DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 60

MICROSTRUCTURAL CHANGES ACCOMPANYING
THE ANNEALING OF CAST BRONZE

(Cu 88, Sn 10, Zn 2)

BY

HENRY S. RAWDON, Assistant Physicist

Bureau of Standards

ISSUED JANUARY 25, 1916

WASHINGTON
GOVERNMENT PRINTING OFFICE
1916

ADDITIONAL COPIES
OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.
AT
10 CENTS PER COPY

a, Bronze directly after casting, very slowly cooled. 100X; etching, ammonium hydroxide and hydrogen peroxide

b, Bronze in the annealed condition after "cold-working." The light and dark parallel bands indicate the twinned crystals. 100X; etching, ferric chloride

FIG. 1

MICROSTRUCTURAL CHANGES ACCOMPANYING THE ANNEALING OF CAST BRONZE (Cu 88, Sn 10, Zn 2)

By Henry S. Rawdon

CONTENTS

	Page
I. Introduction.....	3
II. Aim and scope of the work.....	4
III. Structure of the alloy.....	5
1. Cast condition.....	5
2. Annealed state.....	7
3. Transition of one form to the other.....	7
IV. Samples used—Preparation and treatment.....	7
1. Chemical composition.....	7
2. Preparation.....	8
3. Mechanical treatment.....	8
4. Thermal treatment.....	9
V. Results.....	10
VI. Discussion of results.....	13
1. Increase of crystal size.....	13
2. Persistence of the dendritic structure.....	13
3. Absorption of the eutectoid.....	14
4. Recrystallization follows only distortion.....	15
5. Progress of recrystallization upon heating.....	15
VII. Summary.....	17

I. INTRODUCTION

Most of the questions which this study attempts to answer arose in connection with the work on the standard specimens of zinc bronze (Cu 88, Sn 10, Zn 2), described in Bureau of Standards Technologic Paper No. 59. The samples were chosen from the series prepared in that investigation.

The physical properties of cast bronze are very materially altered by heat treatment, this modification of properties being accompanied by decided changes in the microstructure of the metal. These structural differences are especially marked if the sample has been subjected to mechanical work of any kind previous to the heat treatment (rolling, hammering, etc.). Fig. 1,

a and *b*, illustrates this change. The structural changes here noted as accompanying the annealing after distortion may be taken as typical of the copper-rich brasses and bronzes, and in general of most alloys in which copper is the predominating constituent.

II. AIM AND SCOPE OF THE WORK

The general purpose of the work is to show the stages through which the alloy passes in going from structure *a* (Fig. 1) to that shown by *b* of the same figure, and the conditions necessary for this change. While it is quite widely understood that condition *b* is the result of annealing, following distortion of the original structure, the opinion is by no means general that this is always true. Inasmuch as the common commercial use of annealing, in the application to brass or bronze, is to relieve the internal stresses of material which has been mechanically "cold worked" in some way, the resulting change of structure is often, though erroneously, regarded as an effect of annealing, pure and simple. By some this change of microstructure is seemingly regarded in the same light as the grain refining of steel.

Instances are found in the literature on this subject of the recrystallized state resulting directly from the cast condition upon annealing without the intermediary of the preliminary distortion of structure.¹ One of the main objects of this work is to see if such be true for this bronze—i. e., whether a condition resembling, in the effect produced, a "cold working" of the alloy can be brought about by very unusual methods of cooling. In speaking on this subject Portevin suggests that such may be the case. As the alloy in question is a complex one, ordinarily consisting of two phases in the cast condition and with a transformation of one of the phases involving a volume change, it appears that Portevin's suggestion may be the correct answer to the question. The temperatures at which the different structural changes are completed, together with the period of heating required at these temperatures, are also carefully noted.

No attempt is made to test out any of the various theories as to the exact nature of this process of recrystallization, although the

¹ Portevin, *Rev. de Mét.*, 6, p. 713; 1913. Several instances of such observations are summarized here.

results obtained appear to substantiate materially one of the two main theories on this subject. In short, answers to the following questions were sought: Is distortion of the crystalline structure a necessary condition for recrystallization? Can such distortion be approximated by any unusual methods of cooling? What changes other than "recrystallization" accompany annealing and at what temperatures are they completed?

III. STRUCTURE OF THE ALLOY

1. CAST CONDITION

The microstructure of the metal upon solidifying after casting is shown in Fig. 1a. The metal is composed essentially of a matrix consisting of a solid solution, α , of tin in copper (disregarding the zinc, which does not cause any pronounced structural change) in which are embedded inclusions of a eutectoid consisting of this same α solution and another one, δ , which is hard and brittle. The formation of the cored structure of the α matrix which is characteristic of solid solutions, will be made clear by reference to Fig. 2, which shows a portion of the equilibrium diagram of copper and tin.

Upon cooling, the bronze under consideration solidifies gradually through the range of temperature represented. The solidification is a case of selective freezing; that portion separating out first as a solid is richer in copper than the surrounding melt and may be represented in composition, approximately, by the point m' . The process of solidification begins simultaneously throughout the mass of the metal at many points. The subsequent successive additions of solid material to the nuclei thus formed will be lower in copper than the nucleus and may be represented in composition by successive points on the line m to a .

It has been found by observation that it is characteristic of solid solutions of metals for the crystals to grow faster in certain directions than in others during the process of solidification, thus fingers or branched forms result rather than geometrically symmetrical shapes. At 790°C , approximately, the alloy consists of crystals, each of which is an entangled treelike structure with a liquid portion, rich in tin, filling the crevices between the branches. At

790° this liquid portion (*B* Fig. 2) reacts chemically with some of the adjacent solid to form a solid solution (*b*) of copper rich in tin. This second constituent is termed the β solution. On further cooling, this β constituent breaks up into a eutectoid of the α and δ solutions of copper and tin at 500°C. The name "bronzite" has been suggested for this eutectoid.

FIG. 2

tin, the branches of which are not uniform in composition but have a core rich in copper. These branches intersect during the growth of the crystal and form a tangled mass with tiny crevices in the angles between the branches which are filled with a second metallographic constituent—a eutectoid comparable to the constituent, pearlite, in steel.

ite" has been suggested for this eutectoid. The changes in composition, due to diffusion during cooling, may be disregarded here and in cast alloys in general.

The bronze, then, after casting, consists of grains, or crystals, relatively large in size, i. e., large enough to render them plainly visible to the unaided eye. The size of such crystals will depend upon the rate at which the alloy cooled during the solidification, the freedom from mechanical disturbances, etc. Each grain has a complex structure and consists of a treelike framework of a solid solution of copper and

2. ANNEALED STATE

In its annealed state, represented by *b*, Fig. 1, the alloy consists of crystals generally much smaller than before. The large macroscopic grains have disappeared, as has also their interior dendritic structure with the accompanying eutectoid inclusions. The crystals are not only smaller, but are polyhedral forms quite characteristic of this condition. "Twinned" crystals are also common, as is illustrated in *b*, Fig. 1. The grains or crystals are now very homogeneous in structure throughout and consist of the α , or solid solution of copper and tin richest in copper. It may be noted, also, in passing that inclusions, such as stannic oxide, are unchanged in amount or distribution by the annealing process.

3. TRANSITION OF ONE FORM TO THE OTHER

Guillet² has suggested a method for converting a ternary alloy into a pseudo-binary one for purposes of discussion and comparison. In the case of the alloy under discussion (Cu 88, Sn 10, Zn 2) the method shows that the alloy may be considered for all practical purposes as one with a tin content of approximately 11 per cent. Reference to the copper-tin equilibrium diagram (Fig. 2) shows that such an alloy should consist of but one phase, the α solution. As Portevin³ has pointed out, the first step in the annealing process is to bring the alloy into the condition of "physico-chemical equilibrium," as shown by the equilibrium diagram. The second or tin-rich phase will disappear by being absorbed by the α solution; the heterogeneity of the α solution will disappear by diffusion. To produce the condition of "crystalline equilibrium"⁴ shown in *b*, Fig. 1, the large crystals must be subdivided, reoriented, and rearranged. The main point of this study is to show the conditions under which this crystalline equilibrium results.

IV. SAMPLES USED—PREPARATION AND TREATMENT

1. CHEMICAL COMPOSITION

The material used was chosen at random from broken test bars used in the investigation of the properties of the standard

² L. Guillet, *Rev. de Métal*, 3, p. 243; 1906, or C. H. Desch, *Metallography*, p. 191.

³ Portevin, *Rev. de Métal*, 6, p. 677; 1913.

⁴ Portevin, *loc. cit.*

zinc bronze. The chemical analysis of a casting made by melting about 25 broken test bars gave the following results:

	Per cent.
Copper.....	88.2
Tin.....	9.5
Zinc (by difference).....	2.3
Iron.....	Trace.
Lead.....	Not detected.

2. PREPARATION

In order to answer the questions set forth in Section II, the thermal treatment during preparation was made such as to include all possible cases arising in practice. The following series were prepared:

1. Hexagonal bars, 1 inch on a face, cast in green sand.
2. Small crucible melts, approximating 25 grams in weight. One set of these was quenched at 700° C, i. e., shortly after solidifying, so as to retain the β constituent; another set was allowed to cool rather rapidly in the air from the same temperature; and a third one was permitted to cool very slowly and uniformly in the furnace.
3. Small crucible melts (25 grams) quenched while in the molten condition, at 1200° C, by dropping the crucible and contents into cold water.

3. MECHANICAL TREATMENT

The samples received a certain amount of "cold work" either by being turned down in the lathe to a desired diameter or by drilling a central hole in the sample. Some specimens received both treatments. In this way only the surface layer of metal is distorted and the greater portion may be used for comparison after annealing. In the first series, although the amount of work each sample received was approximately the same, no special effort was made to make this so. The final series (2 b, below) was carefully prepared from samples which had cooled very slowly in the furnace. All samples which were to be annealed at the same temperature were cut from one specimen to insure identical structural conditions in each and were then copper plated so as to prevent loss by oxidation during the heating.

These series were treated as follows:

Series 1.—One-inch hexagonal sand casting, turned down to a three-fourths inch cylinder.

Series 2, a.—Cylinders fifteen-sixteenths inch diameter (25-gram melts), cooled at various rates upon casting, as indicated above, turned down to three-eighths inch diameter.

Series 2, b.—Some of the furnace-cooled specimens of No. 2 above were used. The cylinders of fifteen-sixteenths inch diameter were turned down to a diameter of three-eighths inch, the depth of the lathe cut being the same in each case. Samples for annealing at the same temperature (400°, 600°, and 800° C, respectively) were cut from the same cylinder. All samples were copper-plated to prevent surface oxidation.⁵

Series 3.—Melts quenched directly from the molten state, no work being done on them.

4. THERMAL TREATMENT

The samples were heated in a platinum resistance furnace which, because of its excellent insulation could be easily maintained at any desired temperature, which was measured by means of a thermocouple and potentiometer. The annealing temperatures decided upon were 400°, 600°, and 800° C, as the temperatures used ordinarily in a commercial way fall well within this range; 800° C was decided upon as the upper limit, as this temperature is very close to the beginning of the melting zone for this alloy, and it was desired not to complicate the annealing process by the introduction of incipient fusion of the metal. The samples were held at each of these temperatures for periods of one, two, four, and eight hours. If the process of recrystallization is a progressive one from the exterior in, the thickness of the recrystallized layer should be proportional to the time of annealing, and thus be easily measured.

⁵ This plating the samples with some metal such as copper renders it easy to heat small metallographic specimens—e. g., steel—without fear of oxidation or decarburization and without the use of a vacuum furnace. This is valuable in case the sample is to be examined up to the extreme surface. It also permits the use of very small specimens.

V. RESULTS

The results of the observations are tabulated in the following four tables:

TABLE 1

Series 1

[This series may be considered as typical of this bronze as prepared in commercial foundry practice. Due to surface oxidation, the measured thicknesses do not represent the true depth to which the metal was affected. Average weight of sample, 20 grams.]

Specimen ^a	Thermal treatment	Thickness of "re-crystallized" layer ^b	Amount of eutectoid ^c	Dendritic structure
		mm		
B 1	1 hour at 400° C.....	0.056	$\bar{d}$ 85	Very evident.
B 2	2 hours at 400° C.....	.076	$\bar{d}$ 82	Do.
B 3	4 hours at 400° C.....	.088	$\bar{d}$ 85.5	Do.
B 4	None.....	None	$\bar{d}$ 83.0	Do.
B 6	8 hours at 400° C.....	.07	$\bar{d}$ 76.5	The outlines of the cores are indistinct; diffusion has begun.
	Average.....	.072		
B 7	1 hour at 600° C.....	.161	41.5	Very evident.
B 8	2 hours at 600° C.....	.181	19.0	Do.
B 9	4 hours at 600° C.....	.163	12	Cores are quite faint and not continuous.
B 10	8 hours at 600° C.....	.183	6.5	Do.
	Average.....	.172		
B 11 ^e	$\frac{1}{4}$ hour at 800° C.....	.34	4.0	Dendrites are found in some grains, and are quite plainly seen in oblique illumination. ^f
B 12 ^e	1 hour at 800° C.....	.33	0.4	Very faint trace of cores under oblique illumination.
B 13 ^e	2 hours at 800° C.....	.34	0.0	All dendritic structure is erased.
B 14 ^e	4 hours at 800° C.....	.37	0.0	
	Average.....	.347		

^a In the mechanical treatment two hexagonal bars, 1 inch face, were cast in green sand. These were turned down to three-fourths inch diameter and cut into sections three-fourths inch long.

^b The thickness of the recrystallized layer in all cases was directly measured by means of a micrometer ocular.

^c To obtain an approximately quantitative expression of the rate of absorption of the eutectoid, the number of inclusions in the successive microscope fields taken contiguously along a diameter of the sample (magnification 100X) was counted and an average made. On account of the difference in size of the particles this method is a rough approximation only.

^d The eutectoid was found shattered to a depth of 0.10 mm in specimen B 1, 0.113 mm in specimen B 2 and 0.15 mm in specimen B 3, or an average for the three specimens of 0.121 mm.

^e These samples were slowly heated for two hours until 800° was reached, then held for the time indicated.

^f When the dendritic structure appears to have disappeared entirely, upon examination under vertical illumination, with oblique illumination it is still to be seen.

TABLE 2

Series 2a

[This series is to illustrate the effect of annealing upon samples cooled at different rates during the preparation; all the samples were heated to 800° C upon annealing. Average weight of sample 7 grams.]

Specimen ^a	Preparation	Mechanical treatment ^b	Thermal treatment	Thickness of "recrystallized" layer ^c	Amount of eutectoid ^d
B 29	Crucible melt, cooled in furnace	} O and D..	1 hour at 800° C.....	mm Outer 0.34 Inner .31	} 18.5
B 31	do.....		2 hours at 800° C.....	Inner .33	
B 40	Crucible melt, cooled in air	O.....	do.....	Outer .31	26.0
B 41	do.....	None.....	do.....	None observed	
B 44	Quenched at 700° C.....	D.....	do.....	Inner .37	9.1
B 48	do.....	None.....	do.....	None observed	17.0
B 32	Crucible melt, cooled in furnace	O.....	4 hours at 800° C.....	Outer .37	4.3
B 43	Crucible melt, cooled in air	None.....	8 hours at 800° C.....	None observed	1.0
B 38	Crucible melt, cooled in furnace	None.....	do.....	None observed	2.5
B 30	do.....	} O and D..	do.....	Outer .34 Inner .32	} 0.0
B 33	do.....		do.....	Outer .37	
B 39	Crucible melt, cooled in air	D.....	do.....	Inner .38	0.0
B 45	Quenched at 700° C.....	} O and D..	do.....	Outer .35 Inner .42	} 1.0
B 47	do.....		do.....	None observed	

^a No recrystallization or "twinning" was observed except in samples which were mechanically worked before annealing.

^b "O" indicates that the outer portion of the specimen was turned off; "D" indicates that the central portion was drilled out.

^c "Outer" refers to the layer of recrystallized material on the surface of the cylinder; "inner" to that surrounding the hole drilled in the specimen.

^d See note c, series 1.

TABLE 3

Series 2b

Specimen ^a	Thermal treatment	Thickness of outer layer of recrystallization ^b	Specimen ^a	Thermal treatment	Thickness of outer layer of recrystallization ^b
		mm			mm
B 35-1	2 hours at 400° C.....	^c 0.067	B 34-1	8½ hours at 600° C.....	None
B 35-2	4 hours at 400° C.....	^c 0.075	B 37-1	1 hour at 800° C.....	0.187
B 35-3	8½ hours at 400° C.....	^c 0.068	B 37-2	2 hours at 800° C.....	.231
	Average.....	.07	B 37-3	4 hours at 800° C.....	.279
B 36-1	2 hours at 600° C.....	.131	B 37-4	8 hours at 800° C ^d	.260
B 36-2	4 hours at 600° C.....	.131		Average.....	.24
B 36-3	6 hours at 600° C.....	.164	B 34-2	8 hours at 800° C.....	None
B 36-4	8½ hours at 600° C.....	.144			
	Average.....	.142			

^a In the mechanical treatment, samples 35, 36, and 37 were turned down from fifteen thirty-seconds inch to three-eighths inch. Two lathe cuts, with a fine finishing cut, were taken in each case. No. 34 was "un-worked."

^b The measured thickness of the recrystallized layer is very probably the true thickness, as there was no surface oxidation as in series 1 and 3.

^c Shattered eutectoid was detected to a depth of 0.087 mm in specimen B 35-1, 0.104 mm in specimen B 35-2, and 0.08 mm in specimen B 35-3, or an average of 0.09 mm for the three specimens.

^d Because of variation in the heating current, the temperature rose to 840° during this interval

TABLE 4

Series 3

The samples were quenched, while in the molten state from 1200° C, in cold water. No mechanical work of any kind was done upon them.]

Specimen	Thermal treatment	Amount of eutectoid ^a	"Grain size" ^b per square centimeter	Twinned crystals ^c
B 27	2 hours at 800° C.....	2.4	1835	3.2
B 28	4 hours at 800° C.....	0.0	2500	18.0
B 26	8 hours at 800° C.....	0.0	198	1.0

^a See note *d*, series 1.

^b The difference in "grain count" for Nos. 27 and 28 is most probably to be accounted for by a difference in severity of quenching. As the crucible and contents were thrown into water in each case, the rate of cooling of the metal was undoubtedly affected somewhat by the crucible.

^c The average number of twinned crystals were obtained in a manner similar to that used for expressing the amount of eutectoid. (See note *d*, series 1.)

a, B-29, series 2a, heated one hour at 800°C ; $5\times$. The recrystallized layer shows just beneath the protective copper coating on the surface and lining the central hole

b, B 35-3, series 2b, heated eight hours at 400°C ; $100\times$

c, B 36-1, series 2b, heated two hours at 600°C ; $100\times$

d, B 37-4, series 2b, heated eight hours at 800°C ; $100\times$

FIG. 3.—Samples annealed after the surface layer was distorted by machining. The cold-working of the surface layer is practically the same in *b*, *c*, and *d*, since the same depth of lathe cut was used for each; all are etched with ferric chloride

VI. DISCUSSION OF RESULTS

1. INCREASE OF CRYSTAL SIZE

It is impossible to make any direct measurements of the crystals during the process of annealing, inasmuch as such measurements necessitate the preparation of a polished plane surface for observation. The sawing, grinding, and polishing required in preparing this surface will be sufficient disturbance as to cause a recrystallization of the surface metal during the subsequent annealing. Primrose⁶ reports an increase in crystal size upon heating this bronze to 500° C, but gives no substantiating evidence. Portevin,⁷ by observing the relation of the boundary of the crystals to the interior dendritic structure in cast and annealed metals, thought he found evidence of a change in the crystal size. Robin,⁸ by observing directly the crystals of metals which had been cast upon plane surfaces in order to obtain a flat surface for observation and then held at temperatures 20 to 30° below the melting point for two to three hours, was unable to detect any measurable increase in the size of the crystal.

The interior undistorted portions of the samples examined were carefully noted for any increase of crystal size. Several "unworked" samples in series 2 *a* (Nos. 41, 48, 43, 38, and 47), which represent the various methods of cooling, were included primarily to test Portevin's statement by making use of his method of observation. No results suggesting any growth of crystals were obtained. In the specimens which were quenched from the liquid condition, however, a very noticeable crystal growth took place. (See Fig. 4, *c* and *d*.) This is to be attributed, however, to the internal stresses existing in these samples as a result of the severe quenching received. Such strained material behaves the same upon annealing as if it had been severely distorted by mechanical means.

2. PERSISTENCE OF THE DENDRITIC STRUCTURE

The process of diffusion, by which the interior dendritic pattern of the crystals is erased and the metal rendered homogeneous, is a relatively slow one. Portevin⁹ reports this inhomogeneity to

⁶ Jour. Inst. Metals, 1913, IX, No. 1, p. 162.

⁷ Rev. de Métal, 6, 1913, p. 699.

⁸ Rev. de Métal, 7, 1914, p. 494.

⁹ Rev. de Métal, 6, 1913, p. 680.

to disappear in bronze (Cu 0.95) after three hours annealing at 800°. The results tabulated in series 1 show this alloy to be very similar in its behavior. After eight hours at 600° the structure is still plainly visible (Fig. 5, *b*); and when held at 800°, following a slow heating up to this temperature, only a few degrees below the lower limit of the melting range, two hours are required for rendering the structure completely homogeneous. It was noticed that after the dendritic pattern had seemingly disappeared, as seen under vertical illumination, it was still visible in oblique light when the specimen was deeply etched with ammonium hydroxide and hydrogen peroxide. The fact that the β phase is being absorbed at the same time may account for the slowness of diffusion. Fig. 5, *b* and *c*, illustrates this persistence of the dendritic structure after prolonged annealing.

In some of the crystals the dendritic framework appears to be much more resistant than in others. This, however, is only apparent. The primary axis or trunk of the "pine-tree crystal" will naturally vary more in structure from the remainder of the crystal than do the secondary or tertiary branches, and so survive the diffusion process longest. Consequently, if the section of the grain happens to show a longitudinal section of this central branch, the persistence of this core upon heating will be much more evident than if the section of the grain shows a cross-sectional view of the same core. Grains, therefore, which show this longitudinal view of the "tree" have the appearance of being more resistant to diffusion than others. Fig 5, *b*, shows crystals illustrating this point.

The crystals, characteristic of the annealed state, appear long before the dendritic structure has disappeared. This implies that if the metal is finally perfectly homogeneous—i. e., adjoining crystals are of the same composition and structure—there must be a mutual transfer of material across the crystal boundaries during diffusion.

3. ABSORPTION OF THE EUTECTOID

The rate at which the eutectoid disappears upon heating depends much upon how the cast sample was cooled. In those specimens which passed through the solidification zone very slowly and undisturbedly (series 2a) the δ phase is dissolved much

a, Specimen B-46, series 2a, quenched just after solidifying, to preserve the β structure; 100X

b, Specimen B-25, quenched from the molten state; 100X

c, B-28, same as *b*, heated four hours at 800° C; 100X

d, B-26, same as *b*, heated eight hours at 800° C; 100X

FIG. 4

more slowly than in those which were cast in sand, and thus passed through the zone of solidification more rapidly (series 1). The difference is, most probably, largely due to the size of the eutectoid inclusions which result.

Heating at 400° for eight hours has but little effect upon the absorption, as is illustrated by series 1. Not until the α phase becomes fairly plastic is the solution of the δ phase by the α matrix rapid. One hour at 800° or six hours at 600° is sufficient for practically all of the eutectoid to disappear in those samples which did not solidify so very slowly (series 1). In series 2a four hours heating at 800° was required for the disappearance of this constituent. Fig. 5, *d*, shows the characteristic rounded forms the eutectoid assumes as part of it passes into solution; the sharp angular projections disappear first and the particles assume a spherical shape.

4. RECRYSTALLIZATION FOLLOWS ONLY DISTORTION

The observations on all the series warrant the conclusion that only after distortion is there any subsequent recrystallization of the metal upon annealing. That metal which is severely strained internally as the result of the rate of cooling behaves similarly to that which is mechanically distorted is made evident by the annealed structure of the samples of series 3. The number of "twinned" crystals is not as large as might be expected, however. In none of the other cases illustrating rapid cooling after solidification was any indication of a "recrystallization" of the metal found. The structure of the alloy immediately after quenching from the molten condition is of interest in showing that even with this rapid rate of cooling the dendritic structure is partially developed. Fig. 4, *b*, illustrates this and Fig. 4, *c*, and *d*, the structure resulting after annealing such quenched samples.

5. PROGRESS OF RECRYSTALLIZATION UPON HEATING

The uniformity of thickness of the recrystallized layer of samples heated to the same temperature is very striking. So far as the measurements taken indicate there is but little, if any, increase in thickness with prolonged annealing. The process is

not a progressive one from the exterior inward, but starts simultaneously throughout the layer, which is capable of being affected at that temperature. As the length of time of holding at a given temperature is increased the crystals grow in size, but the thickness of the layer, so far as can be seen, remains practically unchanged.

These observations are in accordance with and support of Tammann's theory of recrystallization of "cold-worked" metals.¹⁰ Briefly, this theory may be thus summarized. After distortion the metal is in a state of unstable equilibrium. A force, analogous to surface tension in the gliding planes of the lamellæ into which the crystals have been distorted, is resisted by the "strength of the crystals," i. e., their resistance to change of form. Upon heating, a state of equilibrium results; the "surface tension," if it predominates, causes the crystal fragments to agglomerate, and thus the crystals grow in size until the two forces are balanced. Thus, for any given temperature, the final grain or crystal size is a determined one. All of the observations and measurements made upon the thickness of the recrystallized layers are in support of this hypothesis.

The influence of temperature is also illustrated by this series of measurements. Up to 400° only the most severely worked metal is able to rearrange itself, and thus "recrystallize." When the distortion is very great, the brittle δ constituent of the eutectoid is shattered. Fig. 5, *a*, shows this behavior of the eutectoid. The observations of specimens annealed at 400°, given in series 1 and 2 *b*, show that the thickness of the outer recrystallized layer is well within the limit to which the eutectoid is broken up, and therefore within the limit of high distortion. The inner boundary of the layer of recrystallized metal, for 400° and 600°, is sharp and definite. At 800° this is not so marked; in this case in making the measurements the maximum depth was chosen.

Fig. 3 (*a*, *b*, *c*, and *d*) shows the appearance of this outer layer of recrystallization.

¹⁰ Tammann, *Lehrbuch der Metallographie*, pp. 74-84.

a, Bronze showing the behavior of the constituents under stress, which was applied perpendicular to the black lines (cracks) in the white constituent or eutectoid; 100X

b, B-10, series 1, heated eight hours at 600° C; 100X

c, B-12, series 1, heated two hours at 800° C, obliquely illuminated; 3X

d, B-40, series 2a, heated two hours at 800° C; 100X

FIG. 5

VII. SUMMARY

1. The metal, upon annealing, is first brought into "physico-chemical equilibrium." The dendritic structure persists until heated for approximately two hours at 800° . The absorption of the eutectoid depends much on how the cast sample cooled while solidifying; four hours' heating at 800° is required for its disappearance in those samples which solidified very slowly.

2. No evidence was found suggesting a change of crystal size of cast samples which had not been distorted in any way.

3. Recrystallization, including "twinning," was found only to follow distortion or its equivalent; metal cooled suddenly from the molten state behaves similarly because of the high internal stresses resulting. "Chilled" castings may be expected to act in a like manner upon annealing.

4. The progress of recrystallization upon annealing for different periods of time at the same temperature is in agreement with Tammann's theory of recrystallization.

5. Aside from the crystal size and shape and the modification introduced by "twinning," the end condition of material annealed directly after casting and that annealed after a preliminary distortion of the crystalline structure is the same in the two cases.

To C. P. Karr, of this Bureau, thanks are due for the material which was used in this study. The chemical analyses were made by A. B. Lort. Arthur McCabe, by the skillful and efficient aid given in the preparation of the many specimens required for the microscopic observations, has rendered the work very much less onerous and burdensome than it otherwise would have been.

WASHINGTON, July 21, 1915.