MAGNETOSTRICTION OF MAGNETITE

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Master of Science

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Dean

Committee Chairman

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Date

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ACKNOWLEDGEMENT

The author expresses his gratitude to the Office of Naval Research, for financial support during this investigation, and to Dr. L. R. Bickford, Jr., for his guidance and encouragement.

Appreciation is also due to various members of this laboratory, especially J. Pappis and M. Nerenstone.
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ABSTRACT

The magnetostriction of Fe$_3$O$_4$ was measured, using the strain gauge technique. Two methods were used to measure the magnetostrictive behavior of single crystals and polycrystalline samples as a function of orientation and temperature.

Values for the principal magnetostriction constants of the single crystals were obtained from these data, which were sufficiently complete to remove the necessity of assuming any particular initial domain distribution.

Strain gauges were calibrated at low temperatures by comparison of measurements of the thermal expansion of brass obtained both by strain gauges and by standard dilatometric techniques.

These measurements show that magnetostriction does not play an important part in causing a peak in initial permeability in magnetite near its low-temperature transition. Another conclusion is that magnetostriction measurements can be used for this type of material as a tool to detect changes in the direction of easy magnetization.

Results are compared with those of other workers.
PURPOSE

This investigation was an integral part of a project supported in part by the U.S. Office of Naval Research to study the low-temperature transition in Fe$_3$O$_4$ and related ferrites. The strain gauge technique had been first applied to magnetostriction measurements by Goldman$^1$. It had been adapted on this project to determine distortions occurring at the transition$^2,3$. This work and other results of the project indicated that it would be highly informative to obtain detailed information about the magnetostriction of Fe$_3$O$_4$ in the temperature region above the transition.

The purpose of this investigation was to measure the magnetostriction of magnetite in the temperature range between room temperature and liquid nitrogen temperature. The special goal was to determine its behavior in the temperature region (−145°C.) where an anomalous peak in initial permeability occurs in magnetite$^4$. This peak has been associated with a change in the direction of the spontaneous magnetization,$^5$ which changes from [111] (above) to [100] (below), while the crystal anisotropy passes through zero.

Both crystal anisotropy and magnetostriction play an important part in determining the initial permeability, which should be high if the magnetostriction is very small, especially along the direction of easy magnetization$^6$. From this, the magnetostriction might be expected to vanish for either the [111] or the [100] direction at this temperature.

During the latter stages of this investigation information was obtained in other phases of the project that small additions of cobalt ferrite to polycrystalline Fe$_3$O$_4$ raise the temperature of the
permeability peak by a great amount. Several of these compositions were investigated to determine their behavior with respect to magnetostriction at the temperatures of their permeability peaks.
PROCEDURE

All magnetostrictive measurements in this investigation were made with electrical resistance strain gauges. A strain gauge is a plane grid of fine resistance wire formed in a saw-tooth pattern so that the angles between adjacent legs are very small, and most of the wire is oriented in one particular direction. This grid is bonded in a sandwich of paper, which is affixed to the specimen.

The resistance wire has the property that when it is strained, either in tension or in compression, its electrical resistance changes by an amount which is proportional to and of the same order of magnitude as the strain. This change is the basis of measurement.

In use, one gauge was cemented to the sample (in this case with ordinary Duco cement), and another was cemented to a piece of brass, which is unaffected by magnetic fields. The two gauges form two legs of an A.C. bridge, which measures the difference in their resistance. The bridge is adjusted to a balance, which is disturbed when the sample is strained. The amount of unbalance, which is measured by a recording galvanometer, is proportional to the amount of strain. The proportionality constant can easily be calculated from the known characteristics of the gauges.

Two strain gauge bridges were used in this investigation: one, a Sanborn instrument containing both amplifier and recorder; the other, a Baldwin portable bridge, used in conjunction with separate amplifier and recorder. The former has the advantage of a built-in calibration device and much faster response, while the latter is somewhat more sensitive. The sensitivity of the Sanborn instrument is about $3.5 \times 10^{-6}/\text{mm.}$,
while that of the Baldwin instrument and recorder is about $1.3 \times 10^{-6}$/mm. This difference is partially offset by the fact that the line width of the Sanborn recorder is much less than that of the Esterline Angus recorder used with the Baldwin instrument.

The gauges used in this investigation were Baldwin SR4 type A-19 gauges. These gauges have an Advance wire grid one-sixteenth inch square. At room temperature these gauges have a gauge factor of about 1.65. The gauge factor, $G$, is defined such that $\frac{\Delta R}{R} = G \frac{\Delta \rho}{\rho}$, where $\frac{\Delta \rho}{\rho}$ is the strain of the gauge (and therefore of the sample) and $R$ is its resistance.

This gauge factor, while it is constant around room temperature, increases greatly at low temperatures. Thus, it was found necessary to make a calibration of the gauges at these low temperatures.
CALIBRATION OF STRAIN GAUGES

The calibration of strain gauges consisted of two parts: first, measurement of the thermal expansion of a brass bar by the use of an ordinary dilatometer, and second, the same measurement accomplished by the use of strain gauges. By means of the data obtained from these two methods, it was a simple matter to calculate the G factor of the gauges, referred to above.

In a previous calibration, these two measurements had been made independently, but it was felt that a simultaneous measurement of both would produce results more easily correlated. A diagram of the experimental set-up is shown in fig. 1.

A brass bar 5.787 in. long was recessed at its center to receive a small slab of fused silica, to which a strain gauge had been cemented. A similar gauge was cemented to a face perpendicular to the slot, allowing only enough room between them to drill a small hole for the measuring thermocouple. The silica slab was taped into the slot with gauge innermost, and the assembly covered by several layers of paper to cut down any possible convection currents.

In addition, four holes were drilled on another face of the bar: one near each end, and two close together near the midpoint. All four holes were used to contain the junctions of a "double differential" thermocouple. This couple consisted of two differential couples connected opposed, and measured the quantity \((T_{\text{top}} - T_{\text{center}}) - (T_{\text{center}} - T_{\text{bottom}})\). This was, to a first approximation, proportional to the difference between the temperature measured at the center of the bar and its true mean temperature. From this, the temperature of the measuring
thermocouple was corrected. In addition, a normal differential thermocouple with its terminals in the end holes was used to check the temperature distribution along the bar.

Since only a low degree of accuracy was desired from the differential thermocouples (maximum temperature correction was 1.5°C.), their voltages were measured directly by two reflecting box-type galvanometers of known sensitivity.

Two heaters were used to aid in the control of the temperature distribution. One was wound evenly over the length of the sample tube, and the other was wound more closely at the bottom. Since the only outside source of heat was from the top of the tube, it was necessary to heat the bottom more strongly to obtain an even temperature distribution. The windings were wired so that they could be regulated either separately or together, and a voltage-splitting control was provided which would increase one voltage and decrease the other simultaneously. The heaters were manipulated as needed by reference to the differential thermocouples.

The fused silica sample tube itself, which was enclosed in a Pyrex tube, held the dial gauge of the dilatometer.

Calibration runs were made with this equipment using both the Sanborn and the Baldwin equipment.

All results were compared, and the mean curve taken for the calibration. (Fig. 2) Agreement between the curves obtained was only fair, and all showed a much greater increase in gauge factor at low temperatures than that reported elsewhere. However, it was felt that this calibration was a very good indication of the actual state of affairs, especially since the results agreed quite well with those of the prior calibration referred to above. Hence, the mean curve was used in correction of all magnetostriction measurements.
PREPARATION OF SAMPLES

The samples used in the investigation were oblate ellipsoids*ca. 1 mm. thick and ca. 8 mm. in diameter. This shape was chosen to make the field distribution inside the sample as uniform as possible. The single crystal samples were made from (110) plane slices, this plane being chosen because it contains all the principal cubic crystallographic directions. Disks were cut from the slices with a circular brass tool, using diamond dust as the abrasive. The disks were mounted on small fiber wafers (with glycol phthalate), ground to the proper thickness, and the edges rounded to give an elliptical cross-section. The polycrystalline samples were prepared in a similar manner. The material was originally fabricated in the form of thin circular disks, from which the smaller disks were cut.

The surface of the sample was cleaned with acetone prior to gauge application. A generous blob of Duco cement was applied to the surface of the sample and stirred around to insure good wetting. The gauge was then quickly set into the cement, and the excess cement squeezed out. This was done under a nine-power binocular microscope, and was continued until the gauge adhered uniformly. At this time the sample was put between several layers of cloth, and a 100 gram weight applied. The sample was left under the weight for at least three days before it was used. (fig. 3) A sheet metal jig was used for orientation of gauges on the single crystal samples. On this jig were ruled three sets of lines, two of them perpendicular and the other at angles to the

* except for the synthetic crystal, which was a circular disk.
first of 35° and 55° respectively. These represented the \( [100] \), \( [110] \), and \( [111] \) directions in the (110) crystal plane. The \( [100] \) direction was marked on the specimens prior to cutting them, by reference to cleavage. This direction was lined up with the \( [100] \) lines on the jig, under magnification, and taped into a circular hole in the jig. The gauge was then cemented on from the other side of the jig in the desired direction (also under magnification). The probable error in setting these gauges was probably not more than three to five degrees.
EXPERIMENTAL SET-UP

A photograph of the experimental set-up during this investigation is shown in fig. 4.

The sample under examination was placed between the poles of an electromagnet, which was capable of field-strengths up to about 7500 Oersteds. (fig. 5) The sample tube (of glass) was held between the poles by a clamping device mounted in a revolving stage, so that the orientation of the sample with respect to the field could be changed at will. For the sake of convenience this rig was used whether or not a magnetic field was required.

Within the sample tube, which was closed, by a one-hole rubber stopper, was a fiber tube which supported the sample holder assembly. This tube served also as an exit for the leads from the holder.

The holder itself (fig. 6, 7) was a C-shaped piece of brass, into which the sample was slid. The holder was provided with two threaded holes, so that the plane of the sample could be mounted either parallel or perpendicular to the field. A phosphor bronze spring was provided to hold the sample securely against the bottom of the holder, in which the thermocouple hole was drilled. This assured reasonably good thermal contact between the sample and the thermocouple. The compensating strain gauge was cemented on the bottom of the sample holder, also very close to the thermocouple. It is important that the compensating gauge be at very nearly the same temperature as that of the measuring gauge, because any change in temperature of one with respect to the other shows up as an anomalous drift in the strain gauge record, making the actual behavior of the sample uncertain. Of course there is a drift with change in
temperature of the sample holder as a whole, due to the different rates of expansion of the brass holder and the sample, but this is predictable from the temperature measurement, and can be allowed for.

The sample holder was mounted on a fiber post which, in turn, was held to the fiber tube by a brass ring. In an earlier model, which was used in some of the work, this post was of brass. However the brass, being a good conductor of heat, made the temperature of the sample holder too sensitive to outside conditions, and thus difficult to control. However, the brass post had the advantage of carrying away more efficiently the heat produced by the strain gauges, and thus with it lower temperatures were attainable.

The sample holder was surrounded by a removable brass shell, whose purpose was to reduce the possibility of convection currents and to keep the surroundings of the sample holder at a more even temperature. Around this shield was wound a heater of fine nichrome wire, doubled so as to make it non-inductive. Its resistance was about 70 ohms, and its maximum power dissipation at working temperature was 3 watts.

Cooling was accomplished by surrounding the sample tube with liquid nitrogen contained in a dewar flask. By manipulation of the height of the flask and the power dissipated by the heater, the sample temperature could be held constant within 2 or 3° at any temperature between 25° and -190°.

The strain gauges were connected by a group of four parallel leads to the measuring bridge. These leads were made as heavy as possible (#18 wire inside the sample tube and #10 outside) so as to avoid fluctuations in their resistance and loss of sensitivity. They were all
the same length and parallel so as to cut down on extraneous pickup.

In use, one of the leads was common to each gauge, and each of the others was connected to the free side of one of the gages (the compensating gauge and the two measuring gauges).
MEASUREMENTS

The measurements made in this investigation were of two types. In the first type, the sample was held stationary between the poles of the magnet and the field was varied. At first the magnet current was varied manually. Later, however, an electronic power supply was designed and built for this purpose by Dr. John Levinson, as another phase of the research project. This device automatically varies the current linearly from 3 A. to -3A. with a triangular wave form, and with periods of 15, 30, and 60 seconds. With this it was possible to record data continuously and automatically, obtaining with each quarter cycle a complete record of the magnetostriction in a continuously varying field, from zero field to a field of about 2000 Oersteds. Data of this type were obtained as a function of temperature, crystallographic direction, and angle between the direction being measured and the field, for the following samples:

1. A natural crystal of magnetite.
2. A synthetic crystal of magnetite.*
3. A synthetic polycrystalline sample of magnetite.**
4. Synthetic polycrystalline samples of Fe₃O₄ with additions of 0.25, 0.5, 1.0, 2.0, and 4.0 percent CoO.**

The second method of obtaining data consisted of holding the sample in a constant field strong enough to saturate it (about 3000 Oersteds) and plotting curves obtained as the orientation of the crystal was changed.

* made by Smiltens and lent by Dr. von Hippel of M.I.T.
** made by J. Pappis.
with respect to the direction of the field. This data was again taken as a function of the crystallographic direction under study, the orientation of this direction with respect to the field, and the temperature. These data were obtained for several temperatures between room temperature and -160°C., for both the natural and the synthetic crystal.

Power for the constant field measurements was obtained from a 48 volt battery system, and from a D.C. generator. The R.F. radiation of the generator brushes was cut down by connecting a condenser between them and ground.
RESULTS

Figure 8 shows magnetostriction curves obtained from the synthetic crystal for the \([100]\) and \([111]\) directions, both longitudinal and transverse. Longitudinal magnetostriction, or \(\lambda_{\text{long.}}\), is the strain produced along a chosen direction when the crystal is magnetized along that direction, while transverse magnetostriction, or \(\lambda_{\text{trans.}}\), is the strain along \(\phi\) direction when the sample is magnetized in a direction perpendicular to it. In each case the field is plotted horizontally and the magnetostriction vertically, temperatures being indicated on the scale at the left. Results at room temperature are shown at the top of the diagram. The structure of all the curves changes at the temperature of the permeability peak. The most striking change occurs on the curve for longitudinal magnetostriction along the \([110]\) direction. Here, the magnetostriction apparently changes from negative above the peak, through zero, to positive below. This would seem to indicate that the magnetostriction does play an important part in the permeability peak.

It will be recalled that, as mentioned above, permeability should be large when the magnetostriction is very small, especially along the direction of easy magnetization (the direction along which the magnetization lies when there is no external field). However, this effect was not noticed in our natural crystal (although Domenicali\(^9\) found such a change in sign for a similar crystal at about \(-88^\circ\text{C}\)), in which there is also a permeability peak at about the same temperature. Also, there were spontaneous distortions in the synthetic crystal at this temperature, even in zero field. Figure 9 shows these distortions, which were found by Bickford\(^2\). Another argument against the influence of magnetostriction on the permeability peak in that the magnetostriction calculated from the
Therefore, one would expect that above the peak, where $[111]$ is the easy direction, its effect would be felt first, and since the $[111]$ magnetostriction and the total polycrystalline magnetostriction are of different sign only in the transverse case, there should be a low-field reversal in this case but none in the longitudinal case. Similarly, below the peak temperature, where $[100]$ is the easy direction, and since the $[100]$ magnetostriction and the total polycrystalline magnetostriction are of different sign only in the longitudinal case, there should be a low-field reversal in this orientation. These suppositions are in fact true.*

The reversal argument was used to explain the magnetostrictive behavior of polycrystalline samples to which small cobalt additions had been made. The initial permeabilities of the same samples were measured independently$^{10}$ as a different phase of the ferrite research project.

* From the above, it seems virtually certain that these reversals are closely connected with the change in the direction of easy magnetization. This conclusion is strengthened by the fact that at -30°C., where the sign of polycrystalline transverse magnetostriction changes from positive (below) to negative (above), the reversals in these curves disappear. This would be expected because, under these conditions, the magnetostrictive behavior of the easy direction ($[111]$) is of the same sign as that of the total polycrystalline magnetostriction. In addition, it can be seen that the above arguments are consistent with the conclusion that the sign of $A_{\text{long.}}$ does not change at the temperature of the permeability peak.

It is to be noted that the change of sign of the polycrystalline transverse magnetostriction is due to a change in the relative magnitudes of the magnetostriction for the individual principal crystallographic directions, and does not mean that the magnetostriction of any one of these directions changes sign.
It was found that the temperature of the permeability peak varied linearly with the cobalt oxide content. Magnetostriction curves showed a region in which reversals appeared. For additions up to one percent cobalt oxide the temperature of reversal appearance in magnetostriction measurements agreed with the temperature of the permeability peak to within the error of temperature measurement. Figure 11 shows a curve of the temperature of the peak as a function of the percentage cobalt oxide addition, along with points at which the reversals occur in the strain gauge measurements. The close agreement between the curves is good evidence that the permeability peak is associated with a change in the direction of easy magnetization. For additions greater than one percent it can be seen that there are discrepancies, which are logically attributable to the effect of the cobalt additions on the magnetostriction itself. However it is interesting to note that up to this point the temperature of the permeability peak is almost exactly proportional to the amount of cobalt oxide added.

One fact which is illustrated very well by the measurements of the synthetic crystal is that it is almost impossible to measure magnetostriction directly, due to the uncertainty as to the initial domain distribution (i.e., the distribution of domains in the demagnetized state) within the crystal. This difficulty can be avoided if, instead of measuring the strain as the sample is magnetized, a measurement is made of the change in strain in a saturated crystal as the orientation of the direction under study is changed with respect to the direction of the magnetic field.

Bozorth and Hamming have developed a simplified procedure for the determination of the five constants of the Becker and Döring
magnetostriiction equation\textsuperscript{13}, from which the individual $\lambda$'s can be calculated. The results of the constant field measurements of the $[111]$ and $[100]$ directions were used in calculating these five constants at various temperatures, for both the synthetic and the natural crystals. For this calculation data are obtained in the form of values of $(\lambda \theta - \lambda \theta = 0)$, where $\theta$ is the angle between the $[100]$ direction and the field, and $(\lambda \theta - \lambda \theta = 0)$ is the difference in strain between this orientation and the orientation $\theta = 0$. Theta is varied in nine steps from 10 to 90\textdegree. Multiplication of the resulting values by the terms of an appropriate matrix, followed by addition of terms, automatically give the values of the constants already corrected to a least square fit. Values of these five constants at various temperatures are given in tables I-II for both crystals.

Each of these five constants involves a term for each of the nine orientations, some as positive contributions and some as negative ones. Thus an individual constant is obtained by addition and subtraction of numbers some of which are frequently three times as large as the constant itself (e.g., in the case of the major constants $h_1$ and $h_2$). In the case of the minor constants the numbers involved may be tens and even hundreds of times as large. Because of this method of calculation, these constants are sensitive not only to the sizes of the recorded curves, but also to their shape. This accounts in a large degree for the variations in the constants at different temperatures. It is thought that more accurate measurements would permit the calculation of the minor constants with much more accuracy.

Values for the principal $\lambda$'s of both crystals were calculated from these $h$'s (Figs. 12-15). In spite of the variation of the individual constants, it can be seen from the curves that these values lie fairly close together.
As a test of the validity of this calculation values were calculated for the difference between longitudinal and transverse magnetostriction for the \([110]\) direction for both the natural and synthetic crystals. These results are plotted on figs. 16-17, along with experimental determinations of these same values. This test was severe in that the \([110]\) data were not used in the calculation of the five constants, and yet it can be seen that the experimental points lie fairly close to the calculated curves.

As a further check, the same difference was calculated for a polycrystalline sample of Fe₃O₄, using the constants calculated from the synthetic crystal data. The results are shown on fig. 18, along with experimental points obtained from the polycrystalline sample already mentioned. These values also lie fairly close to the calculated curve. This test was even more severe, since it involved application of results calculated from one sample to another sample of a different kind.

Values calculated for the principal magnetostrictive constants of the two crystals at room temperature are shown on table III. These values are higher than those measured by Heaps\(^{14}\) from a natural crystal, probably because saturation was not reached, due to the shape of the crystals (octahedrons). However, they compare well with results calculated by Calhoun\(^{15}\) for a synthetic crystal, as shown.
CONCLUSIONS

It is concluded, on the basis of the arguments presented, that the sign of the \{100\} magnetostriction does not change at the temperature of the permeability peak, and that magnetostriction therefore does not play an important part in this peak. Rather, it is concluded that the reason for the apparent change in sign is the unique initial domain distribution in the sample used.

It is further concluded that the low field reversals found in the polycrystalline samples are directly related to the change in direction of the spontaneous magnetization in Fe$_3$O$_4$, and that magnetostriction measurements can be used as an indication of this change in compositions close to pure Fe$_3$O$_4$. 
TABLE I MAGNETOSTRICTION CONSTANTS ($\times 10^6$)
CALCULATED FROM SYNTHETIC CRYSTAL DATA

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<th>T, °C.</th>
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### TABLE II MAGNETOSTRICTION CONSTANTS (x10^6)

CALCULATED FROM NATURAL CRYSTAL DATA

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### TABLE III  COMPARISON OF CALCULATED VALUES OF MAGNETOSTRICTION

OF NATURAL AND SYNTHETIC CRYSTALS AT ROOM TEMPERATURE

WITH THOSE OF CALHOUN\textsuperscript{15} (x10\textsuperscript{6})

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<thead>
<tr>
<th>Direction</th>
<th>Natural Crystal</th>
<th>Synthetic Crystal</th>
<th>Calhoun's value (synthetic crystal)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 long</td>
<td>-13.5</td>
<td>-19.4</td>
<td>-19.7</td>
</tr>
<tr>
<td>100 trans</td>
<td>7.52</td>
<td>8.9</td>
<td>9.7</td>
</tr>
<tr>
<td>110 long</td>
<td>68.7</td>
<td>57.2</td>
<td>98.7</td>
</tr>
<tr>
<td>110 trans</td>
<td>11.5</td>
<td>9.9</td>
<td>9.7</td>
</tr>
<tr>
<td>111 long</td>
<td>83.7</td>
<td>77.7</td>
<td>86.4</td>
</tr>
<tr>
<td>111 trans</td>
<td>-46.7</td>
<td>-41.1</td>
<td>-37</td>
</tr>
<tr>
<td>Polycrystalline (long.)</td>
<td>49.0</td>
<td>40.6</td>
<td>61.9</td>
</tr>
</tbody>
</table>
BIBLIOGRAPHY


FIGURE I

CALIBRATION SET-UP

- DIAL GAUGE
- SILICA PUSHROD
- SILICA TUBE
- HEATER (ONLY ONE SHOWN)
- SILICA SLAB
  WITH STRAIN GAUGE
- STRAIN GAUGE
- T.C. HOLES
- PYREX TUBE

BRASS BAR
FIGURE 2

CALIBRATION
OF STRAIN GAUGES
FIGURE 5 - EXPERIMENTAL SET-UP

REVOLVING STAGE

DEWAR FLASK

SAMPLE HOLDER

MAGNET POLE

LIQUID N₂
FIGURE 6 - SAMPLE HOLDER

- Fiber Tube
- Shell
- Heater
- Sample
- Holder
- Spring
- T.C.
- Compensating Gauge
Figure 12—Calculated $\lambda'$s

Natural Crystal [100]

$\lambda \times 10^6$

$T$, °C

20 0 -50 -100 -150
Figure 13 - Calculated χ's

Natural crystal

[111]

Long.

Trans.

\( \chi \times 10^6 \)

Temperature, \( ^\circ \text{C} \)

\(-150\) to \(-50\)
FIGURE 14—CALCULATED $\lambda$'S

SYNTHETIC CRYSTAL

$\lambda \times 10^6$

$T_j \, ^\circ C$
FIGURE 15 - CALCULATED $\lambda$'S

SYNTHETIC CRYSTAL

$\lambda \times 10^6$

$T_2 \, ^\circ C$
FIGURE 17 - $\lambda$ LONG. - $\lambda$ TRANS.

SYNTHETIC CRYSTAL $[110]$

$\lambda \times 10^6$

$T, ^\circ C$

CALCULATED

EXPERIMENTAL
EXPERIMENTAL

CALC. FROM SYNTHETIC CRYSTAL

FIGURE 18-λ_{LONG.- λ_{TRANS.}

POLYCRYSTALLINE Fe₃O₄