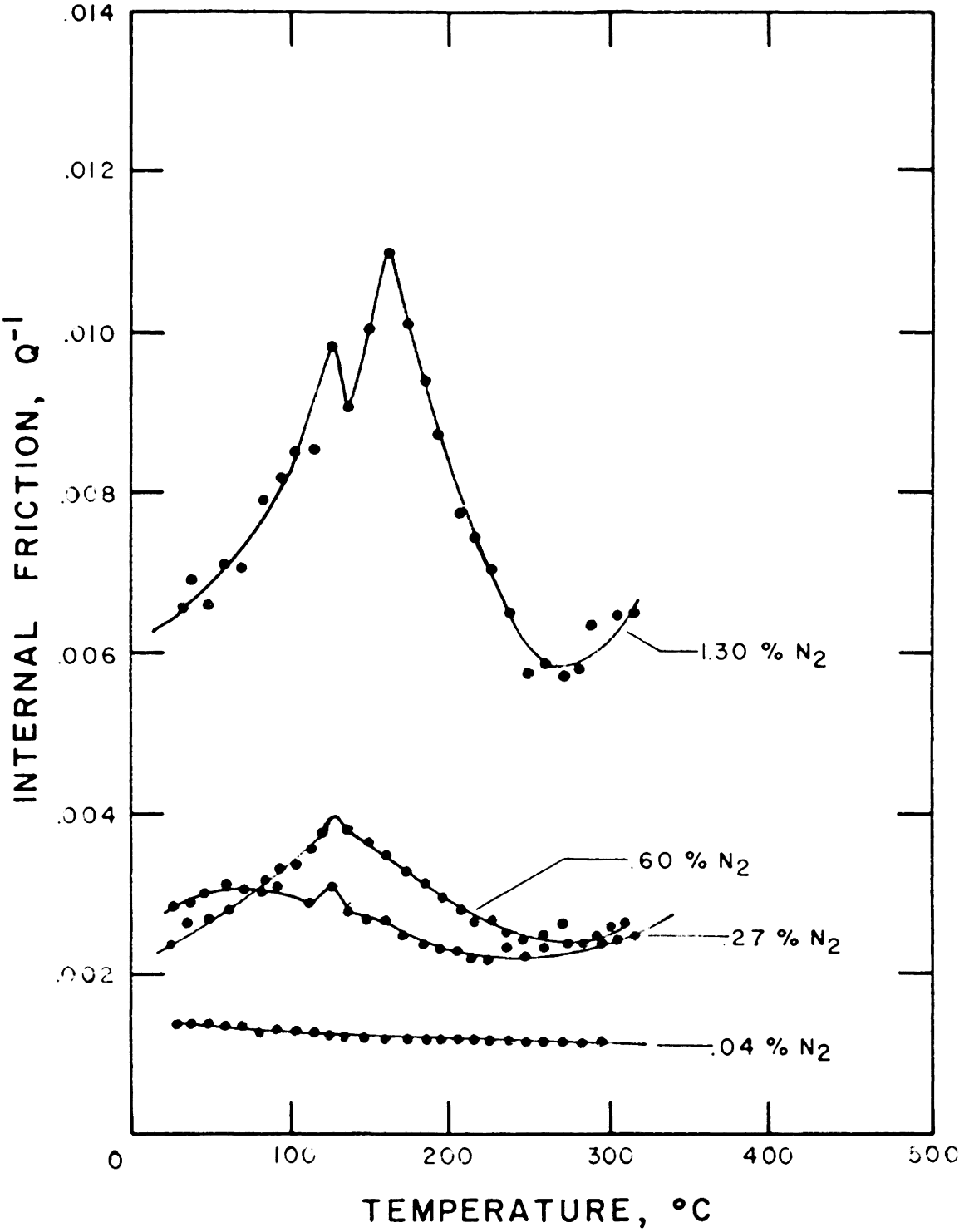

FIGURE 7. INTERNAL FRICTION SPECTRA OF TYPE 304L STAINLESS STEEL CONTAINING 0.04, 0.27, 0.60, AND 1.30 PER CENT NITROGEN RUN AT A FREQUENCY OF 1.1 CYCLES PER SECOND.

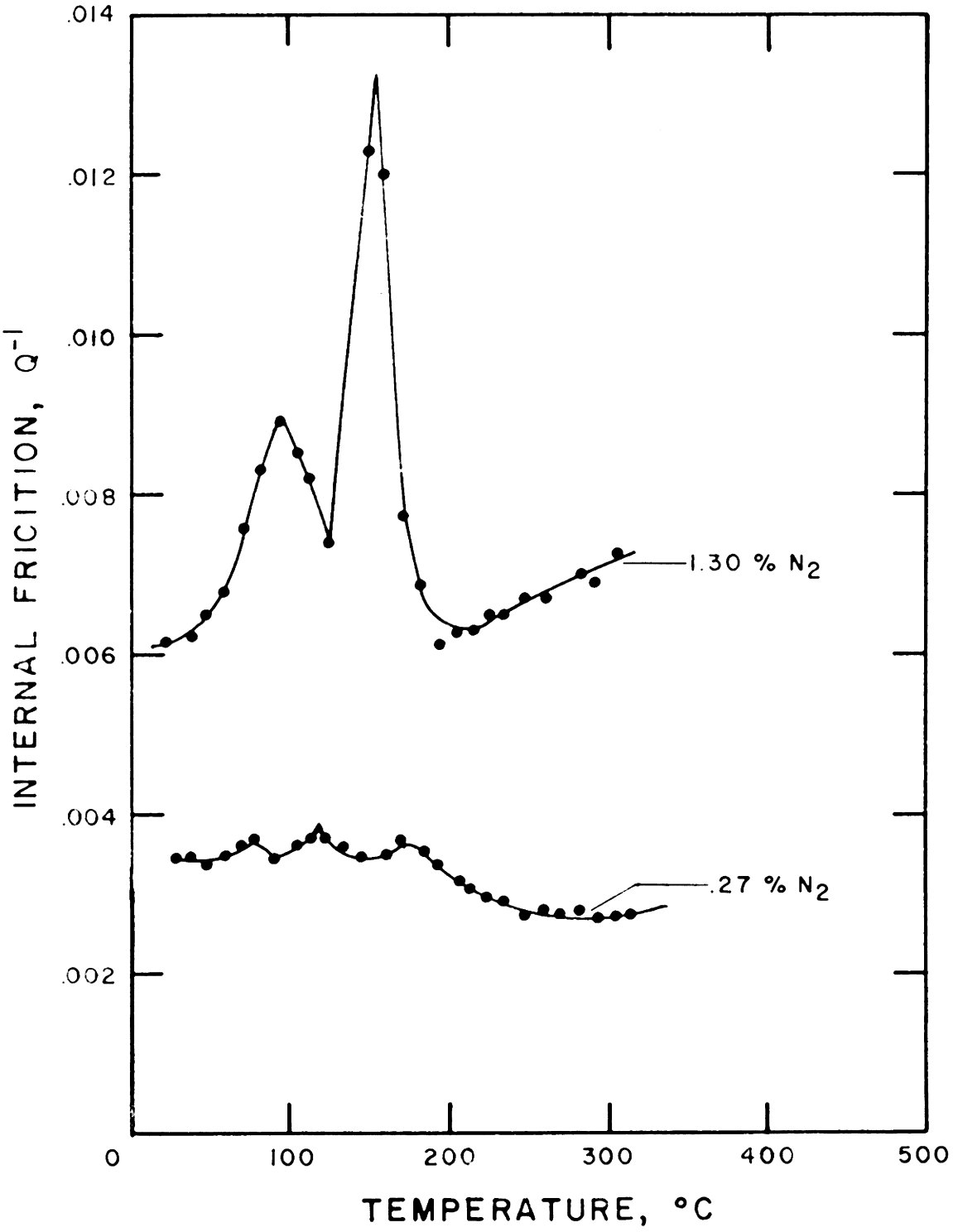

FIGURE 8. INTERNAL FRICTION SPECTRA OF TYPE 304L STAINLESS STEEL CONTAINING 0.27 AND 1.30 PER CENT NITROGEN RUN AT A FREQUENCY OF 0.48 CYCLES PER SECOND.

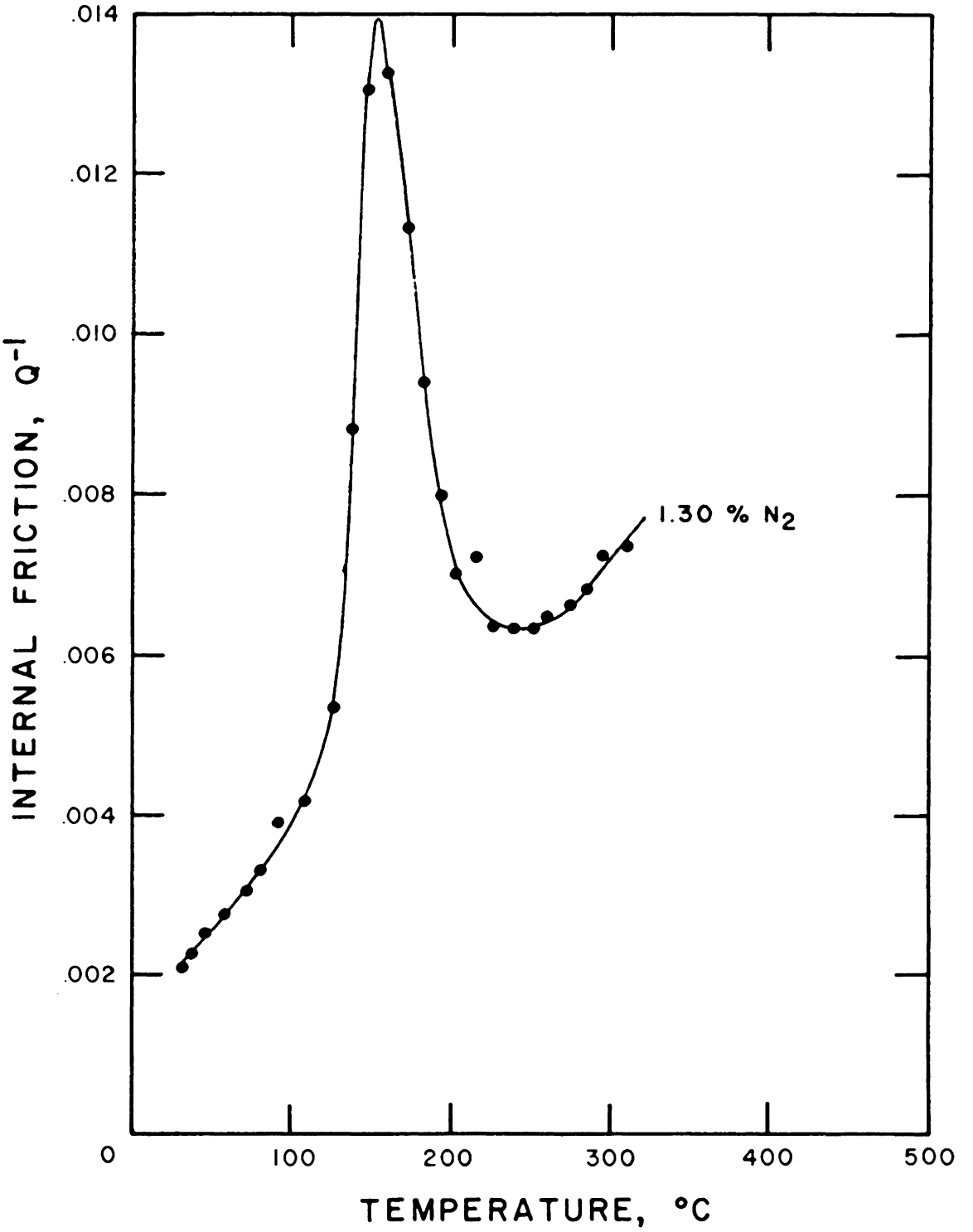

FIGURE 9. INTERNAL FRICTION SPECTRUM OF TYPE 304L STAINLESS STEEL CONTAINING 1.30 PER CENT NITROGEN RUN AT A FREQUENCY OF 1.1 CYCLES PER SECOND AND RERUN AGAIN AT 1.1 CYCLES PER SECOND.

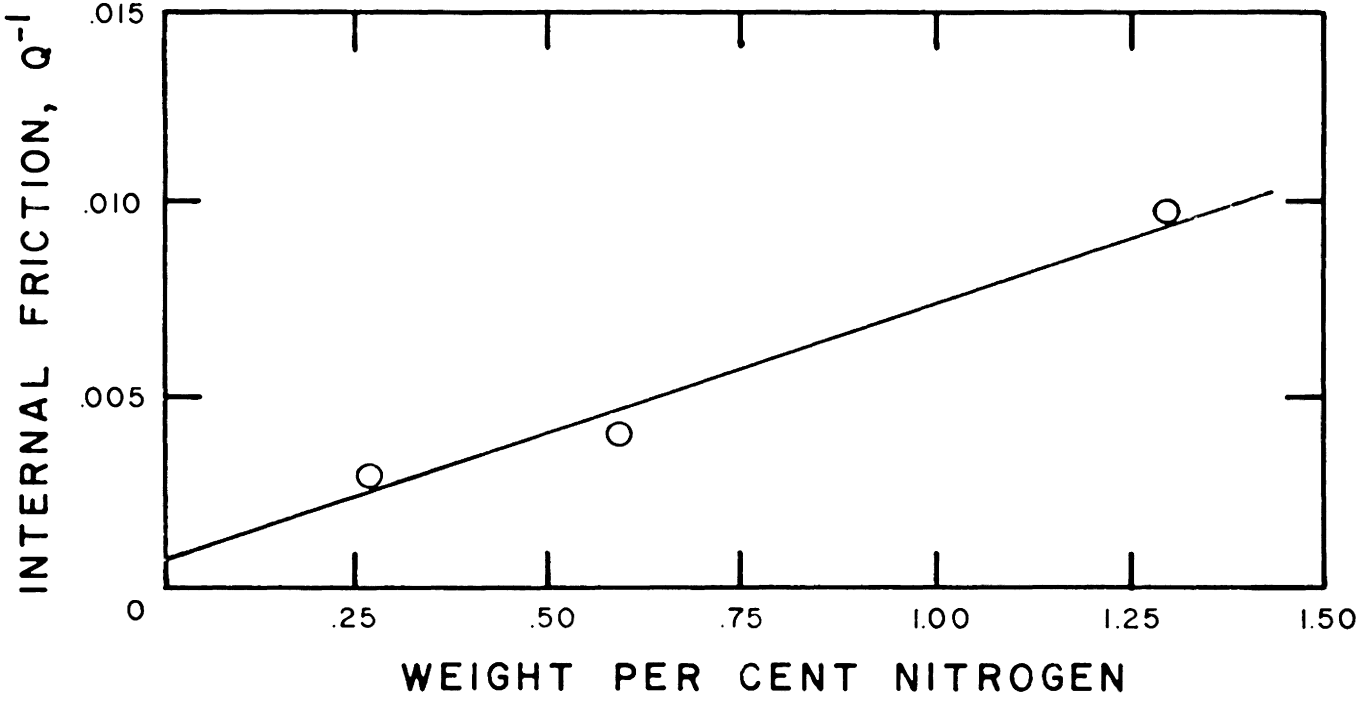

FIGURE 10. INTERNAL FRICTION AMPLITUDE PLOTTED AS A FUNCTION OF WEIGHT PER CENT NITROGEN IN TYPE 304L STAINLESS STEEL.

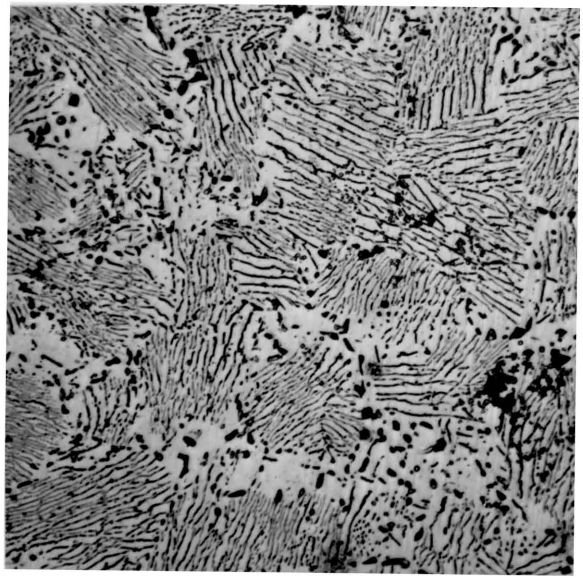

FIGURE 11. MICROSTRUCTURE OF TYPE 304L STAINLESS STEEL CONTAINING 1.30 PER CENT NITROGEN BEFORE TESTING IN PENDULUM. ETCHANT: ELECTROLYTIC ETCH, OXALIC ACID. 500 X.

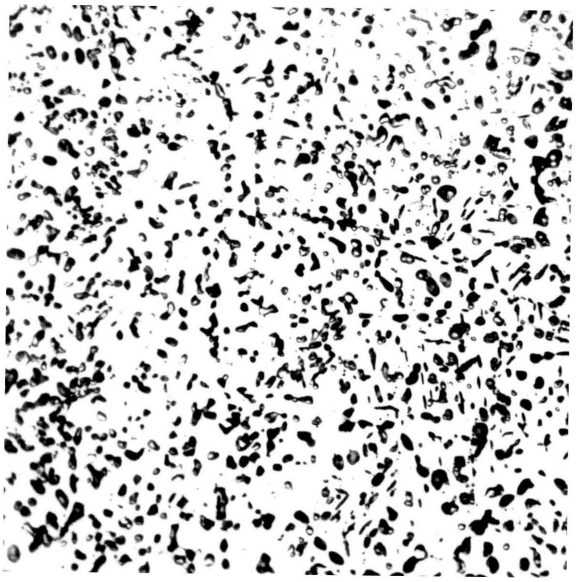

FIGURE 12. MICROSTRUCTURE OF TYPE 304L STAINLESS STEEL CONTAINING 1.30 PER CENT NITROGEN AFTER TESTING IN PENDULUM. ETCHANT: ELECTROLYTIC ETCH, OXALIC ACID. 500 X.

APPENDICES

The calculations of the logarithmic decrement values and the activation energy values were performed on an IBM 1620 Data Processing System. A modified IBM Fortran language called Forgo was used to write the instructional programs for the computer. All instructions and input data were introduced to the computer with card operations.

Appendix A. The program to calculate the logarithmic decrement values is listed below.

```
    1 Read 10, T, A, F
   10 FORMAT (3F8.5)
      S=3
      X=3.687
      X1=8.312
      D=((LOGF(X1)-LOGF(X))*S)/(3.1416*A*F)
      C=(5./9.)*(T-32.)
      PUNCH 20,C,D
   20 FORMAT (15X,3HAT ,F6.2,21H DEGREES C,D EQUALS ,F8.6)
      GO TO 1
      STOP
      END
```

Three pieces of input data are given to the computer for each value to be calculated. These are T, temperature in degrees Farenheit; A, distance on strip chart recorder shown in Figure 6; and F, the frequency of the pendulum in cycles per minute. The program contains

three constants which are common to every calculation. These are S, the speed of the recorder chart in inches per minute; X, the distance from zero to photoelectric cell three on the logarithmic scale of the screen (Figure 6); and X1, the distance from zero to photoelectric cell five on the logarithmic scale of the screen. The logarithmic decrement is found using Equation 2 where X1 is the initial amplitude and X is the amplitude after N oscillations. The program also converts degrees Farenheit into degrees centigrade. The output for each measurement is given as

AT 293.33 DEGREES C, D EQUALS .002351

AT 303.33 DEGREES C, D EQUALS .002422

AT 315.56 DEGREES C, D EQUALS .002478

where D equals the logarithmic decrement. The computer can perform the calculations for an entire spectrum in about 90 seconds.

Appendix B. The program to calculate the activation energies is listed below.

```
    1 READ 10,T1,T2,F1,F2
   10 FORMAT (8X,4F8.5)
      R=1.986
      DEL H= R*((LOGF(F2/F1))/((1./T1)-(1./T2)))
      PUNCH 20, DEL H
   20 FORMAT (10X,14DELTA H EQUALS, F8.1,18H CALORIES PER MOLE)
      GO TO 1
      STOP
      END
```

Four pieces of input data are given to the computer for each value to be calculated. These are T1, T2, F1, and F2. T1 is the temperature in degrees Kelvin at which the internal friction peak is located for a frequency of F1. T2 is the temperature in degrees Kelvin at which the internal friction peak is located for a frequency of F2. The program contains one constant R, the gas constant in calories per degree Kelvin per mole, which is common to every calculation. The activation energy is calculated by using these five values in Equation 8. The answer output for each calculation is given as

DELTA H EQUALS 34,203.3 CALORIES PER MOLE

where DELTA H is the activation energy. The computer can perform this calculation in less than one second.

ABSTRACT

An investigation was conducted to determine the effect of nitrogen concentration between 0.27 per cent and 1.30 per cent on the internal friction peaks in 304L stainless steel. The amplitude of the internal friction peak associated with the stress-induced diffusion of interstitial nitrogen increased as a linear function of the nitrogen content. The activation energy of diffusion was found to decrease with an increase in nitrogen content.

The presence of another internal friction peak was observed in the spectrum of the sample containing 1.30 per cent nitrogen. A metallographic investigation and a change in the magnetic properties of the specimens after testing along with the disappearance of the internal friction peak caused by nitrogen diffusion when the specimen was rerun indicated that the second peak probably resulted form a chromium-nitrogen interaction.