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SUBJECT

The Effect of Type of Cold-Deformation
on the Recrystallization Properties
of Armco Iron

By

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The Effect of Type of Cold-Deformation on the Recrystallization Properties of Armco Iron

NAVAL RESEARCH LABORATORY
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WASHINGTON, D. C.

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Test on Preliminary Model YSC Equipment

ABSTRACT

The recrystallization of Armco iron when cold deformed by torsion, elongation in tension, and rolling, and subsequently annealed, has been studied with care taken to keep variables other than the type of deformation as alike as possible. From the data in each case a recrystallization diagram was constructed. It was found possible to relate the critical degree of deformation at any chosen annealing temperature for the three different types of cold deformation by expressing the degree of deformation either in terms of specific shear or better (following Nadai), in terms of octahedral shear. Experiments on the effect of time of anneal were also carried out.

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I. INTRODUCTION

The study of the various factors which determine the grain sizes and structures of metals which have been cold worked and annealed started about ninety years ago and considerable work has been done in the past twenty years. Some of this work has been done because of the growing scientific interest in single crystals of metals; some because of the industrial importance of avoiding the production of coarse crystalline products in the working and fabrication of metals; and some for sundry and scientific purposes.

The purpose of the present work is a study of the type of deformation in relation to the process of recrystallization on annealing after cold work. Three types of deformation were studied; shearing deformation in a twisted bar, elongation in tension, and compression in rolling. For each case a "recrystallization diagram" was determined with the experimental conditions as nearly alike as was feasible. In addition, various special experiments were carried out to gain an insight in the influence of several other factors which are operative in the phenomena of recrystallization. The two chief factors which determine the grain size which results in a piece of metal when it is cold worked and subsequently annealed are the degree of deformation and the temperature of annealing. It is quite obvious that in spite of the importance of these two factors there are other factors involved, such as the time of annealing, the kind of mechanical deformation, the temperature of deformation, the concentration and nature of impurities in the metal, the grain size and structural peculiarities of the starting material, the previous history of the material, and so on. The nature and number of these factors almost discourages a comprehensive complete treatment of this subject even for a single metal but at least something may be accomplished by concentration of experiments on particular factors.

Narrative of Research Work Done at the Naval Research Laboratory

The experimental work of this report was initiated by Doctor R. F. Mehl, former superintendent of the Physical Metallurgy Division. The work originated in a need to learn more about the procedure required in producing

metal test pieces possessing various grain sizes to be used in other experimental work such as internal friction studies and so forth. The data given were obtained during 1929 and 1930, but due to the demands of other research studies it was not put in form for report or publication.

In view of the fact that such recrystallization studies may be of interest in connection with other metallurgical problems such as the plastic deformation and annealing of iron and mild steel sheet or the indentation and penetration of iron plate by projectiles, the authors have organized and correlated the experimental data obtained.

The experimental work was aided by gifts of Armco iron for research from the American Rolling Mill Company, Middletown, Ohio. Credit is also due to Mr. A. R. Donaldson of this Laboratory for his assistance in preparing the photographs shown in this report.

II. RECRYSTALLIZATION OF ARMCO IRON DEFORMED BY TORSION

Material and Mechanical Deformation

The material used in this work was 1-inch round Armco iron bar stock. Two lots were received having the composition given below:

	Lot 1	Lot 2
C	0.015%	0.011%
Mn	-	0.012
P	0.008	0.004
S	0.027	0.028
Cu	-	0.041
Si	0.006	-

While these compositions are practically the same, Lot 2 is probably of higher purity, for less inclusions were noted in it.

The material was sawed into bars about 11 inches in length which were turned smooth to a diameter of 15/16 inch. (See Fig. 4(a).) These were then packed in asbestos, heated 3 hours at 930° C and cooled in the furnace.

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Light reference lines for subsequent measurement of the amount of permanent torsion were engraved upon the bars. The bars were then twisted various amounts in a torsion testing machine. The amount and uniformity of the torsion in each case was determined from measurements on the engraved lines on the bars. The torsion was surprisingly uniform. A bar (Specimen B) from Lot 1, representing a range of specific shear, 0 - 0.125, equivalent to a range of elongations 0 - 11.5%, was selected for further study. This bar, which furnished the data of Table 1, was sawed into cylindrical samples 1/2 inch in height.

Annealing Treatments

The cylindrical samples were annealed at the following experimental temperatures: 300, 400, 500, 600, 700, 760, 780, 800, 825, 850, and 875° C. The time of annealing was generally taken at 3 hours except where specified otherwise in Table 1. To eliminate the effects of annealing at temperatures other than the temperature studied, it was decided to carry out the heating operation as rapidly as possible and thus the sample wrapped in copper foil (to minimize surface oxidation) was dropped into a hole in a massive copper block held in the furnace at the desired temperature. At the end of the annealing period the sample was quenched.

Metallographic Studies

In the initial part of this work each specimen was examined both on sections through and perpendicular to the bar axis; but since it was found this way that the structures produced by the annealing treatment were equiaxed, only the normal sections were used in later studies. Photomicrographs were taken on the prepared normal section at different distances from the center both before and after annealing. These served to determine the grain size by the usual counting method. In those samples where the largest grains were very large, the largest grain sizes were determined from grain counts between concentric circles drawn upon a macrographic view of the whole section.

Effect of Annealing Time

A study of the effect of the annealing time was made by taking microviews of a Lot 1 section, prepared after annealing for 20, 40, 90, and 170 minutes at 825°. No appreciable differences in these were noted, leading to the conclusion that the chosen annealing period of 3 hours

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was ample to insure full recrystallization. Later, however, a specimen from Lot 2 annealed for 15 minutes under these conditions failed to show a complete structure, so 15 minutes should not be looked upon as sufficient. The metallographic data obtained have been summarized in Table 1, and these data have been used to construct the recrystallization diagram shown in Fig. 1. Fig. 2 shows typical microstructure observed in the torsion specimens from Bar B, Lot 1, annealed at 780, 825, and 875° C. It must be mentioned here that the curves shown in Fig. 1 represent averages of the grain size data from which single determinations of the grain size may differ somewhat. This is especially true for the data corresponding to the lower annealing temperatures 780° C and below. These show clearly how the critical deformation decreases as the annealing temperature increases and also how much more poorly defined the critical degree of reduction becomes at lower annealing temperatures. It may also be noted on these sections that there are regions where the recrystallization did not take place, especially in the specimen annealed at 780° C. Although the ring of largest grains is not very distinct, it appears at a fairly definite position radius. This effect may be due to a non-uniform distribution of the impurities in the metal, causing some regions to behave differently from the rest, but an annealing time study at 825° C showed that there was very little change in the shape and size of these areas as the annealing time was increased from 20 minutes to 3 hours.

In the diagram of Fig. 1 the vertical portions of the curves for 800° C and higher were located to show as closely as possible the minimum shear at which large grains were formed. For the 780° C curve this line could not be drawn because the grain sizes tapered to a maximum over a range of radii. It should be noted that the curve for 760° shows a very slight peak, corresponding to a specific shear around 0.10.

Since the point of main interest in this work was always the effect of a particular annealing temperature on a particular mechanical deformation, no study of the rate of heating was attempted. Slow heating of the specimen would have meant exposures for appreciable lengths of time to annealing temperatures other than the particular one being studied. Although this does not preclude the possibility that the rate of heating may influence the results in this work, a uniform technique of rapid heating was followed.

The effect of time of heating was at first studied only at 825° C and led to the conclusion that the results would be about the same in the range 1/2 hour to 3hours. However, it might be suggested that annealing for long periods of time at lower temperatures might bring about effects not noticed in the initial work. This proved to be so and some results obtained will be described and discussed later in their proper connection.

A factor that could not be studied or controlled well in this work was the initial grain size. Although a much smaller initial grain size would have been welcome, the initial grain size here was probably as small as could be hoped for after the necessary normalizing treatment to remove effects of previous history as far as possible.

To make sure that the data given in Table 1 are truly characteristic of the material, two torsion bars with different degrees of maximum shear were prepared from Lot 2 stock. Samples from these were annealed as before at 780, 800, and 850° and the resulting structures obtained are shown in Fig. 3. The critical deformation given by the critical radii in the two bars are in substantial agreement with each other and also with that obtained from the bar of Lot 1. This agreement will be illustrated again later in connection with the correlation of critical degrees of deformation in various types of cold work, as may be seen in Table 4A.

Table 1

GRAIN SIZE DATA FROM TORSION BAR STUDY #

Annealing Temp. °C	Annealing time hrs.	Radii mm r	Ave.Gr.Size before Anneal*	Ave.Grain size after Anneal	Sp.Shear $\gamma = \frac{r\theta}{l}$ = r x .02083
300	3	3	3.5	4.7	0.063
		6	4.6	4.9	0.125
		9	4.2	6.3	0.188
400	3	0	4.7	4.1	0.000
		3	4.8	-	0.063
		4	-	4.3	0.083
		6	4.0	-	0.125
		8	-	4.5	0.167
		9	4.8	-	0.188
		11	-	6.0	0.229
500	3	0	4.8	-	0.000
		2	-	4.6	0.041
		3	4.0	-	0.063
		5	-	5.4	0.104
		6	4.8	-	0.125
		8	-	5.5	0.167
		10	4.6	-	0.208
		11	-	4.8	0.229
600	3	2	4.8	5.2	0.041
		5	-	5.9	0.104
		6	4.0	-	0.125
		8	-	5.4	0.167
		10	5.7	-	0.208
		11	-	6.6	0.229
700	3	2	4.6	5.1	0.041
		5	5.4	5.9	0.104
		8	5.6	6.7	0.167
		11	6.9	7.9	0.229
760	3	.5	5.8	-	0.010
		2	-	5.8	0.041
		5	5.8	-	0.104
		7.5	-	8.4	0.156
		8	5.5	-	0.167
		9.5	-	9.9	0.198
		11	5.5	-	0.229
		11.5	-	8.7	0.240

All the above data were obtained from Bar Specimen B, Lot 1.

* Grain sizes given in sq.microns x 10^{-3}. The lowest grain size is around ASTM No. 5, the largest reported greater than No. 0.

Table 1 (cont'd)

Annealing Temp. °C	Annealing time hrs.	Radii mm r	Ave.Gr.Size before anneal	Ave.Grain size after anneal	Sp.Shear $\gamma = \frac{rs}{l}$ = r x .02083
780	3	3.5	-	28.1	0.073
		4.5	-	27.5	0.094
		4.9	10.7	-	0.102
		5.5	-	39.0	0.115
		6.5	-	48.1	0.136
		7.5	-	49.0	0.156
800	3	2	6.0	-	0.041
		4.3	-	8.0	0.090
		5	6.0	-	0.104
		5.3	-	723	0.111
		5.4	-	684	0.113
		6.7	-	254	0.139
		8	10.0	34	0.167
		9.3	-	17	0.194
		10.7	-	16	0.223
		11	8.0	-	0.229
825	3	1.7	-	17	0.035
		2.0	8.0	-	0.041
		3.2	-	13	0.067
		4.5	-	21	0.094
		5.0	7.0	1135	0.104
		6.0	-	180	0.125
		7.4	-	75	0.154
		8.0	8.0	-	0.167
		8.9	-	63	0.185
		10.0	-	30	0.208
		11.0	6.0	-	0.229
		11.4	-	17	0.238
850	3	2.0	6.0	-	0.041
		3.9	7.0	-	0.081
		4.3	-	2000	0.090
		4.8	-	225	0.100
		5.0	6.0	-	0.104
		7.8	-	47	0.163
		8.0	8.0	-	0.167
		10.8	9.0	19	0.225
875	3	1.6	-	9	0.033
		2.0	11.0	-	0.041
		3.9	-	220	0.081
		4.4	-	1570	0.092
		5.0	10.0	-	0.104

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Annealing Temp. °C	Annealing time hrs	Radii mm r	Ave.Gr.Size before anneal	Ave.Grain size after anneal	Sp.Shear $\gamma = \frac{r\theta}{l} = r \times .02087$
		5.9	-	133	0.123
		7.9	-	52	0.165
		8.0	10	-	0.167
		9.2	-	30	0.192
		10.5	-	19	0.219
		11.0	10	-	0.229
		11.5	-	21	0.240

Critical Radii of Recrystallization -

Temp. °C - Below	760	760	780	800	825	850	875
Radii, mm.	-	10	8	4.5	4.1	4.0	2.8
Sp.Shear	-	0.208	0.167	0.094	0.085	0.083	0.058

III. RECRYSTALLIZATION OF ARMCO IRON DEFORMED IN TENSION

Material

The material was Lot 2 Armco iron. This was machined into nine bars of the dimensions shown in Fig. 4(b).

Preliminary Heat Treatment

The bars were wrapped in copper foil and an additional wrapping of asbestos paper. Since the material in the stock condition showed an unsatisfactory grain structure, coarse grains being interspersed through the finer grained matrix, it was heated back and forth through A_3 for about three-quarters of an hour. This treatment produced a structure which was fairly uniform.

Deformation

In order to measure the amount and also the uniformity of elongation, scratches were spaced one-half inch apart on the surfaces of the bars. This spacing was carried out by means of the dividing screw of a lathe. The bars were then elongated in a tensile-testing machine to approximately 2, 4, 5, 6, 8, 10, 16, and 20 per cent elongations. Then the elongation and the uniformity of the elongation in the bars were measured by means of a comparator.

Final Heat Treatment

Although the elongation was found to be uniform, the division of the material for heat treatment was carried out according to a system. Fig. 4(b) shows the manner in which the samples were lettered. Each sample (about 1/2" in length) was punch marked with the number of the bar and the letter indicating the position in the bar according to the above sketch. The marking preceded the division of the bars into samples. The "G" samples from each bar were reserved for the comparison of the cold worked stages. The other samples were heat-treated according to the table below:

Sample	Annealing Temperature	Time
E (all bars)	875° C	1-1/2 hours
F (all bars)	825° C	2-1/2 hours
H (all bars)	780° C	2-1/2 hours
D (all bars)	760° C	2-1/2 hours
I (all bars)	700° C	2-1/2 hours

Microscopic Work and Photography

The grain size was measured on the cross-section of each sample. Usually four measurements spaced at about equal intervals across the diameter were taken. In the case of samples manifesting coarse grains, it was frequently noticed that many small grains were located in the grain boundaries of the large grains. Making a grain count gave a result which could give no hint of the size of the large grains. Therefore in such cases grain counts were also made in which the grains having a cross-section small compared to that of the larger were ignored.

After the grain count work was completed, photomicrographs were taken of the cross-sections of all the annealed samples. The purpose of this work was not only to get a series of representative views of typical structures resulting from the annealing, but also to study the base effect which the superimposed deformation of punch marking had upon the samples.

Table 2

SUMMARY OF GRAIN COUNT DATA, TENSION STRAINED AND ANNEALED ARMCO IRON

Annealing Temp.	% Elongation 1.1	3.46	4.72	6.14	7.83	8.63	15.93	19.90
700°C	4.42	5.60	6.51	3.31	5.56	4.19	5.05	6.30 #
760°C	3.56	3.50	4.25	5.34	10.13	8.91	4.23	5.80
760°C*	-	-	-	-	38.00	27.07	-	-
780°C	3.37	5.83	5.35	5.46	52.8	7.30	5.18	6.96
780°C*	-	-	-	10.24	-	47.9	-	-
825°C	3.94	3.90	43.04	54.35	47.65	15.42	4.10	5.48
825°C*	-	-	219.2	108.5	-	35.71	-	-
875°C	3.72	4.76	139.4	100.7	55.0	46.9	5.12	5.71
875°C*	-	-	-	-	-	-	-	-

All grain sizes given in square microns x 10^{-8}.

* Grain counts made neglecting the relatively small grains.

Critical Reductions** Corresponding to the Annealing Temperature.

Annealing Temperature :	875	825	780	760	700
Critical Reduction :	4%	4%	6.2%	7%	-

** These values except the one for 780° were taken as the mean of the two lowest percentages of reduction between which there is a marked change in grain size. That for 780° was chosen as the smaller value because at this value a noticeable increase in grain size was observed.

Discussion of Results Obtained in Tension Work

The data obtained have been plotted in a recrystallization diagram shown in Fig. 5 for comparison to the diagrams obtained from the other experiments. The difficulty of making a satisfactory grain count on the specimens because of the presence of numerous fine grains in the grain boundaries made necessary, as mentioned before, a separate grain count in which the grains of comparatively small cross-section were not counted. For the samples annealed at 875° C this procedure was not required but for those annealed at lower temperatures it was quite necessary in order to show what the size of the largest grains produced by the mechanico-thermal treatment really is. The curves corresponding to each annealing temperature except 875° C have been modified and the modified form of the curve used to plot the space diagram.

Fig. 6 shows a set of macro-views for series E tension-strained samples annealed at 875° C for 1.5 hours. An inspection of the photo-macrographs will show that the above arbitrary procedure was quite justified in order to get a true picture of the size of grains that will result in any particular treatment. These pictures also show the effect that punch marking the specimens had upon the recrystallization structure. This effect seems to vary from sample to sample.

In the diagram constructed, the critical values were more or less arbitrarily chosen as mean values of two deformations between which there occurred a marked change in the grain size. Therefore an inspection was carried out of all the photo-micrographs taken in this work to note the degrees of deformation between which the structure passes from the worked to the non-worked state for any particular annealing temperature.

The results of the above inspection regarding the critical strain corresponding to each annealing temperature have been summarized in the table below and compared with estimates made from the grain size data and from an inspection of the photo-micrographs of the complete sections. The agreement is close enough but there always remains the possibility that the values obtained from an inspection of the photo-micrographs are slightly too high because of a misinterpretation of the appearance of sections.

Critical Deformations Corresponding to Each Annealing Temperature

Temperature	Critical Elongation Per cent		
	Judged by grain size data	Judged from photomicrographs	Judged from appearance of macrophotographs
875°	3.46 - 4.72	3.46 - 4.72	3.46 - 4.72
825°	3.46 - 4.72	3.46 - 4.72	3.46 - 4.72
780°	6.14	6.14 - 7.83	about 7.83
760°	7.83 - 8.63	about 7.83	about 7.83
700°	19.9 (?)	-	10%

That is, what may seem to be a cold work structure may in reality be an initial grain decomposition preceding recrystallization. In the above inspection this difficulty was kept in mind but the structure accepted as completely recrystallized were those in which no trace of either cold work or incipient grain decomposition could be seen.

IV. RECRYSTALLIZATION OF ROLLED ARMCO IRON

Initial Heat Treatment and Preparation of Material

Round bar stock (Lot II) of one inch diameter was machined into square bars 5/8" x 5/8" x 12". These were wrapped in several thicknesses of copper foil and a layer of asbestos. The material was introduced into an electric muffle furnace at about 1600° F (870° C) and was heated and cooled several times through Ag as in previous work. The upper temperature attained was 1695° C (923° C) and the lower temperature 1604° F (923° C). After heating between these limits several times during 1-1/2 hours the material was finally furnace-cooled down to 1670° F (909° C).

This treatment did not produce satisfactory conditions in the material. Therefore another treatment was given in which the material was kept at 1695° F (923° C) for two hours, the idea being to cause growth of the fine grains caused by the transformation and thus equalize out the grain size contrast. This treatment caused excessive grain growth in spots. The material was therefore again heated 1695° F (923° C) and allowed to cool to 1640° F (893° C). This was repeated about three times. The resulting material showed on examination a structure which although not entirely uniform was acceptable.

The above operations caused enough oxidation of the surfaces of the bars to make necessary a light regrinding of two parallel surfaces in order to make an accurate determination of the percentage reduction in rolling.

Deformation

The samples were all cold rolled by several passes through rolls of about 12 in. diameter. The work was started with the smallest required reduction and subsequent pieces were rolled on their first pass to the final required thickness of the previous sample and were finished by two to six more passes through the rolls. The samples requiring the greater reductions were thus rolled down in much severer steps. Micrometer readings of the thickness of the samples were taken before and after rolling at several points along the bar, and their averages were used to compute the actual percent reduction of the bars. The thickness of all of the bars before rolling was very close to 0.565 in. In the rolling it was attempted to obtain certain percentages reduction. These are listed below along with the percentages obtained.

Percentage Reduction of the Samples

Expected % :	1	2	3	4	6	8	10	15	30
Obtained % :	0.70	1.74	2.98	4.15	5.99	8.02	10.05	14.71	29.82

Final Heat Treatment

After rolling, the bars were cut up according to the plan shown in Fig. 4(c) by means of a slow band saw, the specimen being kept cool during the sawing by an air blast.

The samples were heat treated according to the table below:

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Sample	Annealing Temperature	Time
A (all bars)	875	3 hrs.
B (all bars)	850	3 hrs.
C (all bars)	825	3 hrs.
D (all bars)	800	3 hrs.
E (all bars)	780	3 hrs.
F (all bars)	760	4 hrs.
J (all bars)	Not annealed.	--

The samples were wrapped in copper foil and introduced into the furnace at the required temperature. A piece of plate copper, 1/4 inch thick, formed the floor of the furnace. At the end of three hours the samples were removed from the furnace and cooled in air. Each sample was cut across the direction of the bar at a position half-way between the original cut ends of the sample. This cut surface was used for microscopic examination.

Microscopic Examination and Grain Size Determination.

To minimize labor and expense, photomicrographs were not taken so extensively as they were in the experiments on Armco iron deformed in tension. Photomicrographs were taken of each of the worked samples (Series J) and used in determining grain size of these samples but for all other samples grain size counts were made on the ground glass of the metallographic camera. However, each sample was carefully examined over its whole section to note peculiar features. In some of the samples large grains appeared in isolated places on the sample or in groups in certain regions of the section.

To measure the sizes of these large grains they were traced on the camera back and their areas obtained by means of a planimeter. As in the work on tension strained Armco iron it was noted that fine grains often coexisted with very large grains. The planimetric tracings did not take these into account and thus gave a measure of the sizes of the largest grains formed. Grain size measurements were made as exactly as possible on each specimen, in the center of the section and slightly to each side of the center. The grain size and structure data for these experiments is somewhat more involved than in the experiments on pieces strained in torsion and elongation. The data are arranged below in Table 3 to give not only the exact grain size values determined at the centers of the sections but also structural peculiarities and sizes of large grains. Fig.7 shows the recrystallization diagram constructed from the data. Here the planimetrically determined grain sizes are understood.

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Table 3

GRAIN SIZE DATA OBTAINED FROM ROLLED AND ANNEALED ARMCO IRON SAMPLES

Annealing Temp.	Percentage Reduction	Grain Size at Center ($\mu^2 \times 10^{-3}$)	Sizes of Largest Grains ($\mu^2 \times 10^{-3}$)	Remarks
875°	0.70	19.76		Unrecrystallized.
"	1.74	21.54		Unrecrystallized.
"	2.98	17.64		Large grains appear near surface.
"	4.15	20.08		" " " " "
"	5.99	21.57	606	Large grains in surface. Center still unrecrystallized.
"	8.02	37.10	303	Large grains form in center. Corners remain unrecrystallized.
"	10.05	31.57	136	Large grains form in center. Corners remain unrecrystallized.
"	14.71	23.16		Structure uniform.
"	29.81	15.93		Fine grain.
850°	0.70	18.57		Unrecrystallized.
"	1.74	21.26		Unrecrystallized.
"	2.98	18.96	68	Large grains appear near surface.
"	4.15	19.31	134	Large grains on surface and sides.
"	5.99	18.11		Large grains on surface and sides.
"	8.02	40.31	107	Large grains form in center.
"	10.05	35.28	140	Uniform recrystallization structure.
"	14.71	21.38		Fairly uniform.
"	29.81	12.76		Fine grain.
825°	0.70	21.09		Unrecrystallized.
"	1.74	25.75		Unrecrystallized.
"	2.98	27.31		Unrecrystallized.
"	4.15	22.56		Unrecrystallized.
"	5.99	25.38		Large grains appear on surface and sides.
"	8.02	83.80	428	Large grains on whole section except in corners.
"	10.05	49.07	140	Large grains on whole section except in corners.
"	14.71	24.08		Complete recrystallization.
"	29.81	13.01		Fine grains.
800°	0.70	23.82		Unrecrystallized.
"	1.74	19.26		"
"	2.98	25.51		"
"	4.15	17.64		"
"	5.99	23.09		"
"	8.02	23.41		Slightly larger grains in surface
"	10.05	33.93	110	Large and small grains formed except in corners of section.

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Table 3 (cont'd)

Annealing Temp.	Percentage Reduction	Grain Size at Center ($\mu^2 \times 10^{-3}$)	Sizes of Largest Grains ($\mu^2 \times 10^{-3}$)	Remarks
800°	14.71	18.21		Fairly uniform structure.
	29.81	9.53		Small grain size.
780°	0.70	22.10		Unrecrystallized.
"	1.74	17.49		Unrecrystallized.
"	2.98	24.52		Unrecrystallized.
"	4.15	19.62		Unrecrystallized.
"	5.99	22.04		Unrecrystallized.
"	8.02	20.82		Unrecrystallized.
"	10.05	22.43		Slightly larger grain size.
"	14.71	15.64		
"	29.81	8.61		
760°	0.70	-		
"	1.74	20.37		Unrecrystallized.
"	2.98	19.46		Unrecrystallized.
"	4.15	-		
"	5.99	19.28		
"	8.02	-		
"	10.05	17.09		Possibly a slight grain size increase.
"	14.71	-		
"	29.81	6.37		

Critical Percentages of Deformation

as Judged from the Preceding Data

Annealing Temperature	Critical Reduction for Surface Grains	Critical Reduction for Center of Section
875	2.98	5.99 - 8.02
850	2.98	5.99 - 8.02
825	4.15 - 5.99	5.99 - 8.02
800	8.02	8.02 - 10.05
780	10.05	10.05
760	10.05	10.05 - 14.71

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Discussion of Results

In this work the same sort of difficulty in grain size determination appeared that was found in the work on tension strained Armco iron. As before, there was also noted non-uniformity of the recrystallization structure for the smaller degrees of deformation. It was found that rather small reductions of 3 and 4 percent were sufficient to cause the appearance of very large grains just below the rolling surface of the piece. Then as the percentage reduction was increased, the large grains began to be formed on the sides of the section as well as just under the surface. On increasing the deformation, there appears an inversion of the structure. Large grains appear at the center of the section while the borders are relatively small grained. As the deformation is still further increased, the structure becomes more and more uniform over the whole section. This series of transitions is illustrated in Fig. 8 by a series of macrographs for the samples of series A.

The observed phenomena are easily attributed to non-uniformity of deformation in the rolling process. In spite of this non-uniform structure it is possible to follow the practice of past workers on recrystallization and limit consideration to the center region of the cross-section. The grain size curves accompanying this report have been obtained from the grain size values obtained for the center of the section. It was found that, as was found in the work on tension strained material, the relatively large grains formed for a critical percent reduction were often accompanied by minute grains in the grain boundaries which, when an accurate grain count was taken, would bring the grain size down to a value giving no hint of the size of the largest grains produced. Accordingly, at those deformations at which relatively large grains appeared, the values of the sizes of the largest grains, determined from planimetry of tracings, were used to modify the grain size curve. In plotting the space diagram these modified grain size curves were used, to conform with the practice adopted in plotting the results of the tension-straining and annealing work.

The critical percentages of reduction necessary for the formation of large grains at the center of the section and also near the surface of the specimen have been tabulated. Of course these cannot be stated exactly because the critical reduction generally would occur between two values used in the experiment. These limiting values are therefore used in the table. It will be noted in this table that the critical deformations corresponding to the metal near the surface are much less in value and more sharply defined than those corresponding to the center of the section. Those for the center of the section are considerably higher than the values reported in the work on both tension and torsion strained Armco iron. A very similar result was obtained, however, in some

experiments on the recrystallization of hammered Armco iron.

V. REVIEW OF RESULTS

The primary interest of this report is not in the grain sizes that appear in the recrystallization but rather the relationship between the amounts and kinds of cold deformation required to produce recrystallization. The grain size is dependent not only upon the deformation but also upon such other factors as impurities, initial grain size, etc. The reader may gain an insight into these various factors from some of the references given at the end of this report.

Most of the data of this report have been summarized in Fig. 1, 5, and 7 which are the recrystallization diagrams obtained when the kind of deformation was torsion, elongation and rolling, respectively. Fig. 2, 6, and 8 give macro-photographs which illustrate the general features of each case. These recrystallization diagrams, it must be repeated, are valid only for conditions under which the experiments were performed. One of these is that the time of annealing was shown of moderate length so that the subsequent effects of grain growth were minimized. The effect of time of annealing will be mentioned again.

In the diagrams of Fig. 1, 5, and 7, there may be seen a curve in the base plane of the diagram which gives approximately the critical deformation or that minimum degree of deformation required to bring about recrystallization at any given annealing temperature. In general these curves show that the higher the degree of deformation, the lower the temperature required to produce recrystallization. It must be mentioned, however, that toward the lower annealing temperatures, these critical deformations are more and more poorly defined, so that only at the higher temperatures can we look upon the critical deformation as a quantity capable of being determined even roughly. It would be more correct to regard the critical deformation versus temperature curve here discussed as the center of gravity of a zone or range of deformations which narrows down as the temperature in question is increased.

The curves in the base of Fig. 5 and 7 are rough curves which are intended to show the practical deformation which must be exceeded in any case to bring about a large grain structure throughout the section in the case of tensile elongation and rolling. These curves do not indicate the degree of deformation at which large crystals may start to grow here and there in the section. This must be mentioned to avoid misunderstanding in the following discussion which centers about the critical deformation required to initiate the recrystallization process in any given case.

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Therefore Table 4 below has been compiled which lists for each of the three types of deformation studied and for each annealing temperature used, that degree of deformation which is just great enough to initiate recrystallization. In the case of torsion strain, this is taken as the critical specific shear corresponding to that radius in the cylinder bar where a zone of recrystallized grain starts. It might be argued that a slightly greater radius corresponding to the mean centers of the first ring of grains would be the more likely starting point, but as said before, we are here interested in determining the least possible degree of deformation at which recrystallization has taken place.

In the cases of tension and rolling, the critical deformation cannot be stated more closely than the intervals in the steps by which successive specimens were deformed. Therefore, in the table the critical values for tension are indicated by the ranges in which the critical deformation is judged to lie, while in rolling, the least reduction at which recrystallization took place was chosen because here the intervals were smaller, thus allowing better judgment.

It being the object of this paper to relate the recrystallization behavior of Armco iron deformed in various ways, it is necessary to express the degree of deformation in any case in terms of a more universal unit. This desired relationship between different types of plastic strain has also been sought for in connection with strain -- hardening brought about when metals are cold -- deformed in different ways. Sachs* has considered this subject and concluded that the hardening in any type of deformation was determined by the magnitude of the deformation measured in the direction of the greatest amount of deformation and furthermore that if q is the greatest specific elongation or reduction, it is related to the specific shear γ by the relation:

$$q = 1 - e^{-\gamma/2}$$

when γ is small (< 0.1), this is approximated by $q = \gamma/2$.

The values of γ corresponding to q have been computed in the case of elongation and rolling and entered in Table 4. If the averages of these are plotted against the corresponding annealing temperature as is done in Fig. 9(b), it will be seen that there is fair agreement in the case of tension and elongation but the points for rolling are definitely out at temperatures of 825° and less.

* Mechanische Technologie der Metalle, Leipzig, 1925, pp.54-62.

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CORRELATION OF DIFFERENT KINDS OF CRITICAL DEFORMATION

IN TERMS OF SPECIFIC AND OCTAHEDRAL SHEAR

A. DEFORMATION BY TORSION

Ann.temp.°C

T_R	760	780	800	825	850	875
Sp.shear r	0.208	0.121 0.166	0.094 .141 .151	0.085	0.0584 .050 .061	0.584
Oct.shear r	0.169	0.107 0.136	.076 .114 .124	.069	.048 .041 .050	.047
r_n mean	.0169	0.121	0.105	.069	.0463	.047

Where more than one determination is listed for a temperature, the results are from samples from different bars. Bars from both lots I and II are represented here.

$$r_n = \left(\frac{2}{3}\right)^{1/2} \ln\left[1 + \frac{r^2}{2} + r\left(1 + \frac{r^2}{4}\right)^{1/3}\right]$$

B. DEFORMATION BY ELONGATION IN TENSION

T_R	700	760	780	825	875
$q = \frac{\Delta l}{l_o}$	0.1593	0.0783 .0863	0.0614 .0783	0.0346 .0472	0.0346 .0472
$r(-2\ln(1-q))$	0.3420	.160 .178	.123 .160	.0704 .0965	.0704 .0965
r_n	0.210	.104 .115	.0820 .115	.0470 .0640	.0470 .064
r_n (mean)	0.210	.110	.098	.056	.056

$r_n = \sqrt{2} \ln (1 + q)$

C. DEFORMATION BY ROLLING

T_R	760	780	800	825	850	875
$q = \frac{\Delta h}{h_o}$	0.100	0.100	0.080	0.0599	0.0298	0.0298
$\frac{r_n}{(-2\ln(1-q))}$	0.210	0.210	0.167	0.124	0.060	0.060
r_n	0.135	0.135	0.108	0.0823	0.0417	0.0417
(r_n^*)	0.172	0.172	0.136	0.101	0.049	0.049

$(r_n^*) = -2\left(\frac{2}{3}\right)^{1/2} \ln (1 - q)$ Case of pure shear as in rolling wide plate.

Nadai** has set up a mathematical theory of plastic strain by which plastic strains of finite magnitude may be expressed solely in terms of a pure shear termed the octahedral shear. He has given formulae by which the octahedral shear may be computed from the specific shear on the principal specific extensions or contractions. The formula for each case is given in Table 4, together with the values of the octahedral shear r_n computed from the data. When the mean octahedral shear values are plotted against their corresponding annealing temperatures we obtain the graph of Fig. 9(a) which is more satisfying than the one of Fig. 9(b) obtained by using the specific shear as the abscissae unit.

In Table 4, part C, may be noted some values of octahedral shear (r_n^*) which are obtained from the formula for the case of pure shear which is about the state of strain found when rolling wide sheets. As may be expected, these values do not agree with the other data because in this work the rolling was carried out in narrow bars in which the deformation is more like a simple compression.

When it is considered that the agreement of points plotted in Fig. 9(a) is good at the higher temperatures where the critical degree of deformation can be determined more definitely and still join at the lower temperature. The correlation between the results obtained from the three different types of cold work is satisfactory.

** Nadai, "Plastic Behavior of Metal in the Strain Hardening Range," I, J. Applied Physics, Vol. 8, Mar.1937, pp. 205-217.

Furthermore, it must be remembered that the critical degree of deformation is not only related to the temperature of anneal but also to such other variables as time of anneal, chemical constitution, etc. In fact, the critical degree of deformation is so decidedly dependent on the time of anneal that if it were not for the fact that in this work the annealing times used at the different temperatures were always about the same, the above correlations might not have been effected at all.

To determine just how important the time factor is, a few experiments were carried out, the results of which are illustrated in Fig. 10 and 11. The former shows the effect of changing the annealing time at 875° from 15 minutes to 3 hours. One may note an appreciable change in the critical radius and also a decided effect on the size of the grains themselves. It must be mentioned, however, that a previous experiment was made in which two annealing times were 1/2 hour and 3 hours and much less difference in results was noted so that it is likely that the 15-minute anneal created only a partial recrystallization; that is, as Ahrell has pointed out, the time factor is very important at the beginning of the recrystallization process.

However, this does not mean that the time factor vanishes as the time of anneal is increased indefinitely. Fig. 11 shows the effect of increasing the time of anneal from 1 hour to four days at 3 different annealing temperatures, all of which had little or no effect when used for only 3 hours.

While at first it may be very disconcerting to learn how decidedly the critical degree of deformation depends on the length of annealing time, this is what might be expected to hold from kinetic theory. Van Liempt has made an attempt to deduce the temperature of recrystallization theoretically by considerations similar to those used by Lindemann in his theory of melting. He derived an expression for the recrystallization temperature

$$T_R = \frac{K}{\ln 4\gamma t}$$

where K is constant or a function dependent on factors other than time, such as degrees of deformation, etc., γ the characteristic frequency, and t the time of anneal. This equation bears out the experimental observation that either the critical degree of deformation or the recrystallization temperature will depend upon the time of anneal.

In this connection it is necessary to point out the need for more precise use of the term recrystallization temperature. At present it is being used indiscriminately for a number of purposes. For example, by it some authors mean the temperature at which a work hardened metal will regain its original hardness, irrespective of whether or not a change in grain structure has taken place; others again use it in place of a temperature metallurgists call the equi-cohesive temperature; while still others look upon it as fundamental characteristic temperature of the metal. This last impression arises from the fact that at higher degrees of deformation the recrystallization temperature changes but little with increasing deformation.

It was the hope of early workers on recrystallization to relate the structural changes in recrystallization to the changes in hardness brought about, but in general it was found that those changes in the microstructure characterizing recrystallization usually occurred at temperatures quite above the highest annealing temperature required to remove the effects of work hardening. The hoped for relation between hardening and recrystallization received an additional set-back by the novel experiment of Beck and Polyani* who showed that an Al single crystal when bent increased in hardness and when straightened gained still more in hardness, but showed less tendency to recrystallize on annealing as compared to a piece that had only been bent. Similar effects were observed in polycrystalline Al by van Arkel and van Bruggen**. In the present work on Armco iron attempts were made to relate the changes in hardness to the changes in structure produced by recrystallization. Since no obvious relationship could be found these results have been omitted from this paper.

Fig. 12 shows plotted on a single sheet the available data in the literature on the relation of critical degree of deformation to annealing or recrystallization temperature for pure iron and mild steels. The considerable variation in these curves obtained by different workers indicates the importance of the effect of impurities on the recrystallization process. That all of the data could be grouped about a mean curve suggests that there is a fundamental relationship between critical degree of deformation and annealing temperature characteristic of the metal itself. To compare the results obtained in this work with these data, points from this work are included in the figure. It may be noted that

* Nature 19 (1931) pp. 505-6.
** Z.Physik 80 (1933) pp. 763-791.

these agree somewhat with values taken from data by D. J. McAdam for American ingot iron and also quite oddly with values determined by Hanemann in hot deformation (deformation carried out at anneal temperature) for mild steels. In general, the recrystallization temperature of Armco iron and mild steels is definitely higher than for electrolytic iron.

In conclusion, it must be repeated that the phenomena of recrystallization are complex and that much experimental work directed at particular factors, keeping other factors as alike as possible, must yet be carried out to get our knowledge of these phenomena on a more rational basis.

VI. SUMMARY

1. The recrystallization of Armco iron when cold deformed and annealed has been studied for the cases in which the cold deformation consisted in torsion, elongation in tension, and rolling. The results have been summarized in recrystallization diagrams.

2. By expressing the degree of deformation in each type of cold work in terms of the corresponding octahedral shear, the critical degree of deformation so expressed is the same for the three kinds of cold work studied. Therefore, by means of recrystallization studies in torsionally-strained bars, it is possible to establish the most important part of the recrystallization diagram (small deformations - high temperature) with attendant simplification of technique.

3. Studies on the effect of annealing time have shown that as has been anticipated by theory, the critical degree of reduction decreases as the length of annealing time is increased.

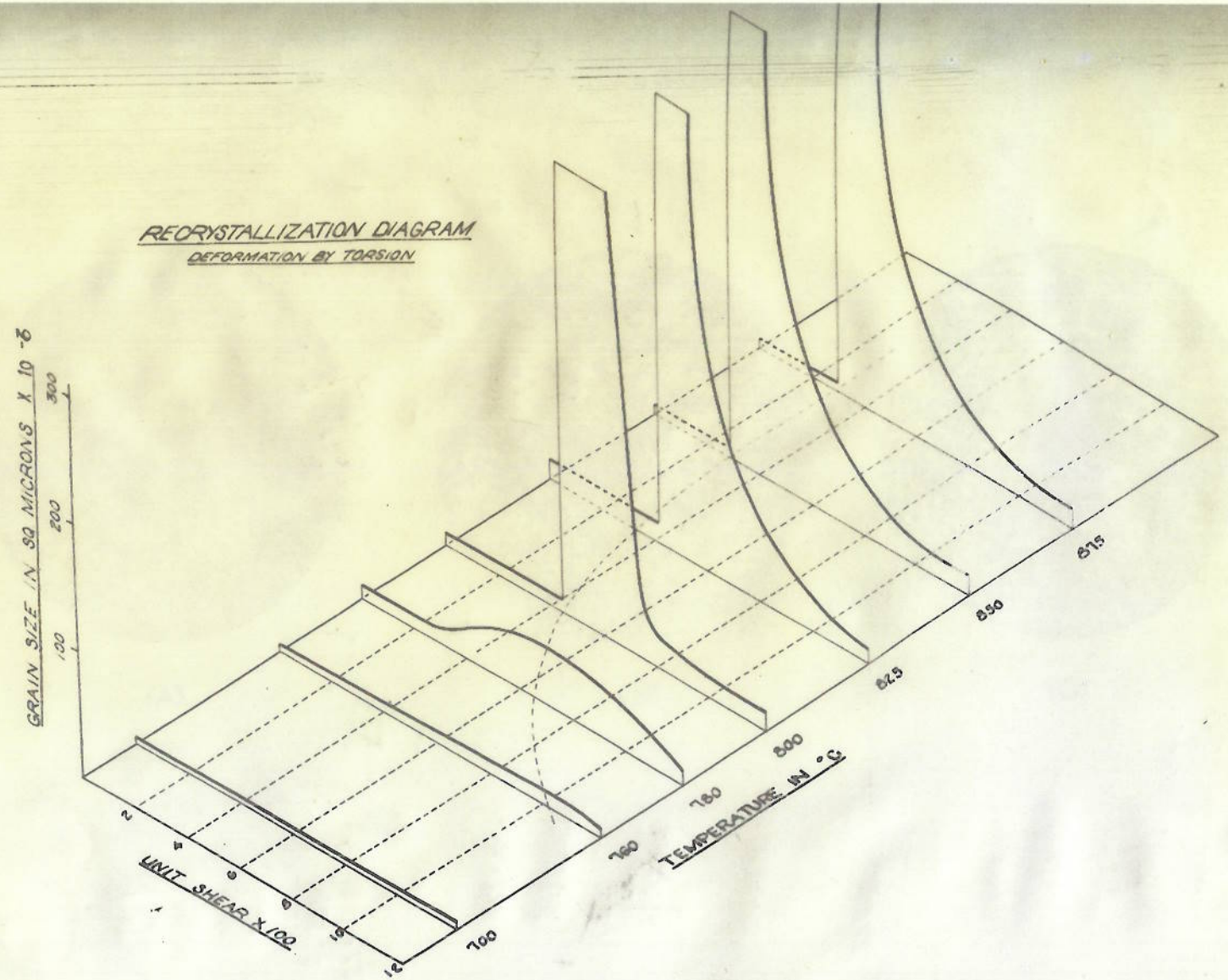
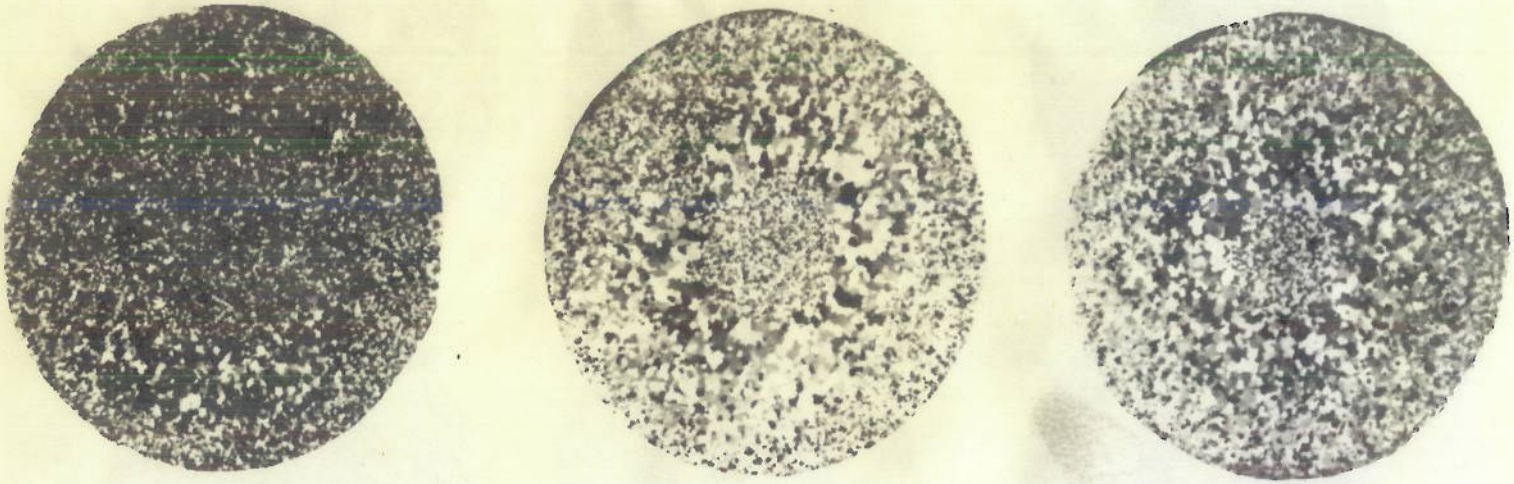


Fig. 1. **Recrystallisation diagram for Armco iron as determined by studies on torsionally deformed bars.**



(A) (B) (C)

Fig. 2 Macrographs of torsion bar samples after annealing (a) 780°C., 3 hrs.; (b) 825°C., 170 minutes; (c) 875°C., 2 hrs.

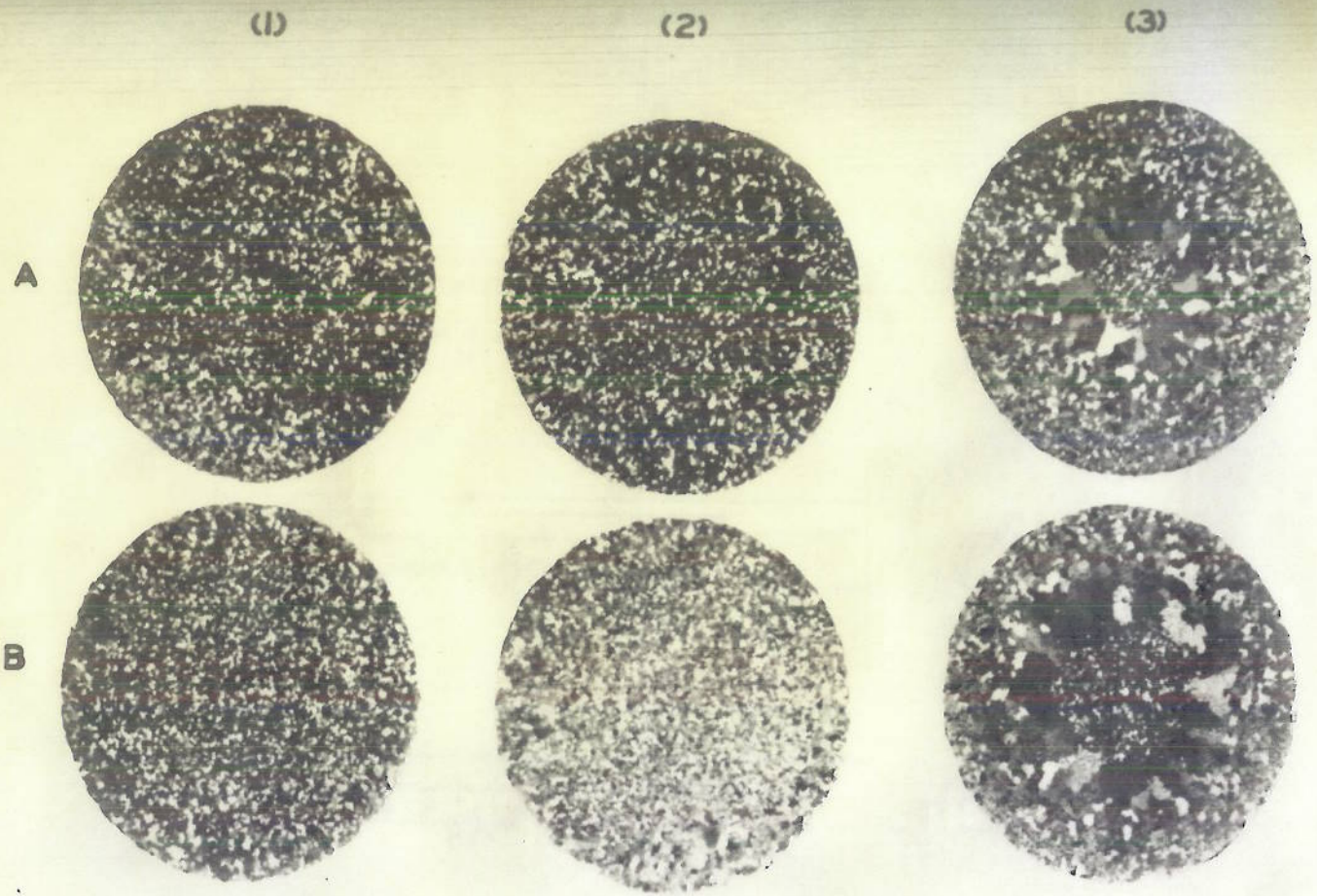


Fig. 3. Macrographs of torsion bar samples from two bars subjected to like annealing treatment. Column (1) 780°C., 3 hrs; (2) 800°C., 3 hrs.; (3) 850°C., 3 hrs.; top row, Bar A = x .0235 radians/mm. Bottom row, Bar B = x .0154 radians/mm.

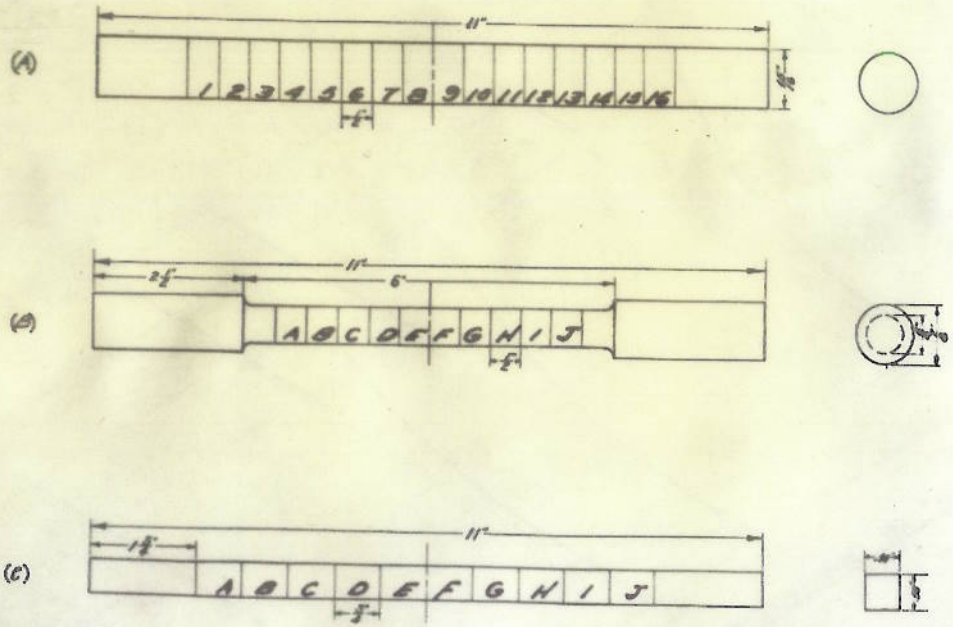


Fig. 4. Initial Dimensions of Samples. A. Torsion bar. B. Tensile bar. C. Rolling bar. Transverse lines indicate manner of taking samples.

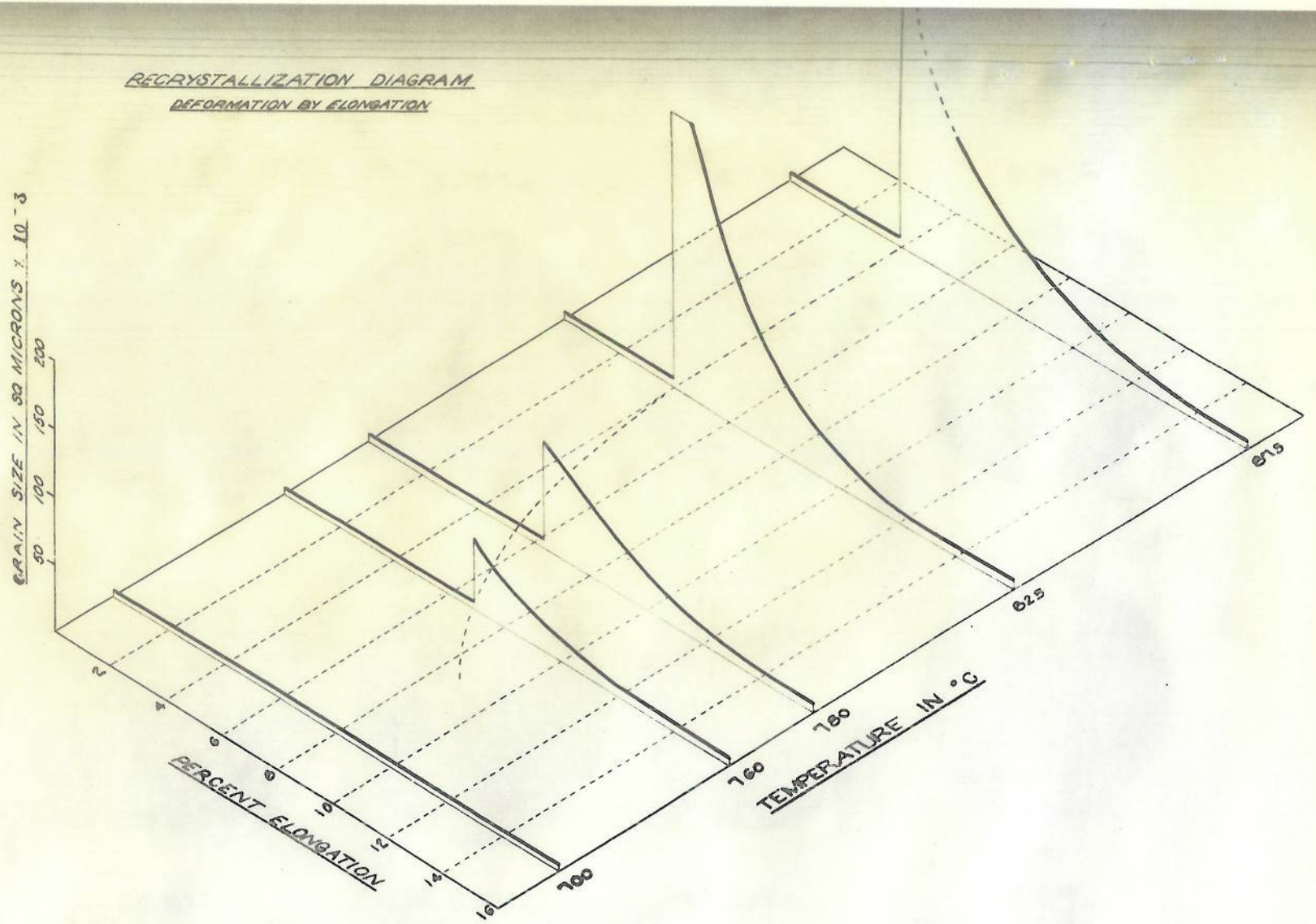


Fig. 5. Recrystallization diagram for Armco iron as determined by studies on tensile bars.

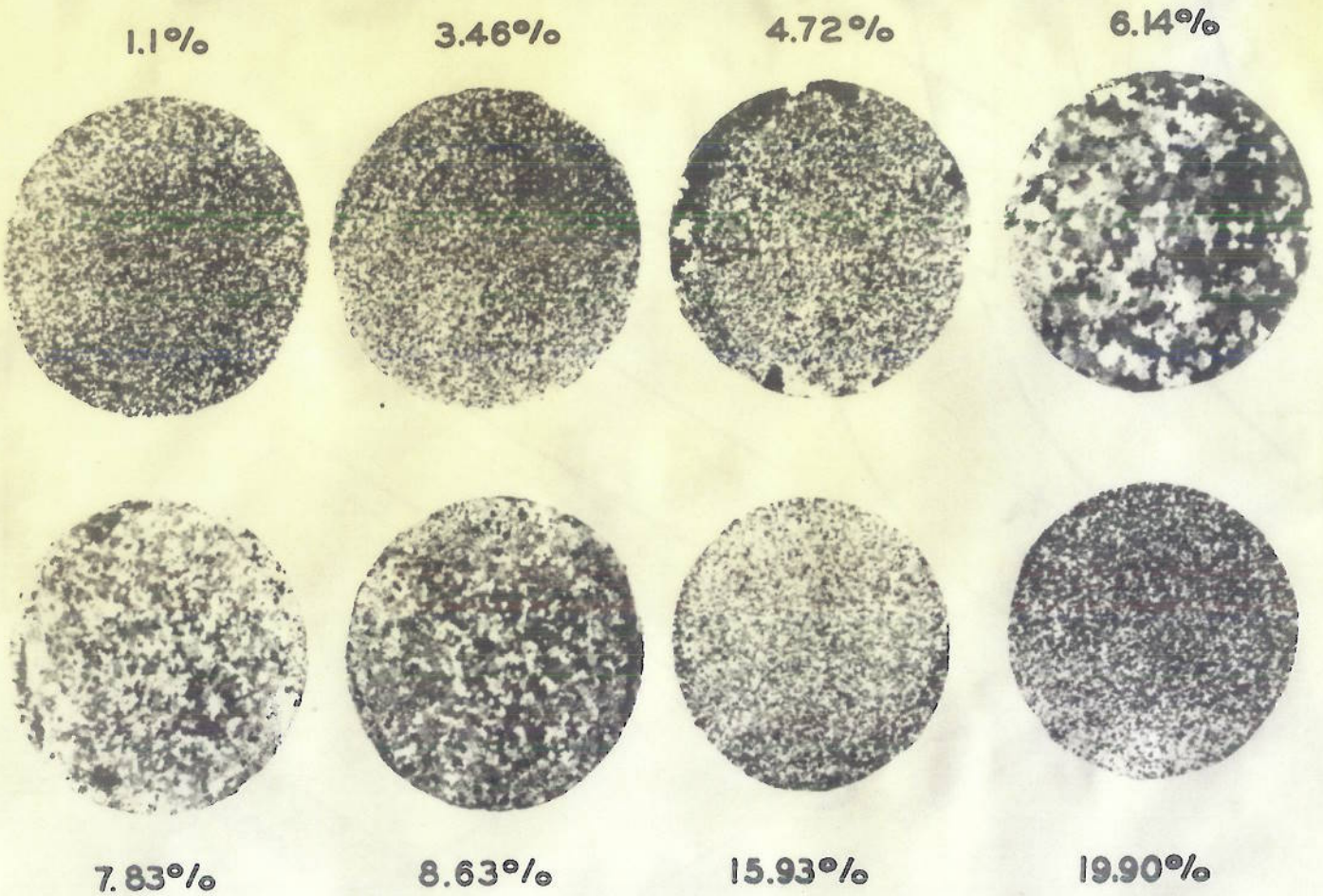


Fig. 6. Macrographs of tensile specimens elongated amounts shown and annealed at 875°F. 1.5 hours. (Series E).

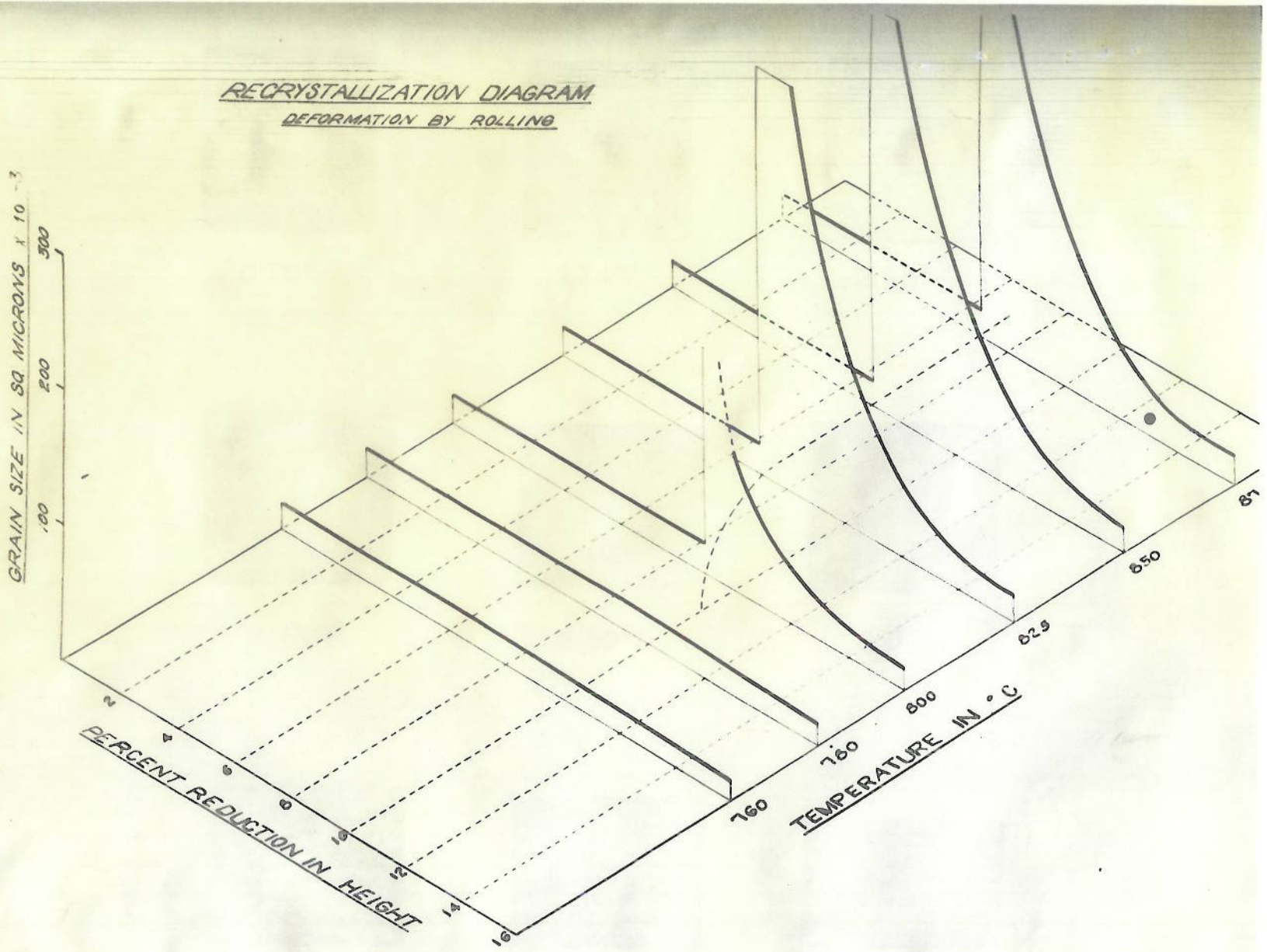
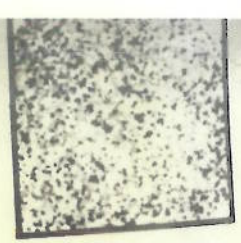


Fig. 7. Recrystallisation diagram for Armco iron as determined by studies on rolled bars.

0.70%

1.74%



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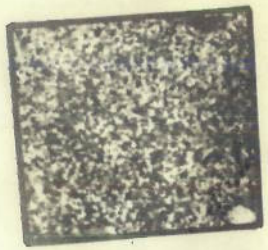

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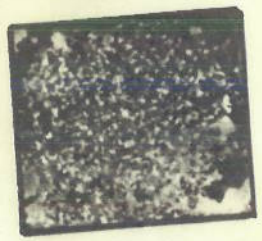

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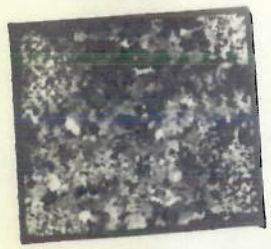

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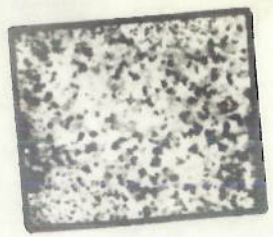

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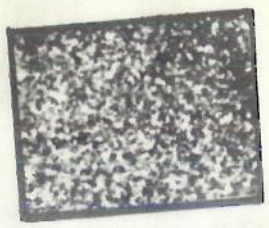


14.71%

29.82%

Fig. 8. Macrographs of rolling bar specimens rolled indicated and annealed at 875°C., ½ hrs.

1935

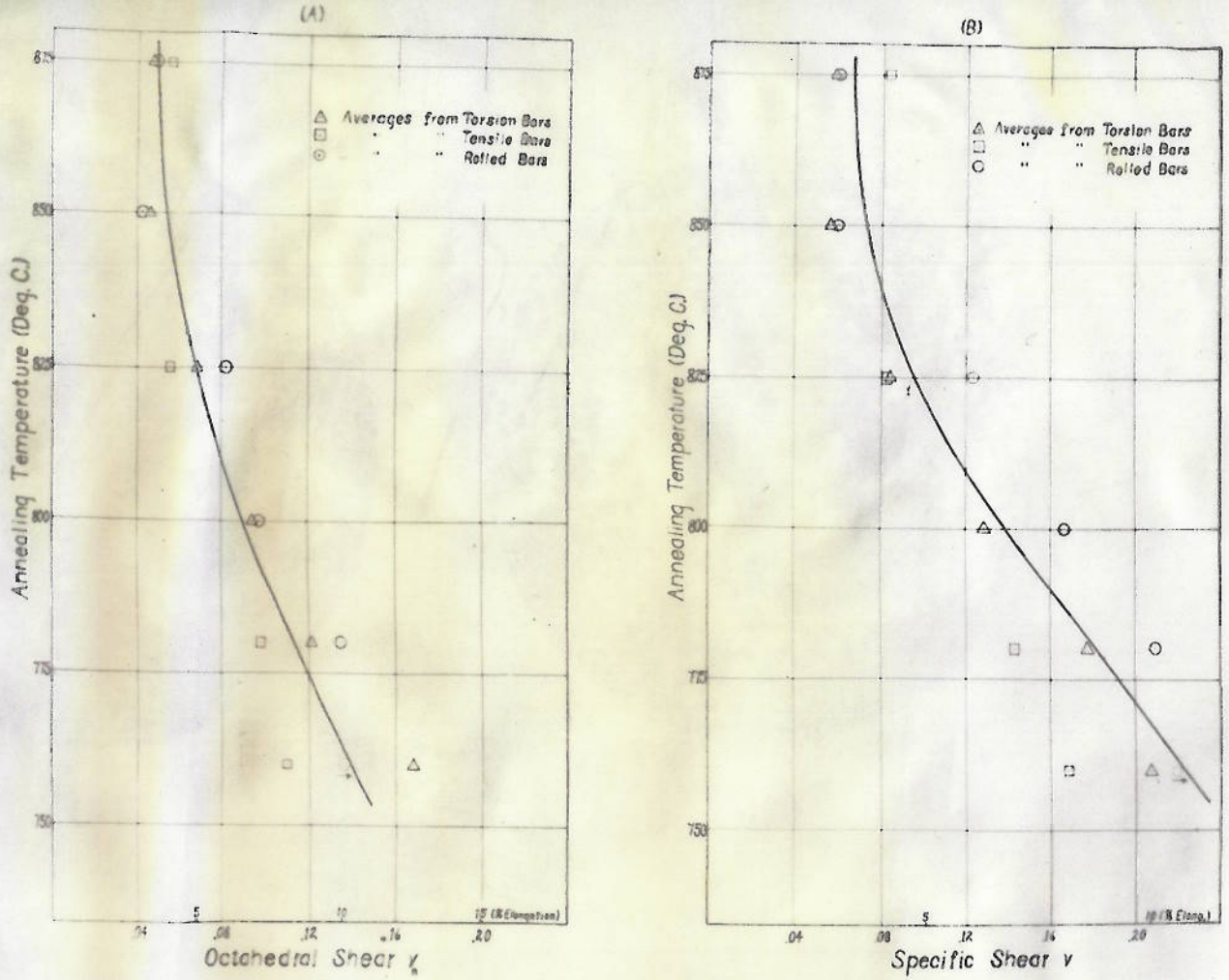
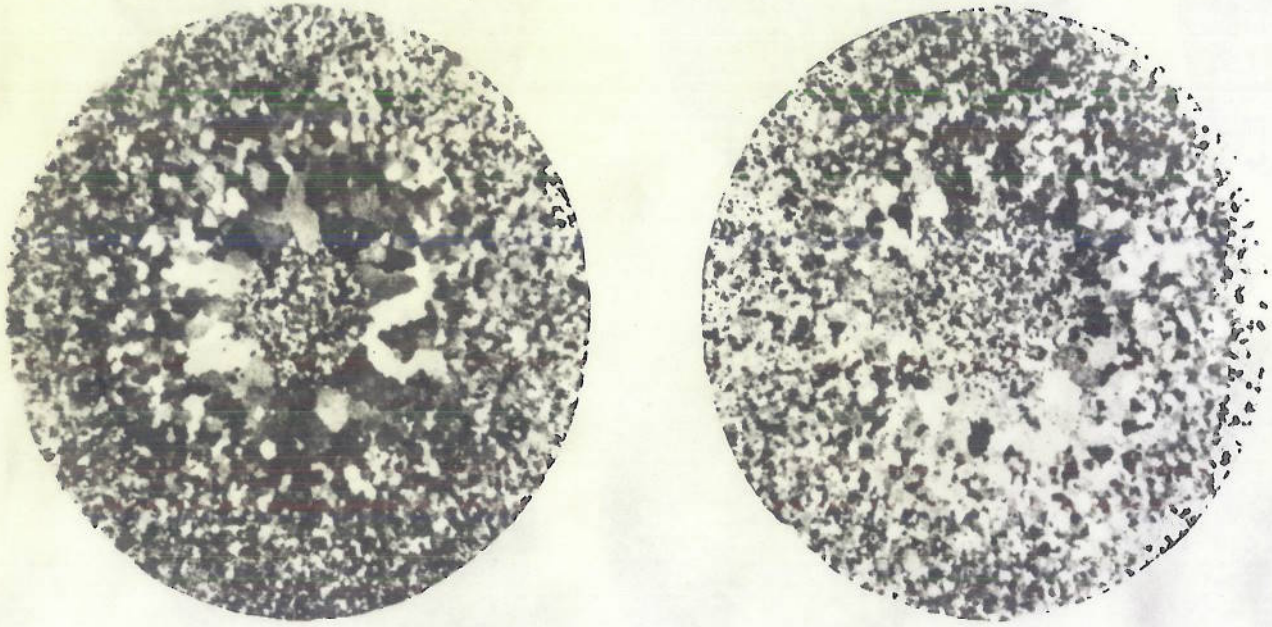


Fig. 9. Curves showing relation of critical degree of deformation to annealing temperature (a) when critical deformation is expressed as octahedral shear; and (b) when expressed as specific shear.



(A) (B)

Fig. 10. Macrographs of two torsion-strained bar samples annealed at 875°C. (a) 3 hrs., (b) 15 min.

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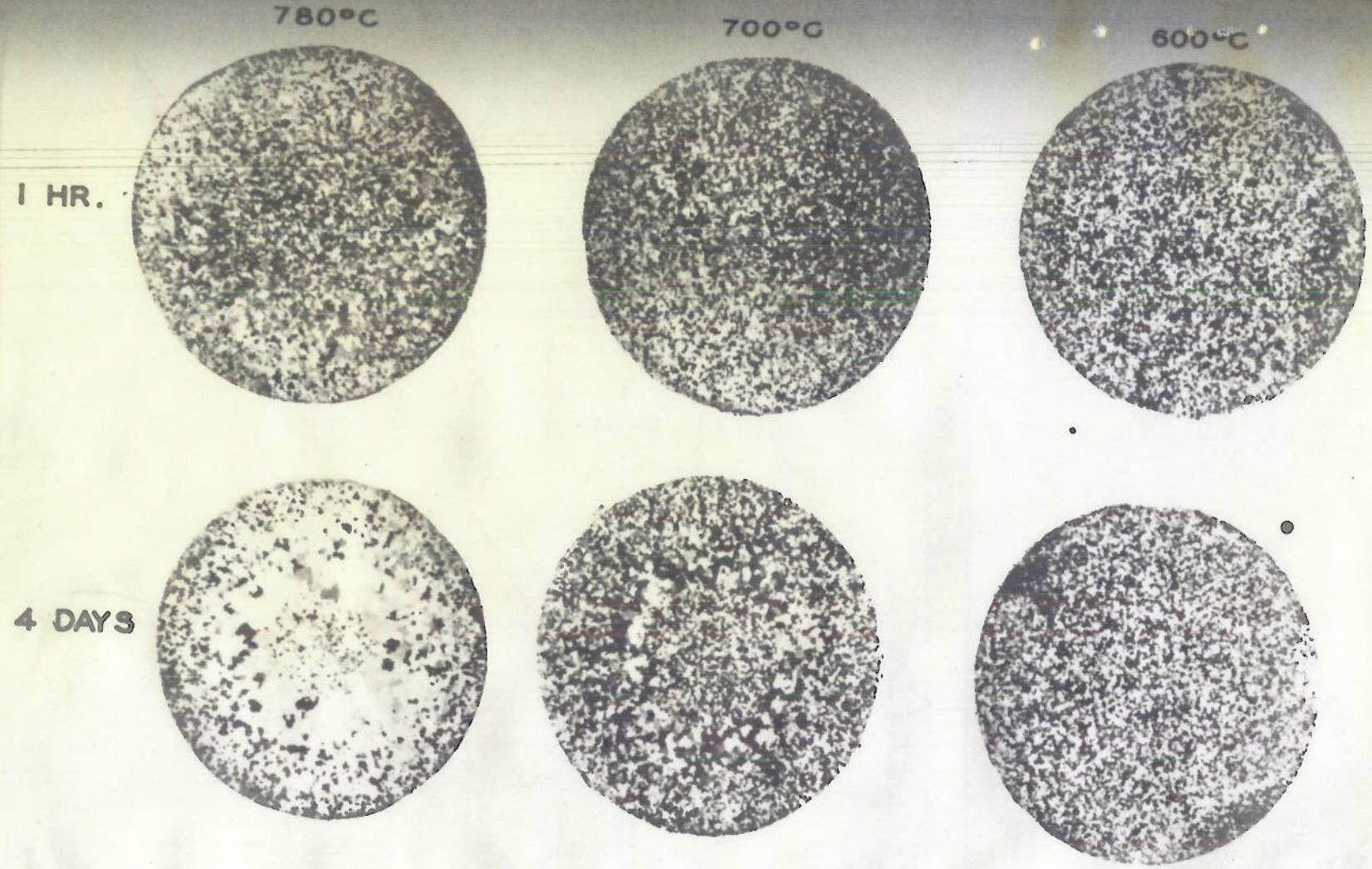

Fig. 11. Macrographs of torsion-strained and annealed samples (Bar A Lot II) comparing the effect of annealing 1 hour and 4 days at each of the temperatures 780, 700 and 600°C.

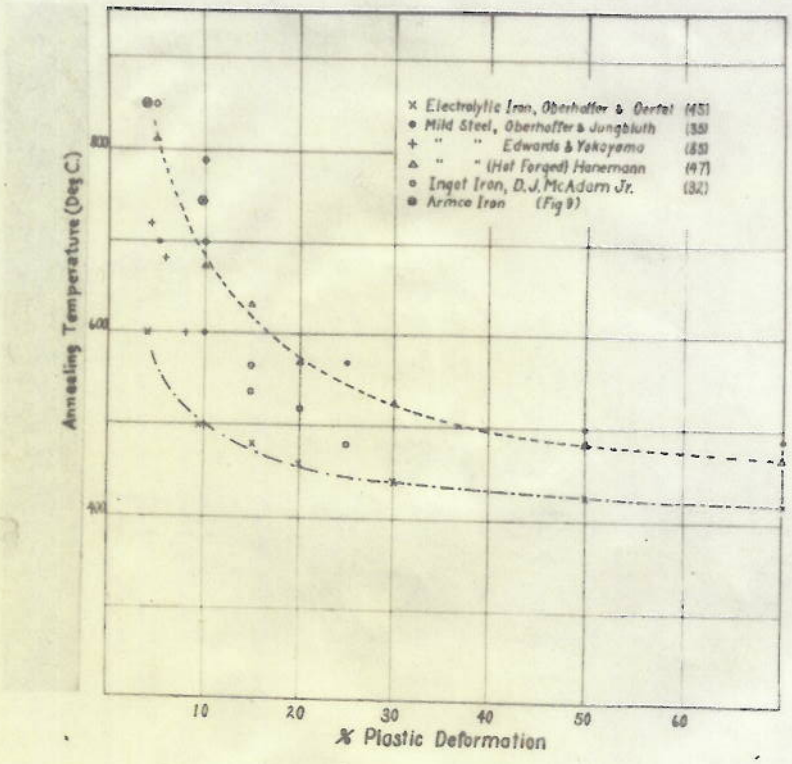

Fig. 12. Plot of existing data in literature on relation of recrystallization temperature to degree of plastic deformation for iron and mild steels.

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