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INTERFEROMETER MEASUREMENTS ON THE EXPANSION OF IRON

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ABSTRACT

The interferometer has been applied by many investigators to the measurement of thermal expansion but has failed to yield the precision that they apparently expected and sometimes have claimed. In their explanation of the resultant discrepancies, most observers have attributed them to actual differences in the physical properties of the sample; however, some have admitted failure to find a satisfactory explanation.

Several sets of data, taken on relatively pure iron by different observers and different procedures, are compared. The results show good agreement between data taken with those interferometer methods that are free from tilting of spacers and air-film errors, whereas the failure to eliminate these two errors produces data that cannot be duplicated except by chance. The interferometer data that are free from these errors also agree satisfactorily with data that have been obtained by other precision methods.

Some investigators claim to have found indications of a characteristic temperature effect in the expansivity curve of iron in the temperature range from 0° to 250°C. It is shown that when the expansion data are freed from errors of tilting and changes in air films, the indications of such effects do not appear.

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I. INTRODUCTION

Many observers have measured the expansion of relatively pure iron. Several different methods of measurement have been used, among which that employing the Fizeau [1]* type of interferometer, in the form described by Peters [2] and Merritt [3], has been considered [4] extremely accurate. However, as will be shown later, the data taken by different observers, with this particular form of interferometer, do not always agree. Often data obtained on different samples of the same material by a single observer have shown differences far in excess of the precision that should be attainable with this instrument.

Considerable stress has been placed on the presence of certain reversals in curvature which have been found in the expansivity curve of iron in the range 0° to 400° C. In some cases these effects appear to be mainly the result of inadequate precision. That is, these reversals in curvature occur at different temperatures in data obtained

*Figures in brackets indicate the literature references at the end of this paper.

on similar samples by different observers and also in data obtained by a given observer on different samples. This indicates that they are not a definite characteristic of relatively pure iron. This indication is supported by the results of Driesen [5], which show that the reasonably definite irregularities in the expansion of impure iron in this temperature range practically disappear when the carbon content is less than 0.1 percent. However, some later results obtained by the interferometric method have been interpreted as showing that an irregularity still appears in this temperature range even when the carbon content is less than 0.01 percent.

This paper gives the results of additional measurements of the expansion of high-purity iron. These measurements were made by the Fizeau interferometer method with a procedure which substantially eliminates errors caused by a tilting of the spacers and by variation in the thickness of air films between the spacers and interferometer plates at their points of contact.

II. SOME PREVIOUS INTERFEROMETRIC RESULTS ON IRON

Of those who have investigated the expansion of iron by the interferometric method, Dorsey, Austin and Pierce, Adenstedt, and Nix and MacNair have obtained results that will be considered here.

In 1907 Dorsey [6] measured the expansion of relatively pure iron with an interferometer arrangement using a vacuum furnace and the ring type of sample proposed by Reimerdes [7]. Data taken with this type of sample, in vacuum, are practically free from both air-film errors and tilting of spacers caused by differential expansion. Having eliminated both tilting and air-film errors by this method, Dorsey then considered the sources of error that the assumed precision of the measurements indicate as possibly inherent in the method. The indicated errors were found to be relatively small, but concerning the expansion data he adds this illuminating statement, "individual intervals for some specimens, however, show differences among themselves far in excess of the above-indicated errors." However, Dorsey's data are in fair agreement with data obtained by others who employed equally precise methods (see fig. 4).

In 1934 Austin and Pierce [8] published data on 10 samples of relatively pure iron. The method used was that described by Merritt and subsequently [9] found to be subject to the previously mentioned errors. The expansivities obtained for the 10 samples differed among themselves in the range 0° and 400° C. The differences from sample to sample were attributed by Austin and Pierce to small differences in impurities. However, these differences in expansivity, and the irregularities also, do not appear to the author to exceed the possible lack of precision of the measuring method employed.

In 1936 Adenstedt [10], while using practically the same interferometer arrangement that Austin and Pierce used, measured the expansivity of iron in a wide range below 0°C and observed that the width and orientation of the interference bands did not remain constant as the temperature changed. Consequently, he discarded all tests (15 out of 36) in which this effect, caused by a tilting of the samples, was observed. As a result of this experience, Adenstedt devised an ingenious arrangement for supporting his samples and

binding them together in an essentially ring-like spacer that eliminated tilting. This appears to have improved the precision considerably beyond that resulting from the rather questionable expedient of discarding the apparently less desirable tests. Unfortunately, he did not use this device in testing iron.

In 1941 Nix and MacNair [4] also used the interferometer method as employed by Austin and Pierce except that they replaced visual observation by a photographic method for recording the shift of the interference fringes. The data which they published for iron were procured by testing three samples obtained from the National Bureau of Standards. The total impurities of these samples varied from approximately 0.008 to 0.012 percent. Concerning the differences in the results on these samples, Nix and MacNair stated that the "variations from sample to sample are quite considerable in the region above room temperature where presumably the effects of extremely small amounts of impurities can materially affect the ferromagnetic change in length." As computed, their results for the expansivities resemble somewhat those obtained by Austin and Pierce. That is, these results indicate a plateau-like effect in some of the expansion curves near the rather low temperature of 140°C.

Before the measurements discussed in the following pages were made, the expansion of other samples of the particular iron studied by Nix and MacNair was also measured by others, including the present author. In none of these cases was the plateau-like effect observed but, in the range 25° to 150°C, the results of the present author were lower than those of Hidnert and Emerson as shown in figure 9 of the publication by Cleaves and Hiegel [11]. It was believed that this discrepancy in the author's data, which reached a maximum of about 3 percent of the expansivity at 25°C, was the result of small temperature gradients that developed (on heating at a rate of 1 degree C per minute) between the refraction thermometer and the rather massive specimens of iron that were used. In procuring these data the interest centered primarily in eliminating erratic errors that might cause spurious plateau-like effects rather than in carefully avoiding all small errors that might appear because of slight gradients. Such gradients develop rapidly on starting and then slowly diminish as the heating rate rises from zero to the constant value chosen. Any material discontinuity in the heating rate, during a test, will produce such changes in the thermal gradients and often gives rise to corresponding irregularities in expansion data. That is, to attain consistent results and to prevent spurious irregularities, a uniform heating rate should be maintained. Also any adjustments that change the heat conduction within the specimen chamber or that permit unsymmetric radiation from it should be avoided if possible.

III. COMPOSITION AND TYPE OF SAMPLES TESTED

In the present investigation, the expansivities of several samples from the high-purity iron that yielded the results presented by Nix and MacNair and that was previously tested at this Bureau were measured between 25° and 260°C. No significant differences from sample to sample were found. To study the possible effects of differences in the amounts of impurities in relatively pure iron, tests were also made on samples from an electrolytic iron and from an

open-hearth ingot iron. (The impurities of these irons are shown in table 1.) For this comparative study of the three irons, the tests were extended to 400°C.

TABLE 1.—*Composition of samples*

Elements	Samples			Elements	Samples		
	A	B	C		A	B	C
C.....	0.001	0.005	0.045	Cr.....		<.001	.006
Cu.....	<.002	<.001	.046	Mn.....		.002	.04
Si.....	.001	.001	.001	Ni.....		<.001	.019
Be.....	<.001			Mo.....			.002
Al.....	nil	<.001	.002	Co.....			.008
S.....	.002 ₃	<.004	.03	As.....			.012
P.....	<.0005	<.003	.003	Sn.....			.007
O ₂000 ₃	.003	.07	Total of identified impurities.	<0.008	<0.024	<0.30
N ₂000 ₂	.002	.004				
H ₂000 ₂	.000 ₁					

In some of these later tests, three individual tripod spacers were used as in the previously mentioned measurements, but they were no longer of the massive type. That is, as a possible aid in reducing thermal gradients, the weight of the spacers was reduced to one-third by cutting away much of the unnecessary portions. In the remainder of the tests, a single T-shaped spacer was used, and its weight seldom exceeded 1.5 grams, although its height usually exceeded a centimeter. As a further possible aid in reducing gradients, the shape of the specimen was designed, whenever possible, to facilitate the transfer of heat between it and the surrounding gas. These steps are advisable because the rate of heat transfer through the point contacts between plate and spacer is low.

IV. METHOD OF MEASUREMENT

Most of the following data were obtained visually by means of the usual viewing instrument. However, in some cases a new type of photographic recorder¹ was used. Sample records are reproduced in figure 1 to show the small amount of film required. The recorder was attached directly to the viewing instrument without disturbing the interferometer in the furnace, when the change from visual to photographic recording was made.

In assembling the interferometer, the weighted spacer method (see reference [9]) was used, and the reference point was located directly above the contact between this spacer and the upper plate. To stabilize the air films between this spacer and the interferometer plates, the assembly was always given a preliminary heating to 150° C and allowed to cool to room temperature before the first run for procuring data. The tests were made in air at atmospheric

¹ This type of recorder yields an accurate and continuous record of the changes in the interference fringes. The interferometer itself may be observed during the exposure without interfering in any way with the recording, and a surprisingly small amount of film is needed. One foot of 35-mm film is sufficient to record a shift of more than a hundred fringes. The recorder (designated an interferograph) produces the record (called an interferogram) in somewhat the same way that a panoramic camera operates. An image of the interferometer fringes is projected into the plane of the photographic film. A screen, located close to and in front of the film, having a slit similar to that of a focal-plane shutter, obstructs all of the image except a narrow strip. The film is caused to move perpendicular to the slit. The resultant exposure produces a record of the fringe movements relative to any point whose image falls in the exposing slit. This photographic recorder will be described in a later publication.

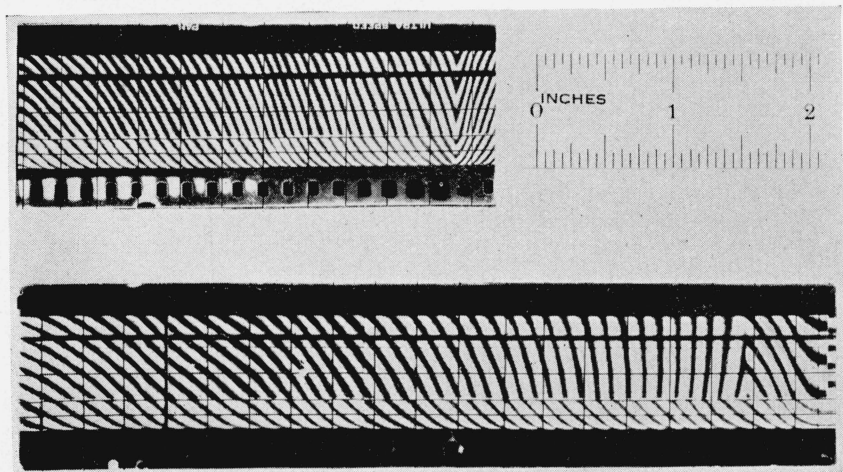


FIGURE 1.—Two photographic records (interferograms) of both temperature and expansion of iron from room temperature to 260° C.

The heating rate for the upper record was 1 degree C per minute and for the lower record, it was one-half degree per minute. The same difference in length of record, for a given temperature interval, is obtained by using the same heating rate and two different rates of film motion.

pressure and no effects resulting from oxidation were noted.² The usual heating rate was approximately 1 degree C per minute except for the second photographic record, in which the rate was one-half degree per minute. Observations were made at the passage of each fringe in the thermometer plate.

V. RESULTS

Figure 2, A, shows a plot of the expansion curves obtained on one of the samples of the high-purity iron. The data of the first run were obtained visually. Those for the second and third runs were obtained photographically without disturbing the interferometer. In order to record the fringe shifts, both at the reference point over the weighted spacer³ and at the median point (as defined in reference [9]), both points were included in the record. The tilting of the unweighted spacers is therefore made quite evident.

The plot of the observations (fig. 2, A), which were made at intervals averaging about 7 degree C, show excellent agreement when they were made at the weighted spacer. Also, there was no material difference between the visual and photographic data. As was to be expected, the data at the median point deviate considerably from those at the weighted spacer.

To make the discrepancies between these data more apparent, a type of residual plot that makes their magnification possible is shown in figure 2, B. To produce this figure, the ordinates of the straight line, $y=0.13T-1.50$, were subtracted from the corresponding ordinates of the expansivities plotted at different values of the temperature, T . As before, the agreement between the weighted spacer data appears to be excellent, but the deviation of the results at the median point from those at the weighted spacer point are shown to be very large except near room temperature, where the expansion curves were made to intersect. For comparison, that part of Nix and MacNair's results that was obtained in this range and on the same iron is also plotted in both figures here.

This departure of the curve, for the median point, from the curve for the weighted spacer point (as the temperature increases) is typical of the method. Also the author's curve for the median point is typical of the condition in which the spacers are all untilted as the heating begins, but in which at least one of the spacers develops a continually growing tilt as the temperature rises. At least a part of the median-point curve would have fallen below the weighted spacer curve had there been, at room temperature, an initial tilt of the kind that is caused by cooling the interferometer from some higher temperature at which the spacers have been brought into an untilted condition. That is, if this higher temperature had been above the temperature attained in these tests, the median-point curve would have fallen further and further below the weighted spacer curve as the temperature rose. However, if this temperature had been between the highest and lowest temperatures of the tests, this departure would have risen to a maximum and then decreased.

² Prolonged observation at a constant temperature of 400° C showed no progressive change in readings.

³ Data recorded at the median point, on the second photographic record, deviated much more than it did during the first recording, amounting to over 7 percent. This deviation was so large that the corresponding results are omitted from figure 2, as its inclusion would require too much space.

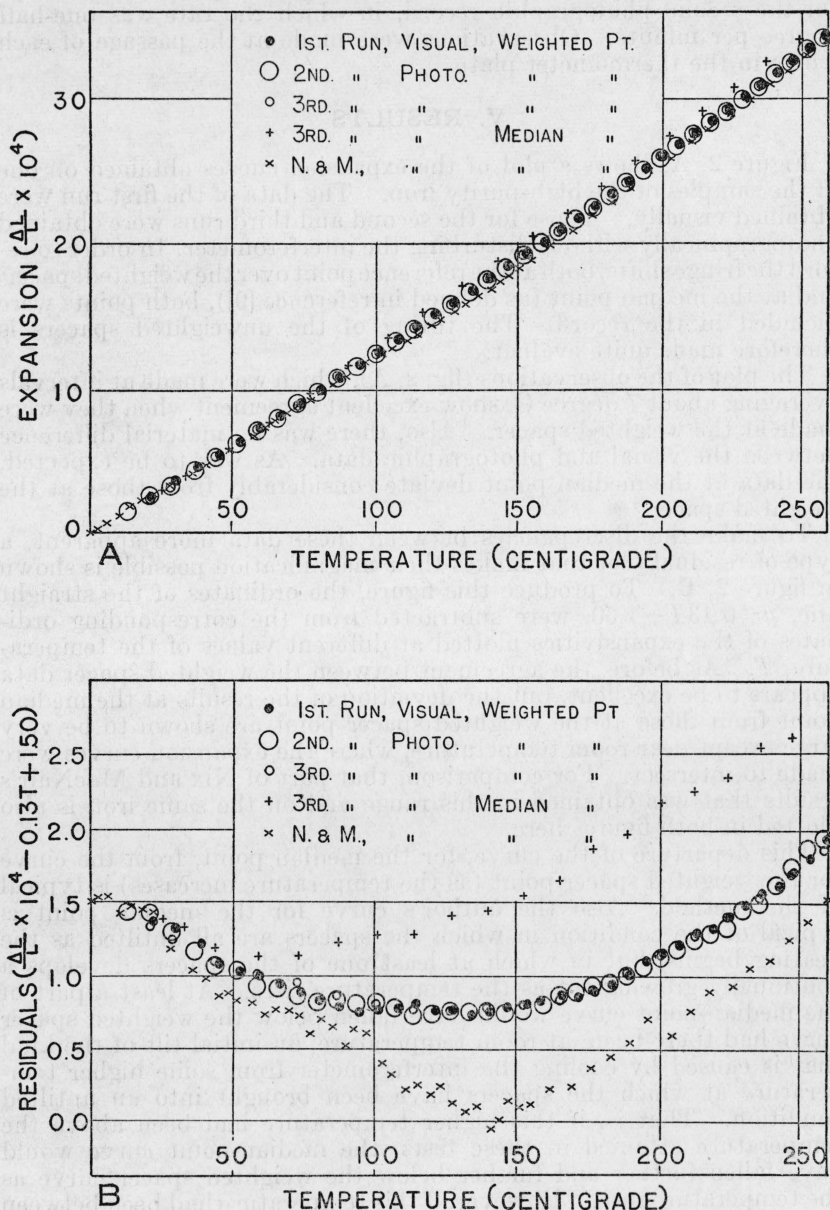


FIGURE 2.—Linear thermal expansion of iron as determined by several interferometer procedures.

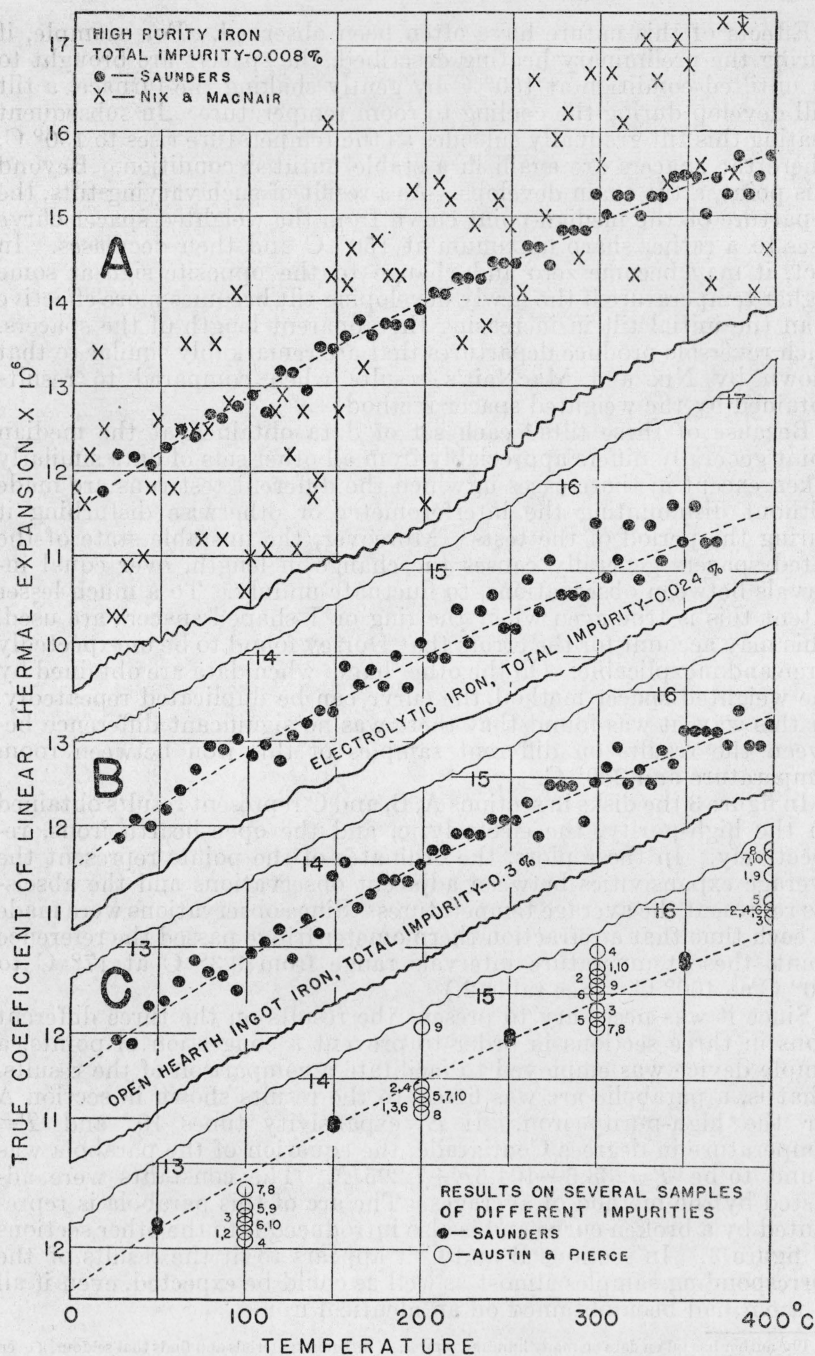
Effects of this nature have often been observed. For example, if during the preliminary heating described, the spacers are brought to an untilted condition at 150° C by gently shaking the furnace, a tilt will develop during the cooling to room temperature. In subsequent heating this tilt gradually subsides as the temperature rises to 150° C, where the spacers are again in a stable untilted condition. Beyond this point, a tilt again develops. As a result of such varying tilts, the departure of the median-point curve from the weighted spacer curve rises to a rather sharp maximum at 150° C and then decreases. In fact, it may become zero and change to the opposite sign at some higher temperature if the newly developing tilt becomes more effective than the initial tilt in increasing the apparent length of the spacers. Such reversals produce departures that are remarkably similar to that shown by Nix and MacNair's results when compared to results obtained by the weighted spacer method.

Because of these tilts⁴ each set of data obtained at the median point generally differs appreciably from all other sets of data similarly taken except in those cases in which the different test runs are made without dismounting the interferometer or otherwise disturbing it during the period of the tests. Moreover, the unstable state of the tilted spacers generally causes the change in length, over equal intervals between observations, to fluctuate unduly. To a much lesser extent this is true even when the ring or T-shaped spacers are used. This may account for the errors that Dorsey found to be unexpectedly large and inexplicable. On the other hand, when data are obtained by the weighted spacer method, the curve can be duplicated repeatedly. In this way, it was found that there was no significant difference between the results on different samples of this iron between room temperature and 260° C.

In figure 3 the disks in sections A, B, and C represent results obtained on the high-purity, the electrolytic, and the open-hearth irons, respectively. In these plots, the ordinates of the points represent the average expansivities between adjacent observations and the abscissas represent the average temperatures. Since observations were made at each time that a refraction thermometer fringe passed the reference point, these temperature intervals range from 9.3° C at 17° C to 6.0° C at 400° C. (See table 2.)

Since it was necessary to present the results on the three different irons in three sections in order to prevent a congestion of points, a simple device was employed to facilitate a comparison of the results. That is, a parabolic arc was fitted to the results shown in section A for the high-purity iron. If E = expansivity times 10^6 and T = temperature in degrees Centigrade, the equation of the parabola was found to be $T = 238.6 - 104.6E + 7.295E^2$. The constants were adjusted by the method of averages. The arc of this parabola is represented by a broken curve and is also introduced into the other sections of figure 3. In sections B and C it appears to fit the results of the corresponding samples almost as well as could be expected, even if all of them had been obtained on an identical iron.

⁴ The author has taken data on many hundred samples of different materials and finds that seldom, if ever, does the top interferometer plate fail to show vagrant changes in its inclination with respect to the bottom plate.



Pt equals point, and N&M equals Nix and MacNair.

FIGURE 3.—Comparisons of expansivity data for three samples of iron, having different amounts of total impurities, with some results already published.

TABLE 2.—Observed linear thermal expansion of relatively pure iron

Temperature	Expansion in microns per centimeter			Temperature	Expansion in microns per centimeter		
	Sample A	Sample B	Sample C		Sample A	Sample B	Sample C
°C				°C			
12.4	0.00			229.4	28.40	28.18	28.34
21.7	1.10	0.99	0.98	236.2	29.34	29.16	29.31
30.8	2.21	2.06	2.08	242.9	30.33	30.14	30.28
39.9	3.31	3.14	3.18	249.6	31.30	31.10	31.25
48.8	4.38	4.20	4.27	256.3	32.26	32.06	32.22
57.5	5.45	5.26	5.34	262.9	33.22	33.03	33.18
66.0	6.52	6.31	6.40	269.5	34.19	33.99	34.13
74.3	7.55	7.35	7.46	276.0	35.13	34.97	35.10
82.4	8.58	8.39	8.49	282.5	36.09	35.93	36.07
90.5	9.60	9.43	9.52	289.0	37.05	36.88	37.03
98.5	10.63	10.47	10.53	295.5	38.02	37.83	37.98
106.4	11.64	11.47	11.53	301.8	38.98	38.82	38.92
114.2	12.63	12.47	12.55	308.2	39.95	39.77	39.89
121.9	13.64	13.45	13.56	314.5	40.91	40.72	40.85
129.6	14.66	14.45	14.55	320.8	41.86	41.69	41.79
137.2	15.64	15.46	15.57	327.2	42.82	42.64	42.75
144.7	16.65	16.45	16.55	333.5	43.79	43.62	43.69
152.0	17.63	17.43	17.57	339.8	44.72	44.57	44.65
159.2	18.61	18.42	18.57	346.0	45.69	45.55	45.60
166.5	19.62	19.41	19.55	352.2	46.65	46.50	46.56
173.7	20.59	20.40	20.53	358.4	47.60	47.47	47.52
180.8	21.57	21.37	21.51	364.6	48.55	48.45	48.43
187.9	22.54	22.35	22.48	370.8	49.51	49.39	49.44
195.0	23.54	23.32	23.47	376.9	50.45	-----	50.39
202.0	24.55	24.31	24.45	383.0	51.41	-----	51.35
208.9	25.49	25.27	25.42	389.1	52.34	-----	52.28
215.8	26.45	26.23	26.39	395.2	53.28	-----	53.22
222.6	27.42	27.21	27.36	401.2	54.22	-----	54.15

A direct comparison of interferometer data that are taken with and without the weighted spacer procedure is afforded when the data obtained on sample A and the data taken by Nix and MacNair, on a sample from the same iron, are treated similarly. These data are plotted in section A of figure 3. To make this comparison of the two sets of data on the same basis it is necessary to use approximately equal temperature intervals in both cases. Fortunately, this is easily possible, since Nix and MacNair made observations at intervals that were either approximately the same or about one-half as large as those used by the present author. Consequently, although approximately equal intervals were used always in the computations, their data are represented by double the number of points (X's) in the range below 150° C, in which they used the shorter intervals. The dispersions of the points resulting from this treatment of data may seem rather large, but it will be found not to be unusual if a similar searching analysis is applied to any other data that result from interferometric measurements made at the median point when three individual tripod spacers are used.

In the lower section of figure 3 are plotted the expansivities of the 10 different samples that were reported on by Austin and Pierce [8]. These expansivities are represented by open circles, and the small numbers directly to the right and left of the circles represent the numbers assigned by the authors to the corresponding samples. According to them, "these data were derived from a curve representing a graphical average of the observations which include at least 30 points for each sample." The relative magnitudes of the expansivities represented by these points change considerably with temperature, and it is reasonable to assume that the differences in physical conditions

of the samples may have been partly responsible for these changes, but it is improbable that they were the only cause. It is believed that the total variation in impurity of these samples did not exceed the variation in total impurity of samples A, B, and C, which show no such relative change. Particular attention is called to the results on sample 8, for which the expansivity obtained is highest at 100°, lowest at 200° and 300°, and again highest at 400° C. Values derived from the expansion data of samples A, B, and C (see table 2) are also plotted here for comparison.

TABLE 3.—*True coefficient of linear thermal expansion of high-purity iron with comparative results on less pure irons*

Temperature	Sample A (total impurity 0.008%)	Sample B (total impurity 0.024%)	Sample C (total impurity 0.3%)	Difference (total spread)
°C	$\times 10^{-6}/^{\circ}\text{C}$	$\times 10^{-6}/^{\circ}\text{C}$	$\times 10^{-6}/^{\circ}\text{C}$	$\times 10^{-6}/^{\circ}\text{C}$
0	11.50	11.46	11.59	0.13
50	12.23	12.23	12.31	.08
100	12.87	12.89	12.93	.06
150	13.44	13.49	13.50	.06
200	13.96	14.04	14.01	.08
250	14.45	14.54	14.49	.09
300	14.91	15.02	14.94	.11
350	15.34	15.46	15.37	.12
400	15.75	15.89	15.77	.14

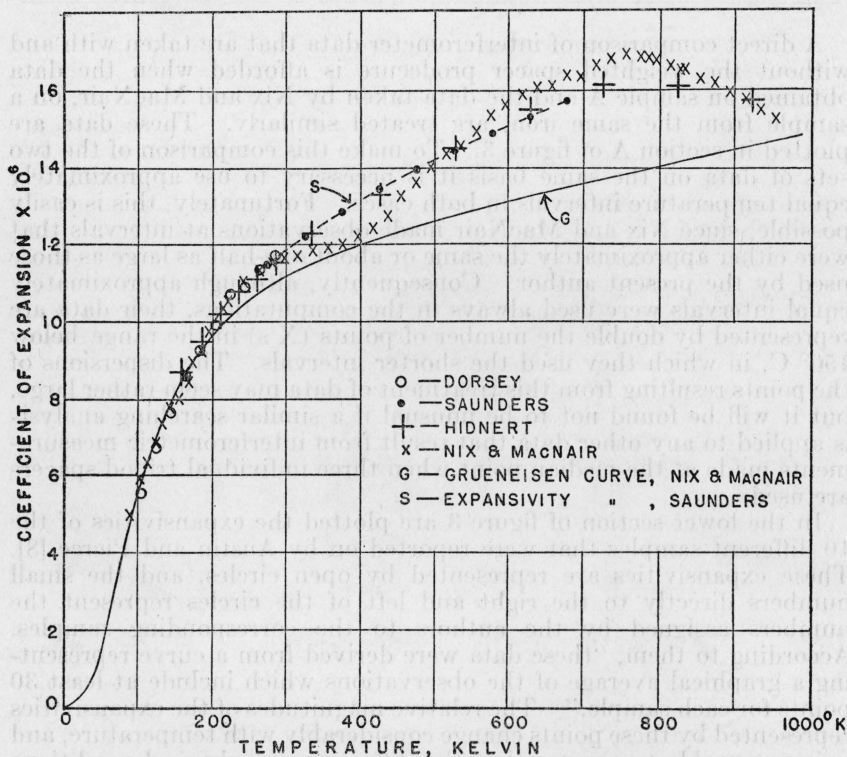


FIGURE 4.—*Expansivity of iron as determined by several observers, using different arrangements of the interferometer, compared with the precision comparator method.*

Figure 4 is a reproduction of Nix and MacNair's figure V [4], on which the results of the present investigation and those of Hidnert and Dorsey have been superimposed. The purpose is to show that the results obtained in the present investigation for the range from room temperature to 400° C are a smooth continuation of the trend followed by the results obtained by others for relatively pure iron in the range below 0° C. This plot also shows that the improved data now obtained are in reasonable agreement with those that Hidnert obtained by a different method [11]. Moreover, it appears that other values than those used by Nix and MacNair for the required constants must be used, if a Grueneisen curve is obtained that will conform with the expansivity as temperature changes.

VI. CONCLUSIONS

1. According to the results obtained, impurities do not affect materially the expansivity of iron in the range from room temperature to 400° C as long as the carbon content is less than 0.05 percent and the total of the usual impurities does not exceed 0.3 percent.

2. In these precise measurements there are no appreciable irregularities in the temperature range from room temperature to 400° C.

3. The precision of the interferometer method has been greatly increased by making observations at a point directly over the contacts between the interferometer plates and a stable spacer.

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