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UNIVERSITY OF ILLINOIS BULLETIN

Vol. 43

November 23, 1945

No. 21

ENGINEERING EXPERIMENT STATION
BULLETIN SERIES No. 359

GRAIN SIZES PRODUCED BY RECRYSTALLIZATION AND COALESCENCE IN COLD-ROLLED CARTRIDGE BRASS

BY

HAROLD L. WALKER



PRICE: SEVENTY CENTS

PUBLISHED BY THE UNIVERSITY OF ILLINOIS
URBANA

Published every five days. Entered as second-class matter at the post office at Urbana, Illinois, under the Act of August 24, 1912. Office of Publication, 358 Administration Building, Urbana, Illinois. Acceptance for mailing at the special rate of postage provided for in Section 1103, Act of October 3, 1917, authorized July 31, 1918.

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MINING AND METALLURGICAL ENGINEERING

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GRAIN SIZES PRODUCED BY RECRYSTALLIZATION AND COALESCENCE IN COLD-ROLLED CARTRIDGE BRASS

I. INTRODUCTION

1. *Purpose and Scope of Investigation.*—The phenomena of recrystallization of cold deformed brass are of great industrial importance, because of the role played in fabrication of the metal, and because of the dependence of physical properties upon the cold working and the annealing processes to produce recrystallization. Because of this industrial importance the subject of recrystallization has received considerable attention, and a rather voluminous amount of literature has been published. However, a great amount of the quantitative data of grain sizes of recrystallized brass is of limited value because of the failure to distinguish between grain sizes due to recrystallization and grain sizes due to coalescence subsequent to recrystallization. The main reason for the existing confusion of grain sizes produced by recrystallization is that of the three variables—(1) the degree of cold deformation, (2) the temperature of anneal, and (3) the time of anneal—the latter usually has been made constant, which results in erroneous data. If the time of anneal is made constant the grain size produced is not necessarily the grain size due to recrystallization alone, but is generally also due to coalescence which follows true recrystallization.

The specific purpose of the experiment described herein was to distinguish between the grain sizes produced by true recrystallization and those produced by coalescence. In addition it was believed that data could be collected which might help in arriving at a better understanding of the processes of recrystallization. The scope of this report is limited to describing the experiments and presenting the data on the grain sizes produced by recrystallization and by coalescence.

2. *Acknowledgments.*—This investigation has been carried on as a part of the work of the Engineering Experiment Station of the University of Illinois, of which DEAN M. L. ENGER is the director, and of the Department of Mining and Metallurgical Engineering, of which PROFESSOR H. L. WALKER is the head. W. H. BRUCKNER, Research Assistant Professor of Metallurgical Engineering, and EARL J. ECKEL and BERNARD G. RICKETTS, Associates in Metallurgical Engineering, have offered valuable suggestions during the progress of the work.

Special acknowledgment is also made to the management and technical staff of the American Brass Company, Kenosha, Wisconsin, who furnished the materials and helped in preparing the experimental specimens. FRANK A. CIBOCH, formerly a graduate student in Metallurgical Engineering, made most of the grain size measurements and hardness number determinations.

II. DISCUSSION OF RECRYSTALLIZATION PHENOMENA

Metals which have been plastically deformed by cold working are unstable and, therefore, tend to revert to a stable state. The tendency to revert to the stable state is accelerated by an increase in temperature. The processes contributing to the production of the stable state consist of three phenomena, as follows: (1) recovery, (2) recrystallization, and (3) coalescence. Of the three processes the most important is recrystallization. It is necessary to understand some of the effects of cold work upon brass before these phenomena can be understood.

3. *Effects of Cold Working Brass.*—Permanent deformation of metals at such temperatures that no recrystallization takes place during the deformation constitutes cold working. This type of deformation produces fundamental changes in both the structure and the physical properties. The microstructural changes are first noticed as slip lines in those grains in which the atomic planes are most favorably oriented for yielding under the applied stress. With an increase in stress, or increased deformation, the following phenomena are observed: The slip lines appear throughout the structure and are found in practically all the grains; the crystallographic planes, twin boundaries, and twinned sections of the crystals become curved and warped; and the grains rotate and become elongated in the direction of working. Deformation takes place on preferred atomic planes and in preferred directions with the result that preferred orientation of the atomic planes is generated, which can be described in terms of lattice symmetry, and directional physical properties are found in the deformed metal. A number of structural changes can be observed in the photomicrographs of cold-rolled cartridge brass in Fig. 1.

The process of permanent deformation affects the physical and mechanical properties by producing increases in hardness, yield strength, ultimate strength, coefficient of thermal expansion, Young's Modulus of Elasticity, and internal friction, and by an initial slight increase in density which is followed by a slight over-all decrease with

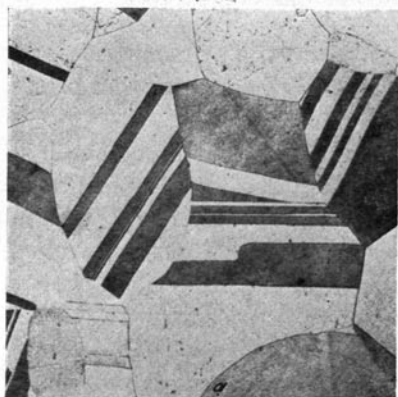
drastic deformation. There is a decrease in ductility (elongation and reduction of area), impact strength, formability in drawing and pressing operations, and electrical conductivity.

In addition to these changes the following is also observed. If a thermocouple is constructed from two wires, one in the hard-drawn and the other in the annealed state, the potential difference between them can be measured. Normally, the current flows from the worked to the annealed metal at the cold junction. However, this is not always the case, for in some metals the worked metal is positive with respect to the annealed metal and in other metals it is negative. The rate of solution of cold-deformed metal in dilute acids is considerably greater than for the same metal in the annealed state. If two pieces of a metal, one in the cold-deformed and the other in the annealed condition, are placed in a solution of one of the salts of the metal to form a cell, the cold-worked metal is found to be electronegative toward the annealed metal and will tend to dissolve first.

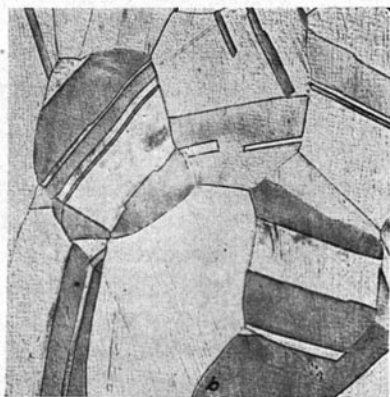
The X-ray reflections from cold-deformed metals differ in several respects from those of undeformed metals. The following changes are observed:

- (1) Changes in the spacings of the crystal planes.
- (2) Changes in the character of the reflections:
 - (a) Elongation of the Laue spots
 - (b) Spreading of the angle over which characteristic X-rays are reflected, and an increase in the size of the reflected spots.
- (3) Broadening of Debye-Scherrer rings, and loss of resolution of the alpha doublet.
- (4) Changes in the intensity of reflection, which may affect all reflections or only some of them.
- (5) Preferred orientation of the crystallographic planes and crystallographic directions is exhibited with increased amounts of plastic deformation. The X-ray patterns show arcs, or localized intensity maxima, superimposed on the usual Debye-Scherrer rings.

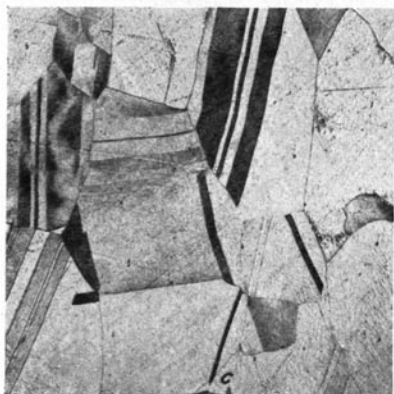
The net result of the plastic deformation is to create a system of complex internal stresses of a non-homogeneous character. These complex stresses are of two types, "macro" and "micro." The macro stresses affect the metal as a whole, and in such a manner that one section may be in compression and another section may be in tension. The micro stresses are localized in very small regions and result from slip on the atom planes, bending of the slip planes, and resistance to



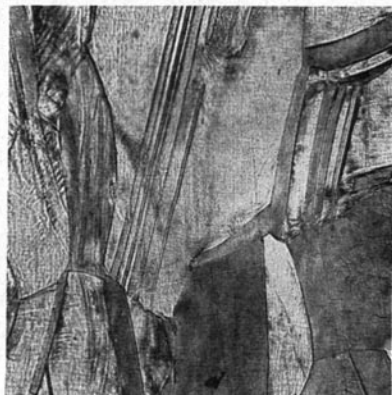
(a)



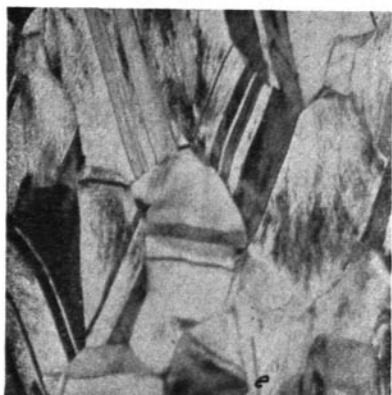
(b)



(c)



(d)



(e)



(f)

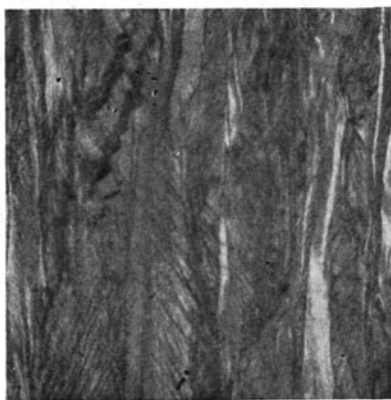
FIG. 1. MICROSTRUCTURE OF COLD-ROLLED CARTRIDGE BRASS
Specimens etched in $\text{NH}_4\text{OH} - \text{H}_2\text{O}_2$. Magnification $40\times$.



(g)



(h)



(i)

(a) Cold rolled 5.3 per cent. The only evidence of deformation is slight bending of some of the annealing twins.

(b) Cold rolled 10.0 per cent. Slightly greater amount of bending in the twins. Deformation lines are not yet visible.

(c) Cold rolled 15.0 per cent. The first deformation lines have begun to appear at the grain boundaries and at the contact surface between twins and the parent grain.

(d) Cold rolled 20.9 per cent. Deformation lines are concentrated at the grain boundaries and the edges of twins.

(e) Cold rolled 28.0 per cent. Complex slip is shown by intersecting deformation lines. The twins which lie at the greatest angles to the direction of rolling show the greatest amount of bending and warping.

(f) Cold rolled 42.8 per cent. The grains are elongated in the direction of rolling and the twins are rotating to a direction parallel to the direction of rolling.

(g) Cold rolled 50.4 per cent. All grains are now elongated in direction of rolling and well fragmented.

(h) Cold rolled 60.4 per cent. The former equiaxed grains have been elongated and reduced in section size until they appear only as narrow bands.

(i) Cold rolled 70.0 per cent. The grains are well fragmented. The zig-zag line running obliquely to the direction of rolling indicates failure may have begun.

FIG. 1. MICROSTRUCTURE OF COLD-ROLLED CARTRIDGE BRASS

Specimens etched in $\text{NH}_4\text{OH} - \text{H}_2\text{O}_2$. Magnification $40\times$.

slip as offered by grain and twinning boundaries. The distribution of these microstresses results in localized regions of high energy and of low energy. The points of highest energy constitute the regions of greatest instability in the deformed metal, and it is about these points that we may expect the first relief from the effects of plastic deformation to take place.

4. *Recovery*.—Recovery represents a change in the properties, particularly those determined by internal stresses, or micro stresses, of a cold-deformed metal which has been annealed at low temperatures without producing any change in the microstructure. Therefore recovery precedes recrystallization in any annealing cycle. The rate of recovery increases with increase in annealing temperature, and may take place so rapidly, at the higher temperature, as to be undetected in some annealing cycles. At low temperatures the course of recovery may be followed by plotting such properties as hardness versus time at a number of given temperatures. The usual trend of hardness during the process of recovery is that of a gradual decrease of the hardness curve; this is particularly so with high purity metals and alloys. However, in some metals and alloys containing small amounts of impurities in a non-equilibrium condition at the given temperature, the impurities may be precipitated simultaneously with the relief of the internal stress, and the hardness may show a slight increase during the recovery period. In the latter case the effects of precipitation hardening are greater than the effects of stress relief, and the effects of annealing on stress relief are masked out.

The effects of annealing at temperatures below which recrystallization takes place may also be detected in the stress-strain curve. If a specimen of brass is elongated by applying a load in tension, the elongation is characterized by a rapidly and continually rising stress-strain curve. If the load is removed and immediately reapplied, the second application of stress rises to and continues to rise beyond the point of interruption of the first cycle of stress; however, if the specimen is annealed at a temperature below the recrystallization temperature, appreciable yielding begins at a low value. Thus, a portion of the initial strain hardening has been lost at the temperature and time of anneal, and yet no recrystallization has taken place. The effects of recovery are also detected in decreasing spring back under the influence of both time and temperature. The phenomenon of "season cracking" in brass is mitigated by a low temperature recovery anneal, and this treatment is regularly applied in industry to prevent this phe-

nomenon in cartridge brass. The magnitude of macro stresses is considerably decreased, as evidenced by the partial return of elastic properties to a condition intermediate between the cold-worked condition and the fully annealed condition and the decreased tendency to crack with further working.

Complete stress relief is not attained by recovery, but is attained by recrystallization. The mechanism of the phenomenon of recovery is not understood, and probably will not be understood until the processes involved in plastic deformation are better defined. Recovery undoubtedly affects the elastic properties through the partial elimination of internal stresses. The partial elimination of internal stresses also decreases the internal friction, the electrical resistance and the velocity of solution in acids, but has little effect on the strength and ductility properties. The resolution of the alpha doublet in X-ray reflections is regained, at least in part, during recovery anneals at temperatures below recrystallization.

The perfection of atom disposition on a space lattice is grossly distorted during plastic deformation; the atomic planes become twisted and distorted, and the atoms no longer occupy precision positions on a space lattice but are distorted to positions less symmetrically disposed. This distortion results in an increase in the internal energy of the metal, and a somewhat localized distribution of the energy. The partial stress removal during recovery is probably associated with a partial rearrangement of the space lattice, straightening of slip planes, and return of atoms to positions more closely approximating the unstrained space lattice. These processes are brought about by the influence of heat, which permits a greater thermal vibration of the atoms with resultant diffusion or movement of atoms to satisfy partially the trend toward returning to the stable condition. The influence of heat, however, is not great enough to permit a complete rearrangement of the atoms on the space lattice, and the complete relief of stress is not obtained.

Internal friction is defined as the capacity of a solid to damp out elastic vibrations. Vibrational energy is absorbed quickly if the internal friction is high, and absorbed slowly if the internal friction is low. Internal friction is the sum of a number of contributing phenomena, such as (a) those found only in ferromagnetic materials and concerned only with the magnetic-elastic theory, (b) results of heat flowing back and forth from point to point during stressing, and (c) sources related to localized plastic flow in the solid as a result of

applied stress. Norton^{1*} and Zener^{2, 3, 4} have contributed much information concerning the damping capacity of alpha brass. Norton shows that specific damping (the ratio of the energy loss per cycle to the elastic potential energy at the maximum amplitude of the cycle) is decreased continuously with increased temperature when cold-worked brass is annealed, and that damping reaches a minimum before the onset of recrystallization. The reduction of damping capacity at low temperatures indicates the relief of internal stresses during the recovery period.

5. *Recrystallization*.—If a metal or alloy, which has been plastically deformed at such a temperature as to constitute cold work, is subsequently heated with the proper balance between time and temperature, new grains will appear and grow until ultimately new unstrained grains will have replaced the old strained grains. Thus, recrystallization consists of nucleation and replacement of strained metal with new and unstrained grains of the same metal. As soon as the replacement of strained metal has been completed, the process of recrystallization is complete, and any subsequent grain growth is due to coalescence. It should be noted that coalescence may be taking place simultaneously with recrystallization, and this process will be discussed under a later heading. The changes in physical properties during recrystallization are diametrically opposed to the changes taking place during cold deformation, and the properties tend to revert to the same values possessed by the metal prior to the cold deformation. The properties after recrystallization are not necessarily the same as original properties but depend upon the difference in the grain size of the two conditions. The changes in physical properties produced during recrystallization are evidence that the process is or has been taking place; however, the changes in themselves do not critically define the beginning and ending of recrystallization.

The recrystallization temperature has been defined as “the minimum temperature at which new grains may be observed under a high-powered microscope.” Such a definition is unsatisfactory because it defines the temperature at which recrystallization begins and not that when it is completed. It is entirely possible that nucleation of new grains could be observed under the microscope, and further annealing for an almost infinite length of time would not complete the process of recrystallization. A comprehensive definition of recrystallization

* Index numbers refer to bibliography on page 55.

temperature cannot be given because of its dependence upon a number of variables. In this report the recrystallization temperature is defined as the minimum temperature at which cold-worked and strained metal is just completely replaced by unstrained grains after annealing a metal or alloy with a given degree of cold deformation for a given fixed time.

The recrystallization temperature of a given metal or alloy depends upon a number of variables and is increased by the following:

(1) *Decreased degree of strain hardening.* The degree of strain hardening is often spoken of in terms of per cent reduction of area during cold working. A decrease in strain hardening probably reduces the energy level of points about which nucleation begins, and it reduces the number of points about which nucleation takes place, as is shown by the dependence of rate of nucleation upon the degree of cold working.

(2) *Decreased time at the annealing temperature.* Recrystallization is not an instantaneous process and is clearly dependent upon time. The amount of diffusion (or movement) of atoms within a solid is a function of both time and temperature. Likewise the rate at which atoms may give up their energy of strain hardening and attach themselves to (or form) an unstrained lattice is a function of the temperature. At the highest temperatures recrystallization takes place so rapidly that it approaches an instantaneous process. Even at the highest rates of heating, for the higher temperatures, recrystallization may be complete before the proposed temperature of annealing is reached.

(3) *Increased temperature at which cold deformation takes place.* An increase in temperature at which deformation takes place decreases the energy of strain hardening (probably by a process of partial recovery) and thus acts in the same direction as a decrease in the amount of deformation.

(4) *Increased grain size prior to cold deformation.* The greater (larger) the initial grain size, the less the energy needed to deform the metal to a given degree of deformation, and this results in a lower energy of strain hardening and thus requires a higher temperature for recrystallization.

(5) *Increased quantity of impurities.* The greater the purity of a metal, the lower the recrystallization temperature. Thus, alloys have higher recrystallization temperatures (in all clearly established cases) than their component metals. The impurities may or may not be in

solid solution. The presence of foreign atoms (substitutional or interstitial type) in the lattice of an element restricts the movement of the solvent atoms to such an extent that an increase in temperature is necessary (at least for a given time) to complete the recrystallization process. This principle is utilized in the manufacture of cold-rolled copper fins for automobile radiators. Lake copper contains small amounts of silver, the presence of which increases the temperature of recrystallization sufficiently to permit soldering of the tubes without destroying the effects of cold rolling. If the impurities are not in solid solution they may also act as obstructions to the movement of the atoms, particularly if they are found in a very fine state of subdivision. Impurities of the latter type may be considerably more effective in increasing the recrystallization temperature than if they were in solid solution. This principle is utilized in certain types of aluminum alloys to increase the temperature of softening.

6. *Nucleation.*—Nucleation of unstrained grains from strain-hardened grains has been studied by a number of investigators and has been the subject of several papers. A completely satisfactory theory has not been advanced. The mechanism of nucleation is complicated because there is no phase change involved, such as solidification from the liquid state or precipitation of a new solid phase from another solid phase (precipitation hardening).

Recrystallization consists of nucleation of strain-free grains and the growth of these strain-free nuclei by feeding upon other strained grains. In order that a nucleus may form there must exist a difference between the metal at that point and the metal surrounding it. The difference is apparently created as the result of permanent plastic deformation, because a strain-free metal does not recrystallize upon heating. (Recrystallization by allotropic modification is another type.) Since permanent deformation is a prerequisite of recrystallization we may assume two cases for the establishment of strain-free nuclei:

Case 1. The points about which nuclei form are the result of drastic deformation. These points are highly stressed, and the energy of deformation retained by these points is considerably greater than the retained energy of the metal surrounding the points.

Case 2. The points about which nuclei form have not undergone deformation and therefore represent strain-free areas.

Most theories of nucleation start with the assumption that the process of permanent plastic deformation generates a pattern of

stresses characterized by localized points of extraordinarily high energy peaks (case 1). These high energy points are the least stable, and during an annealing cycle are the first points to be relieved of the effects of cold deformation. These points, on being relieved, return to the more stable state, and constitute the nuclei about which recrystallization takes place.

The process of the commencement of recrystallization is illustrated by sketches in Fig. 2. Figure 2(a) represents a plastically deformed brass of approximately 20 to 30 per cent deformation. The grains have begun to rotate and elongate in the direction of working, the twin bands are curved and warped, deformation lines are shown, and in some areas there is complex deformation. Relative values for the energy resulting from plastic deformation have been assigned to various areas in Fig. 2(a), and because the deformation is only moderate there are no energy values above 80. If the sample of brass is annealed at a given temperature and for a sufficient time for nucleation to take place without appreciable recrystallization, it is found that only the energy values above 60 are sufficiently unstable to give up their energy of cold deformation, reorient themselves, and begin to form equiaxed grains essentially free from stresses. This condition is sketched in Fig. 2(b), where new grains will be found about the areas of highest energy content within the grains and at the grain boundaries. There are also shown the remaining deformation lines which did not have sufficient energy to nucleate new stress-free grains. If this specimen were to be reheated to the given temperature and held there for a sufficiently long time, the remaining cold deformed metal would disappear by a process of grain growth of existing nuclei and by appearance of new nuclei and their growth. It is evident that some coalescence will take place coincident with recrystallization.

The exact mechanism of nucleation is not understood, but such a process as outlined must form a portion of the *modus operandi*. The results of many researches have demonstrated that (1) the rate of nucleation and the number of nuclei formed are increased by an increase in deformation; (2) The linear speed of recrystallization is increased by an increase in deformation and in temperature.

To return to the second possibility for the establishment of centers of recrystallization (case 2), Professor A. E. van Arkel⁵ in a series of experiments with aluminum concludes "that the centers of recrystallization are the undeformed particles." Professor van Arkel made two small incisions in a slab of rolled aluminum and then elongated the

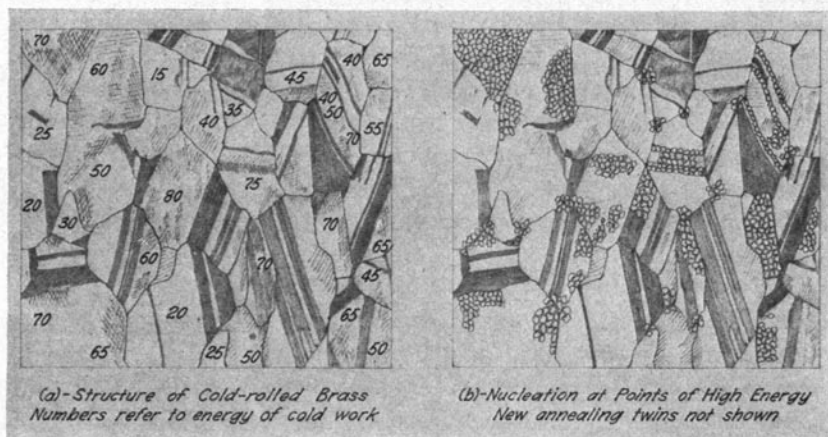


FIG. 2. SKETCHES SHOWING BEGINNING OF RECRYSTALLIZATION
IN COLD-WORKED BRASS

slab very slightly by placing it in tension. Upon heating, nuclei were established exclusively at the corners of the incisions but heating was stopped when the recrystallized grains were a few millimeters in diameter and before recrystallization was complete over the entire slab. The slab was again elongated a small amount and reheated. During the second heating new centers of recrystallization developed which finally replaced the entire volume of the metal with the exception of the parts occupied by the large recrystallized crystals already formed after the first deformation.

Professor van Arkel also concludes that the centers of recrystallization are neither deformed nor intact. He utilizes the hypothesis of M. von Liempt⁶ and an experiment⁷ on the transformation of undeformed and deformed alpha iron to gamma iron to prove the validity of his statement.

Contrary to quite popular belief, the recrystallization of cold-deformed metal with its complex pattern of stresses and preferred orientations is not necessarily evidence of the production of a random or haphazard orientation of recrystallized grains. Recrystallized grains often exhibit preferred orientations and directional properties. The resulting orientation may be identical with, similar to, or entirely different from the orientation found in the cold-worked metal. The presence of the preferred orientations may be verified with the aid of pole figures derived from X-ray diffraction patterns. In some cases the

directionality is apparent under the microscope from the frequency of the twin-band directions in certain preferred directions.

The presence of preferred orientations of the grains in recrystallized metal is detrimental in certain types of work, such as deep drawing operations, since the presence of the directional properties produces ears (fluted edge) and results in prohibitive trimming of the rim of the drawn articles. Relatively little is known concerning the inheritance of preferred orientations in recrystallized metal; however, Burgers⁸ and van Arkel⁵ have conducted experiments with aluminum to demonstrate the dependence of the number of nuclei established, of the orientation of the prior-deformed metal, and of the limitations of grain growth by coalescence upon the mechanism of simple and complex glide processes in the production of "weak and strong local curvatures" of the lattice. Van Arkel demonstrated that the coalesced grain does not transgress upon the domain of other grains existing at the time deformation took place. There is rather definite proof that nuclei are established as the result of slip on definite crystallographic planes; therefore, one would expect to find a similarity in the orientation of nuclei established within a single grain. The similarity in orientation of new grains within the boundaries of old grains would greatly accelerate the process of coalescence, since major readjustments of atoms attaching themselves to the growing grain would not be necessary. There is perhaps a correlation in the directional properties of recrystallized metal with the kind and amount of coalescence following the recrystallization process. Palmer and Smith⁹ have made recommendations for the rolling and annealing schedules to produce brass strip free of directional properties.

7. *Coalescence*.—In this report coalescence is defined as the process of grain growth by the absorption of unstrained grains by other unstrained grains. Coalescence must be preceded by recrystallization, because the grains must be free of strain before they are permitted to grow by coalescence. The driving force for coalescence is believed to be the difference in surface energy of the differently sized grains. The ratio of surface to mass decreases with an increase in grain size and with the consequent closer approach in shape of the polyhedral grains to a sphere. An increase in grain size results in decreased surface energy or, conversely, the stability of the grains is increased with increased grain size. Since stability is increased with an increase in grain size, the larger grains feed upon the smaller grains during the process

of coalescence, and one grain is growing larger while the other grain is decreasing in size. Coalescence takes place by grain boundary migration, the atoms of the smaller grains attaching themselves to the lattice of the larger grains and assuming the orientation of the absorbing grains. Thus, coalescence of solids differs from the coalescence of very fluid liquids, but the end product is the same, i.e., greater stability through increase in size.

The velocity of coalescence is very slow as compared with the velocity of recrystallization, because the energy exchange is considerably smaller. The velocity of coalescence is greatest immediately after the completion of recrystallization, because of grain size contrast, and coalescence thereafter exhibits a decreasing rate of growth as the individual grains approach a uniform size and equal energy content. A small amount of coalescence is produced during long periods of time at the annealing temperature; however, for all practical purposes it ceases within a reasonable length of time.

In the preceding discussion it has been assumed that the grains formed by recrystallization have a regular and undeformed crystal lattice, and that the surface energy is the driving force for coalescence. This is not in accordance with the experiments of Dehlinger¹⁰, who finds that the energy of the grains is not localized on the surface of the grains but in the crystalline lattice. Dehlinger reports that he has found the atomic distance for crystals formed by recrystallization to be a little greater than for crystals formed from the molten metal. According to Dehlinger, the source of energy for coalescence (secondary recrystallization) is different from surface energy.

There is also some experimental evidence to indicate that coalescence is a function of the orientation of the individual grains. According to this view, the amount of coalescence is limited to grains having an orientation very close to that of the growing grain. In this case grain growth by coalescence is limited to certain domains having similar orientation, and one grain cannot grow at the expense of another grain in another domain of widely different orientation.

Van Arkel⁵ describes experiments with a specimen of large-grained aluminum. The boundaries of the large grains were outlined with ink and photographed. The specimen was then cold deformed, this treatment being followed by annealing for recrystallization. The recrystallized specimen was entirely in the fine-grained state, yet the regions formerly occupied by the large crystals could be clearly discerned by the differences in etching characteristics. Continued heating

of the specimen produced coalescence of the small grains into large grains, which then occupied the exact regions formerly occupied prior to cold deformation.

The grain size due to coalescence is increased by (1) increased temperature of anneal, (2) increased time of anneal, and (3) decreased amount of foreign obstruction material. These laws of coalescence are dependent upon the kind and composition of metal.

III. DESCRIPTION OF APPARATUS AND MATERIALS

In this experiment the variables—(1) mass, (2) heating rate, (3) kind and composition of metal, (4) initial grain size, and (5) temperature of cold deformation—were made as constant as possible. The brass used was American Brass Company Alloy No. 24. The experimental brass was prepared in the Kenosha, Wisconsin, mill of the American Brass Company in the regular production line of rolls, and with regular production furnaces. Pertinent information concerning the experimental brass is as follows:

8. *Analysis of Brass.*—The brass was produced from virgin copper and zinc in a low-frequency induction-heating type of furnace. The analyses were made in the chemical laboratories of the American Brass Company. Three bars 4 in. x 9 in. x 36 in. of Alloy No. 24 were used in preparing the samples. These bars were marked A, B, and C, and the analyses were as follows:

	COPPER	ZINC	LEAD	IRON
Bar A.....	70.08	29.91	none	0.01
Bar B.....	70.38	29.60	0.01	0.01
Bar C.....	70.04	29.94	0.01	0.01

9. *Rolling Schedule.*—The three bars were heated in an air furnace to about 730 deg. C. and hot rolled to a thickness of 1.142 to 1.170 in. Each of the bars received eight passes through the rolls; the first two passes were straight through, and the remaining six passes were quarter cross rolled. The bars were cooled to room temperature, in air, and then cold rolled.

Bars A and B were cold rolled in seven passes to a thickness of 0.536 in., representing a cold reduction of 53 to 54 per cent. At least one reversal of 180 degrees (turned end for end) was made in the last three passes.

Bar C was cold rolled in four passes, without reversing, to a thickness of 0.652 in., representing a cold reduction of 43 to 44 per cent. Bar C was not given as great a reduction initially in order that the second cold rolling operation would approximate 35 per cent reduction and not interfere with subsequent annealing.

Following the cold-rolling operation the bars were annealed at 650 deg. C., and each bar was cut into three pieces, the pieces being numbered as follows:

Bar A....A1, A2, A3
 Bar B....B4, B5, B6
 Bar C....C7, C8, C9

Samples were taken of the different bars to determine the grain sizes, which were as follows:

Bar A....0.090 mm. av. diameter
 Bar B....0.091 mm. av. diameter
 Bar C....0.091 mm. av. diameter

Following the annealing operation the bars were scalped. The scalping operation removed approximately equal amounts of metal from the surface as follows:

A1, A2, A3, B4, B5, B6 scalped from 0.536 to 0.497 inch.
 C7, C8, C9 scalped from 0.652 to 0.620 inch.

After scalping (milling) the surfaces, the bars were cold rolled as follows:

	A1	A2	A3	B4	B5	B6	C7	C8	C9
1st pass.....	0.363	0.363	0.363	0.363	0.363	0.363	0.486	0.486	0.486
2nd pass.....	0.273	0.273	0.273	0.273	0.273	0.273	0.440	0.440	0.440
3rd pass.....	0.214	0.214	0.214	0.214	0.222	0.236	0.4205	0.4205	0.4205f
4th pass.....	0.175	0.175	0.178	0.187	0.197	0.217	0.346	0.346	
5th pass.....	0.145	0.151	0.159	0.166	0.184	0.216	0.313	0.313f	
6th pass.....	0.137	0.150	0.147f	0.165	0.175f	0.215f	0.278		
7th pass.....	0.132f*	0.148		0.158f			0.252f		
8th pass.....		0.140f							
Per cent reduction.....	73.4	71.8	70.4	68.2	64.8	56.8	59.4	49.6	32.2

The object of the rolling schedule was to produce specimens of such thickness that the final rolling schedule would produce approximately a constant thickness of specimen (constant mass) with a cold deformation range of approximately 5 per cent to 70 per cent in precalculated steps.

*f = final size.

Following this cold-rolling treatment all bars were annealed overnight at 750 deg. C. Samples were taken of all bars to determine prior grain sizes, which were found to be as follows:

BAR No.	GRAIN SIZE	AVERAGE
	Av. Diam. mm.	Rockwell H Hardness
A1.....	0.51	75.0
A2.....	0.51	76.5
A3.....	0.55	77.7
B4.....	0.57	78.7
B5.....	0.52	79.0
B6.....	0.53	80.5
C7.....	0.48	78.3
C8.....	0.54	78.7
C9.....	0.54	74.0

After annealing, the bars were pickled and cold rolled to final size as follows:

	A1	A2	A3	B4	B5	B6	C7	C8	C9
1st pass.....	0.125f*	0.130	0.133	0.133	0.137	0.187	0.240	0.300	0.375
2nd pass.....		0.126f	0.125f	0.125f	0.126f	0.170	0.225	0.285	0.347
3rd pass.....						0.155	0.205	0.265	0.330
4th pass.....						0.135	0.190	0.245	0.305
5th pass.....						0.123f	0.170	0.230	0.287
6th pass.....							0.150	0.215	0.265
7th pass.....							0.135	0.200	0.245
8th pass.....							0.125f	0.185	0.225
9th pass.....								0.171	0.200
10th pass.....								0.160	0.180
11th pass.....								0.150	0.160
12th pass.....								0.140	0.140
13th pass.....								0.131	0.130
14th pass.....								0.124f	0.126f
Percent reduc- tion.....	5.3	10.0	15.0	20.9	28.0	42.8	50.4	60.4	70.0

During this rolling schedule the slabs were rolled flat until the final pass when they were coiled. During rolling, the material was occasionally turned end for end according to the roller's judgment. At the completion of rolling, the sheets were approximately 10 in. wide. These sheets were passed through a slitler which made three cuts from one edge of the sheet. The flats were each $\frac{13}{16}$ in. wide. The outside flat was discarded and the two inside flats were coiled, and constitute the material upon which the research is based.

*f = final size of rolling.

The final cold working listed in the foregoing produced a series of flats of approximately equal thickness and varying amounts of cold deformation. The percentage of cold deformation for each of the specimens was as follows:

PERCENTAGE DEFORMATION			
A1.....	5.3	B4.....20.9	C7.....50.4
A2.....	10.0	B5.....28.0	C8.....60.4
A3.....	15.0	B6.....42.8	C9.....70.0

10. *Method of Annealing.*—The rate of heating for annealing at the higher temperatures and for the higher degrees of deformation has some effect upon the results of annealing, because recrystallization may be completed before the annealing temperature is reached when the rate of heating is slow. To provide a rapid rate of heating to the temperature of annealing, a bath of molten lead was used for all times and temperatures of 400 deg. C. and above. A lead-bismuth bath was used for temperatures of 300 and 350 deg. C. and for times of anneal up to 24 hours. The brass specimens were washed with lime to prevent solution of the brass in the molten lead and to prevent diffusion of the lead into the brass. For annealing times above 24 hours the brass specimens were placed between two copper ingots, weighing ten pounds each, which had been placed in the muffle furnace and allowed to come to the temperature before charging the specimens. The temperature of the molten baths and of the muffle furnaces was controlled with Micro-max Indicating Potentiometer Controllers.

11. *Determination of Heating Rates.*—The time required to reach a predetermined temperature of anneal was determined with a High Speed Celestray Potentiometer Temperature Recorder. The instrument has a reaction rate of 0 to 3000 deg. F. in two seconds, and the recording chart moves at the rate of 12 in. per min. The thermocouple wires were platinum—platinum-10-per-cent-rhodium of number 40 B & S gauge. The hot junction of the thermocouple was placed at the midpoint of the specimen (0.063 in. below the surface) and held in place by welding a small hollow brass tube to the upper surface of the brass specimen. The thermocouple and its protecting tube were inserted through the small brass tube and held in contact with the specimen at the midpoint. To determine the heating rate the specimen was placed in the molten lead bath, and the time to reach the temperature of the bath was recorded on the high speed instrument. Figure 3 shows the curve for the heating rate of the specimens.

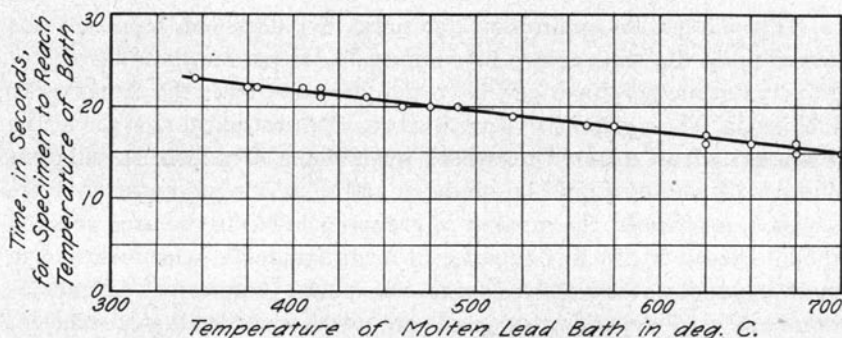


FIG. 3. HEATING RATE IN MOLTEN BATH

For the shorter periods of annealing in the lead bath, the specimen was placed in the molten lead and permitted to remain for the length of time required to reach temperature. The time of annealing recorded was the time the specimen remained in the bath *after reaching the annealing temperature*. It was found at the higher temperatures, for the greater amounts of deformation, that recrystallization was complete at zero time, indicating that recrystallization was completed on reaching the temperature of anneal. To prevent heat effects during cooling from the annealing temperature, the specimens were quenched in cold water at the conclusion of the time at temperature.

12. *Method of Determining Grain Size.*—All grain size determinations were made in accordance with Jeffries' method for grain size measurements¹¹. Briefly, this method consists of counting the number of grains entirely included in a circle of 79.8 mm. diameter (5000 sq. mm. area) and adding one half the number of grains intercepted by the circumference of the circle. The average grain diameter may be calculated from the following formula:

$$d = \frac{1}{\sqrt{\frac{m^2}{5000} \times (\frac{1}{2}W + Z)}}$$

where m = magnification used

W = number of boundary grains

Z = number of completely included grains

d = average diameter of grains in mm.

This method of grain size determination does not represent the actual grain diameters since it is impossible to pass a plane through a polycrystalline specimen and have the plane intersect the diameter of each grain. Thus, grain size determination is a statistical average of the diameters of all grains intersected by a plane of polish. A sufficient number of counts should be made to arrive at a representative average and, if possible, the number of grains included in the area counted should exceed 50. In this experiment a minimum of six counts, and in most instances at least ten counts, was made. A range of magnifications was used, depending upon the grain size, in order that a sufficient number of grains would be included in the area. In the case of the larger grain sizes it was not always possible to include the optimum number of grains because the thickness of the specimens limited the magnification that could be used with the area of 5000 sq. mm. still filled. This limitation resulted in a greater spread of the grain sizes found for the large grains than was found in the smaller grain sizes, as will be noted in Fig. 7 and Table 10.

13. *Hardness Measurements.*—Rockwell B hardness numbers were used to detect the change in hardness by cold work but, because of the range of values to be found in Rockwell B hardness numbers of cold-rolled and annealed brass, the Rockwell H scale was used, to follow the course of softening by annealing, in this experiment. The Rockwell H hardness numbers are the result of a 60-kg. load and a $\frac{1}{8}$ -in. penetrator. The Rockwell H hardness numbers range from 69 to 119 for the annealed and cold-rolled conditions. Numbers above 100 are not recommended for hardness measurements; however, the hardness numbers in this experiment were used for control purposes in following the effects of recrystallization and are relative only. For this purpose the Rockwell H scale served best.

IV. EXPERIMENTAL PROCEDURE

Specimens approximately one inch long were cut from the coils of the $\frac{13}{16}$ -in. flats of various degrees of deformation. When annealing in liquid lead, a bundle of specimens consisting of all nine degrees of deformation was submerged in the lead for the predetermined time. The time of anneal represents the time at temperature and does not include the time required to reach temperature. To prevent heat effects during the cooling cycle the specimens were quenched in water. The hardness of the specimens was determined on the Rockwell Hardness Tester, using the H scale. Longitudinal sections sufficiently far re-

moved from the edge to prevent interference from cold-working effects of the slitler were prepared for microscopic observation to determine the extent of recrystallization. The course of recrystallization was followed by observing changes in Rockwell H hardness numbers and in microstructures until all evidence of strained metal had disappeared. The criterion for judging the existence of strained metal was the presence of deformation lines, curved twin bands, and etching characteristics. Strained metal does not react in the same way to the etching reagent as unstrained metal, though the difference may not be immediately apparent to the inexperienced eye. The etchant used was a solution of ammonium hydroxide and hydrogen peroxide. Grain size measurements were made upon all specimens exhibiting complete recrystallization.

V. EXPERIMENTAL RESULTS

The initial slabs, after breaking down, cold rolling to approximately $\frac{1}{2}$ in., and annealing at 650 deg. C., had the following average grain diameters in millimeters:

	BAR	GRAIN SIZE Av. Diam. mm.
A.....		0.090
B.....		0.091
C.....		0.091

The bars were then scalped and cold rolled. The percentage reduction by cold rolling for each of the bars was as follows:

BAR	REDUCTION Per Cent	BAR	REDUCTION Per Cent
A1.....	73.4	B6.....	56.8
A2.....	71.8	C7.....	59.4
A3.....	70.4	C8.....	49.6
B4.....	68.2	C9.....	32.2
B5.....	64.8		

The bars were then annealed at 750 deg. C., the treatment producing the following grain sizes and hardnesses. These specimens represent the material upon which the final rolling was done to produce the specimens for the experimental work.

BAR	GRAIN SIZE Av. Diam. mm.	AVERAGE Rockwell H Hardness	BAR	GRAIN SIZE Av. Diam. mm.	AVERAGE Rockwell H Hardness
A1.....	0.51	75.0	B6.....	0.53	80.5
A2.....	0.51	76.5	C7.....	0.48	78.3
A3.....	0.55	77.7	C8.....	0.54	78.7
B4.....	0.57	78.7	C9.....	0.54	74.0
B5.....	0.52	79.0			

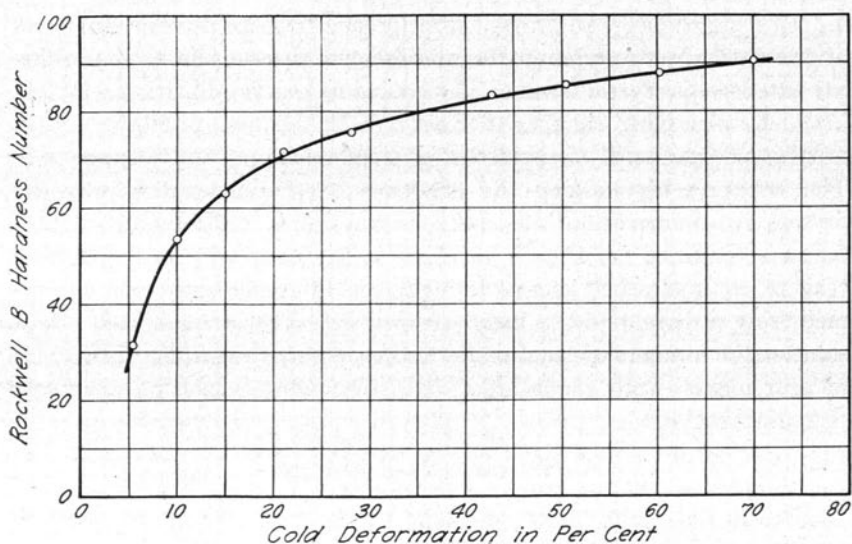


FIG. 4. ROCKWELL B HARDNESS OF COLD-ROLLED CARTRIDGE BRASS

The final cold-rolling schedule produced the following percentage reduction in thickness, and Rockwell B Hardness numbers:

AVERAGE			AVERAGE		
BAR	REDUCTION Per cent	Rockwell B Hardness	BAR	REDUCTION Per cent	Rockwell B Hardness
A1.....	5.3	31.3	B6.....	42.8	83.6
A2.....	10.0	53.5	C7.....	50.4	85.5
A3.....	15.0	63.0	C8.....	60.4	88.1
B4.....	20.9	71.6	C9.....	70.0	90.5
B5.....	28.0	75.8			

In Fig. 4 the percentage cold deformation is plotted against the Rockwell B hardness. The Rockwell hardness values rise very rapidly up to a deformation of 21 per cent, but thereafter the increase is slow. The increase in hardness amounts to 40 Rockwell B numbers when the deformation is increased from 5 per cent to 21 per cent, but thereafter the increase is slow, amounting to only 19 Rockwell B numbers when the deformation is further increased from 21 per cent to 70 per cent.

The grain sizes and Rockwell H hardness numbers produced by annealing the brass specimens with different degrees of cold deformation, for varying lengths of time, and at temperatures of 300 to 700 deg. C., are shown in Tables 1 to 9 inclusive. The blank spaces in the grain size column of Tables 1 to 9 indicate that microscopic ex-

TABLE I
ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS IN MILLIMETERS FOR VARIABLE DEFORMATION
AND TIME AT 700 DEG. C. ANNEAL

Time	Percentage Deformation																	
	5.3		10.0		15.0		20.9		28.0		42.8		50.4		60.4		70.0	
	R _H	GS	R _H	GS	R _H	GS	R _H	GS*	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.8	119.2
0s.....	89.8	83.4	83.5	85.1	86.7	89.9	90.6	91.9	93.0
5s.....	88.8	79.2	82.8	83.3	0.090	85.7	0.080	89.6	0.061	89.5	0.040	90.2	0.040	90.1	0.041
15s.....	86.5	79.5	0.155	82.3	0.108	83.0	0.092	85.1	0.089	89.0	0.082	88.4	0.050	88.2	0.056	89.9	0.052
30s.....	84.8	79.8	0.165	81.0	0.124	81.8	0.101	82.3	0.098	84.6	0.086	86.5	0.076	85.8	0.072	85.6	0.063
1m.....	77.0	0.242	77.5	0.204	79.1	0.158	79.8	0.105	81.3	0.105	82.8	0.088	84.0	0.087	83.4	0.081	85.3	0.086
2m.....	75.5	0.274	76.6	0.245	78.3	0.190	79.9	0.115	79.4	0.125	81.1	0.105	81.4	0.120	81.9	0.128	82.2	0.105
4m.....	74.7	0.305	74.5	0.260	77.7	0.216	77.7	0.140	78.4	0.158	79.5	0.138	77.9	0.151	80.2	0.145	79.2	0.128
8m.....	73.0	0.325	74.2	0.290	76.9	0.265	77.6	0.185	76.7	0.178	78.2	0.194	77.1	0.181	77.8	0.197	77.7	0.169
16m.....	74.6	0.380	73.2	0.348	76.3	0.316	76.4	0.206	75.8	0.203	76.1	0.228	75.9	0.210	75.6	0.235	75.2	0.182
32m.....	74.3	0.400	73.4	0.357	74.7	0.338	75.9	0.240	75.0	0.234	74.6	0.258	75.8	0.232	74.4	0.260	74.4	0.230
1h.....	71.8	0.408	72.3	0.384	73.4	0.383	74.0	0.330	73.5	0.304	72.6	0.324	72.7	0.302	73.2	0.292	72.0	0.307
2h.....	70.0	0.418	71.6	0.398	72.1	0.400	72.2	0.391	72.8	0.359	71.8	0.359	71.6	0.324	73.0	0.331	72.2	0.341
4h.....	69.0	0.418	70.4	0.400	71.6	0.400	71.9	0.398	72.0	0.400	71.8	0.398	71.6	0.360	72.0	0.370	69.8	0.368
8h.....	69.4	0.426	69.4	0.405	70.4	0.404	70.9	0.402	71.7	0.405	70.7	0.408	71.2	0.372	71.0	0.390	69.5	0.398

s-m-h = Seconds-minutes-hours at temperature of anneal.
 R_H = Rockwell H hardness number.
 GS = Grain diameter in millimeters.
 CR = Cold rolled.

TABLE 2
ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS IN MILLIMETERS FOR VARIABLE DEFORMATION
AND TIME AT 650 DEG. C. ANNEAL

Time	Percentage Deformation																		
	5.3		10.0		15.0		20.9		28.0		42.8		50.4		60.4		70.0		
	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.8	119.2	119.2
15s.....	90.5	84.0	82.0	0.098	83.6	0.086	85.7	0.074	91.6	0.046	90.5	0.035	92.7	0.029	94.1	0.027	94.1
30s.....	87.7	84.4	81.2	0.107	83.0	0.092	85.9	0.082	89.7	0.046	90.1	0.038	92.6	0.033	91.0	0.031	91.0
1m.....	80.7	79.0	0.148	80.1	0.113	82.1	0.099	84.9	0.089	87.4	0.048	88.3	0.047	89.3	0.041	89.3	0.038	89.3
2m.....	78.2	0.218	77.3	0.164	78.7	0.118	82.7	0.102	84.4	0.094	86.4	0.058	87.4	0.059	88.2	0.046	88.5	0.042	88.5
4m.....	75.3	0.224	76.6	0.178	78.2	0.120	81.1	0.102	82.4	0.098	85.0	0.079	84.5	0.075	85.3	0.058	84.9	0.068	84.9
8m.....	74.0	0.236	76.2	0.188	80.0	0.127	79.2	0.123	81.0	0.110	82.1	0.094	82.4	0.124	83.8	0.070	82.6	0.091	82.6
16m.....	75.6	0.265	75.8	0.199	77.4	0.153	78.9	0.151	80.0	0.137	80.6	0.132	81.0	0.156	82.0	0.103	81.3	0.121	81.3
32m.....	72.6	0.270	74.9	0.230	76.1	0.204	76.6	0.175	79.2	0.167	77.6	0.164	78.0	0.170	77.6	0.136	78.4	0.154	78.4
1h.....	72.0	0.272	75.5	0.242	75.8	0.218	76.7	0.191	77.5	0.188	75.9	0.192	76.5	0.188	77.0	0.177	76.9	0.192	76.9
2h.....	73.7	0.279	74.2	0.260	76.8	0.246	77.1	0.212	76.2	0.220	74.7	0.206	74.3	0.216	76.1	0.212	76.2	0.238	76.2
4h.....	73.4	0.285	75.1	0.274	74.8	0.266	75.3	0.240	73.8	0.249	76.6	0.244	74.5	0.238	73.3	0.220	74.6	0.245	74.6
8h.....	69.8	0.292	74.5	0.284	71.6	0.288	72.4	0.264	71.1	0.260	73.9	0.254	73.4	0.260	75.5	0.262	75.2	0.274	75.2

s-m-h = Seconds—minutes—hours at temperature of anneal.

R_H = Rockwell H hardness number.

GS = Grain diameter in millimeters.

CR = Cold rolled.

TABLE 3
ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS IN MILLIMETERS FOR VARIABLE DEFORMATION
AND TIME AT 600 DEG. C. ANNEAL

Time	Percentage Deformation																		
	5.3		10.0		15.0		20.9		28.0		42.8		50.4		60.4		70.0		
	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.8	119.2	119.2
15s.....	92.2	96.4	90.2	89.1	90.6	93.4	95.0	97.2	98.4	98.4
30s.....	92.1	94.3	84.2	85.5	87.2	92.5	93.6	95.1	96.2	96.2
1m.....	89.7	84.8	84.0	84.7	87.3	91.2	91.3	94.3	94.4	94.4
2m.....	81.0	82.8	84.3	86.0	90.6	90.0	92.2	92.1	92.1
4m.....	86.8	79.6	79.4	85.0	85.5	87.6	88.6	90.7	90.9	90.9
8m.....	78.5	76.8	80.8	82.8	83.5	86.0	87.6	89.6	89.5	89.5
16m.....	76.9	77.8	80.3	82.2	84.7	84.2	85.7	85.9	86.5	86.5
32m.....	74.4	77.0	79.1	80.7	82.4	83.4	84.2	85.4	85.4	85.4
1h.....	74.5	76.3	80.3	80.2	80.7	81.6	81.9	82.4	83.1	83.1
2h.....	74.0	75.5	78.5	79.7	80.5	81.6	80.0	80.7	80.5	81.0
4h.....	73.6	75.4	77.6	78.4	78.8	78.8	78.9	79.2	79.3	81.0
8h.....	73.0	75.5	75.5	75.5	78.6	77.0	76.9	77.1	79.3	169

s-m-h = Seconds—minutes—hours of anneal at temperature.

R_H = Rockwell H hardness number.

GS = Grain diameter in millimeters.

CR = Cold rolled.

TABLE 4
 ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS IN MILLIMETERS FOR VARIABLE DEFORMATION
 AND TIME AT 550 DEG. C. ANNEAL

Time	Percentage Deformation																		
	5.3		10.0		15.0		20.9		28.0		42.8		50.4		60.4		70.0		
	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.0	119.2	120.0
15s.....	94.5	99.0	103.2	102.1	96.0	98.4	98.5	99.6	101.4	101.6
30s.....	94.5	98.0	101.8	100.0	94.0	96.5	98.1	99.1	101.8	101.6
1m.....	93.9	98.8	94.6	91.8	92.0	93.8	96.0	97.1	98.8	98.8
2m.....	91.8	97.5	92.5	90.8	89.5	93.5	90.2	97.3	98.5	98.8
4m.....	92.8	93.5	87.0	90.8	89.5	92.5	93.2	96.3	98.5	98.8
8m.....	92.8	88.0	82.0	87.4	89.4	91.6	93.7	94.0	95.5	95.5
16m.....	88.5	79.0	81.9	87.5	89.8	91.6	91.7	92.8	93.6	93.6
32m.....	85.5	78.5	79.5	86.5	87.0	89.0	89.5	91.6	92.6	92.6
1h.....	79.9	79.7	80.3	83.9	84.8	89.4	87.1	91.6	92.6	92.6
2h.....	77.9	75.3	79.3	83.7	84.4	87.6	86.6	90.0	91.6	91.6
4h.....	75.3	75.3	79.3	82.6	84.4	85.8	86.6	87.4	88.3	88.3
8h.....	74.3	77.3	77.9	82.0	82.5	84.8	82.2	84.4	85.3	85.3
8L.....	74.3	0.222	0.187	0.176	0.129	0.083	0.072	0.080	0.077	0.077

s-m-h = Seconds—minutes—hours of anneal at temperature.

R_H = Rockwell H hardness number.

GS = Grain diameter in millimeters.

CR = Cold rolled.

TABLE 5
ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS IN MILLIMETERS FOR VARIABLE DEFORMATION
AND TIME AT 500 DEG. C. ANNEAL

Time	Percentage Deformation																		
	5.3		10.0		15.0		20.9		28.0		42.8		50.4		60.4		70.0		
	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.0	119.2	119.2
15s.....	101.3	101.9	106.6	105.2	108.7	102.6	105.8	105.3	107.1	107.1
30s.....	98.0	100.8	104.3	104.4	102.8	98.7	101.3	101.5	103.3	103.3
1m.....	95.5	100.1	104.0	104.7	101.3	96.7	99.8	100.3	102.2	102.2
2m.....	93.5	98.6	103.5	102.0	94.9	96.7	98.5	100.1	101.9	101.9
4m.....	93.3	99.0	100.0	93.3	93.5	94.9	97.0	100.2	100.6	100.6
8m.....	93.9	97.6	91.3	90.7	89.5	94.3	95.2	97.5	98.5	98.5
16m.....	92.8	94.5	87.5	87.3	87.0	93.3	95.4	96.6	97.1	97.1
32m.....	93.3	91.2	83.8	86.3	87.9	83.3	94.0	96.5	92.3	92.3
1h.....	92.1	84.9	82.3	84.5	86.5	82.7	94.1	94.6	95.3	95.3
2h.....	89.5	80.6	81.2	83.0	85.0	81.8	91.8	93.6	93.4	93.4
4h.....	83.6	79.3	80.2	82.2	85.0	80.7	90.9	92.2	91.9	91.9
8h.....	80.3	76.4	81.0	82.4	85.5	80.0	90.6	90.0	89.5	89.5

s-m-h = Seconds—minutes—hours of anneal at temperature.
 R_H = Rockwell H hardness number.
 GS = Grain diameter in millimeters.
 CR = Cold rolled.

TABLE 7
ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS IN MILLIMETERS FOR VARIABLE DEFORMATION
AND TIME AT 400 DEG. C. ANNEAL

Time	Percentage Deformation																		
	5.3		10.0		15.0		20.9		28.0		42.8		50.4		60.4		70.0		
	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.0	119.2
1m.....	98.0	103.2	106.7	108.3	112.0	111.9	109.4	108.1	108.0
2m.....	98.5	102.6	106.6	108.2	111.9	110.6	107.6	106.3	107.2
4m.....	98.4	102.0	106.0	107.7	109.9	108.0	104.6	104.3	106.5
8m.....	97.4	102.0	105.6	107.1	109.9	105.0	103.0	103.2	105.4
16m.....	98.2	101.0	105.4	106.8	107.9	101.5	101.3	102.2	104.3
32m.....	96.9	101.9	105.4	106.8	107.7	99.9	99.1	101.5	103.3	0.016
1h.....	96.6	99.6	104.1	103.6	99.6	96.8	99.0	101.6	103.3	0.016
2h.....	94.6	98.7	100.0	96.3	93.9	95.9	97.7	98.1	102.4	0.017
4h.....	94.7	99.0	96.7	93.0	90.3	95.9	96.7	98.3	100.4	0.017
8h.....	93.9	96.9	94.5	88.7	89.3	93.4	96.0	96.4	98.8	0.017

s-m-h = Seconds—minutes—hours of anneal at temperature.
 R_H = Rockwell H hardness number.
 GS = Grain diameter in millimeters.
 CR = Cold rolled.

TABLE 8
ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS IN MILLIMETERS FOR VARIABLE DEFORMATION
AND TIME AT 350 DEG. C. ANNEAL

Time	Percentage Deformation																		
	5.3		10.0		15.0		20.9		28.0		42.8		50.4		60.4		70.0		
	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	R _H	GS	
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.0	119.2	119.2
1m.....	100.0	103.0	107.8	109.0	111.8	116.0	117.5	117.8	117.0	117.0
2m.....	99.4	102.7	107.2	109.4	111.8	115.4	115.7	114.2	113.9	113.9
4m.....	100.0	102.9	107.1	108.8	111.2	114.8	113.8	114.2	110.8	110.8
8m.....	98.2	102.4	106.4	109.0	111.7	113.4	111.6	109.1	109.2	109.2
16m.....	98.1	102.3	106.5	108.9	111.2	112.4	110.3	107.4	107.1	107.1
32m.....	99.0	102.3	106.3	107.7	110.2	111.1	107.6	105.5	106.4	106.4
1h.....	98.1	102.5	107.8	107.7	110.4	107.3	104.3	104.0	105.6	105.6
2h.....	98.3	103.0	107.1	107.7	109.0	102.6	101.8	101.9	104.7	104.7
4h.....	97.9	102.3	105.9	106.8	107.0	101.7	100.3	101.8	104.2	104.2
8h.....	97.0	100.3	104.2	105.8	104.0	97.5	99.2	99.4	103.0	103.0

s-m-h = Seconds—minutes—hours of anneal at temperature.

R_H = Rockwell H hardness number.

GS = Grain diameter in millimeters.

CR = Cold rolled.

TABLE 9
ROCKWELL H HARDNESS AND GRAIN SIZE DIAMETERS* IN MILLIMETERS FOR VARIABLE DEFORMATION
AND TIME AT 300 DEG. C. ANNEAL

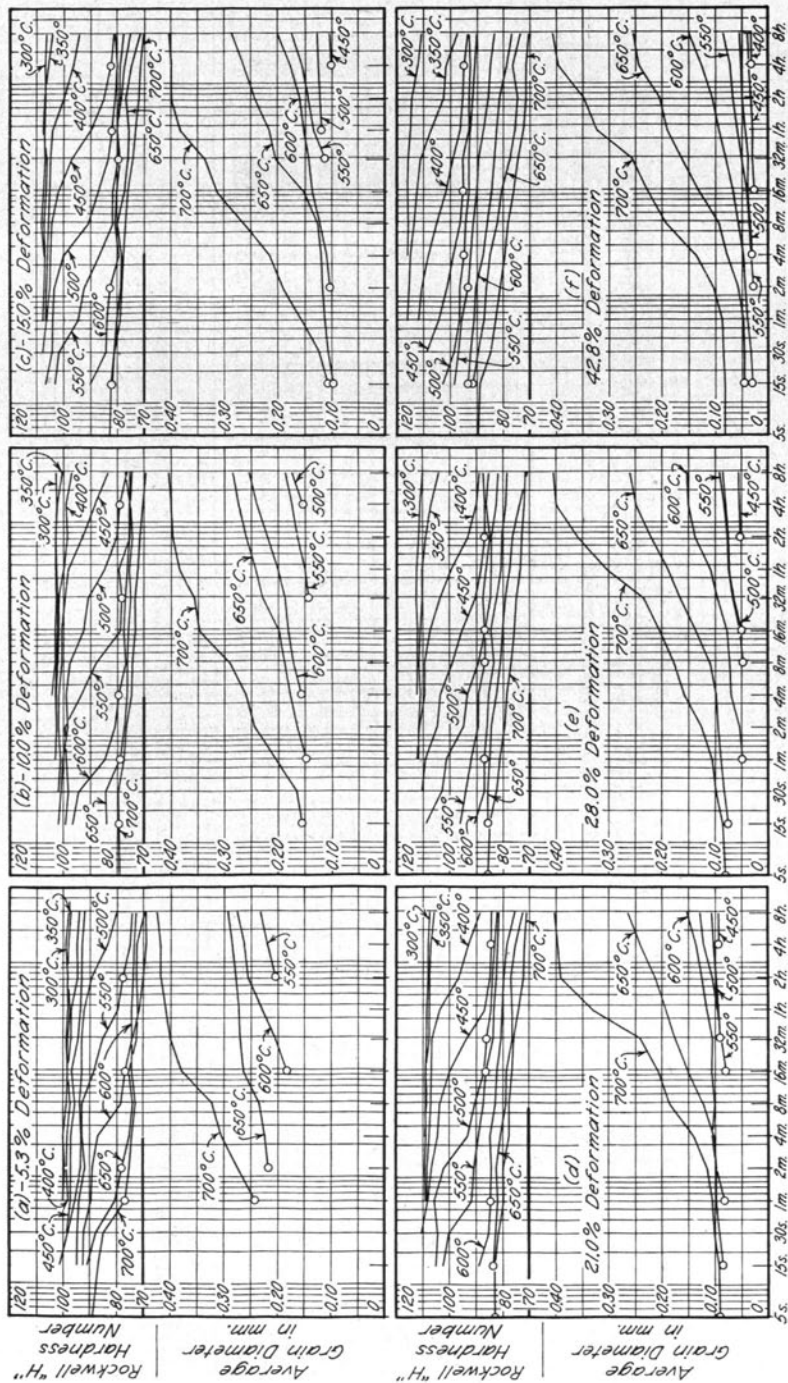
Time	Percentage Deformation									
	5.3	10.0	15.0	20.9	28.0	42.8	50.4	60.4	70.0	
CR.....	100.0	103.8	107.0	111.0	113.5	116.0	117.5	118.0	119.2	
4m.....	99.9	104.3	107.3	109.3	112.2	116.0	118.6	119.0	119.0	
8m.....	98.4	103.0	108.1	108.6	112.1	115.8	117.5	118.1	117.8	
16m.....	98.1	103.0	107.5	108.9	112.0	116.0	117.0	118.0	115.2	
32m.....	98.2	102.7	107.0	108.5	112.5	115.7	116.6	114.0	113.4	
1h.....	97.3	102.8	107.5	108.6	112.5	114.9	113.8	112.0	111.1	
2h.....	98.1	102.7	106.9	108.0	111.6	114.3	112.1	110.8	109.2	
4h.....	98.9	102.4	106.8	108.0	111.2	112.0	108.3	108.2	107.3	
8h.....	97.8	102.8	106.4	107.4	110.2	110.7	107.5	106.3	106.9	
16h.....	96.8	101.3	107.0	107.1	109.9	111.2	106.2	105.9	105.6	
48h.....	97.2	102.2	105.2	107.6	109.1	103.3	100.8	101.9	104.7	
96h.....	96.5	101.6	105.3	106.4	108.1	101.5	100.6	101.2	103.7	
240h.....	95.2	101.9	104.0	106.4	103.2	97.4	100.0	102.0	103.4	
336h.....	96.7	102.1	105.0	105.8	100.9	97.7	99.0	100.1	103.2	

s-m-h = Seconds—minutes—hours of anneal at temperature.

Rg = Rockwell H hardness number.

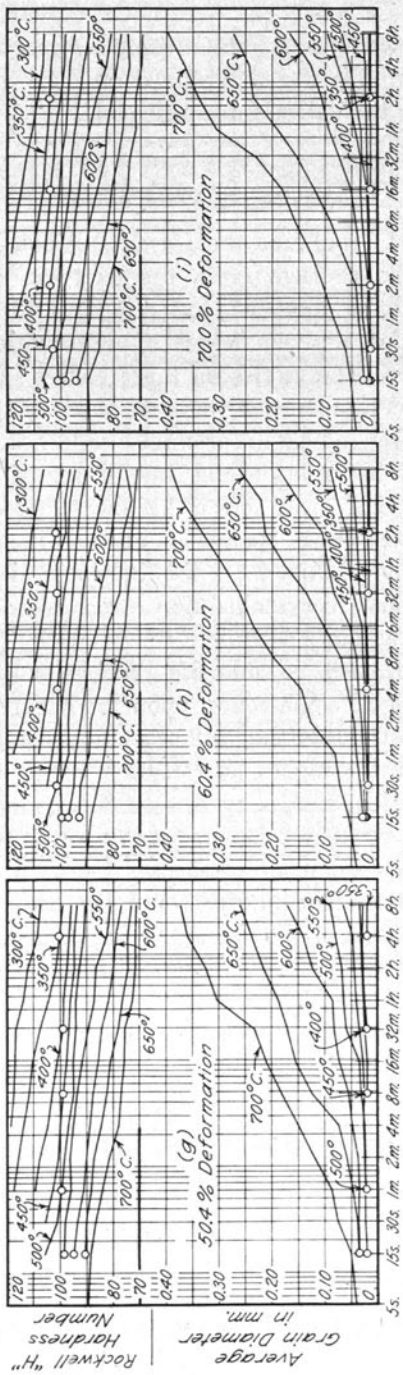
CR = Cold rolled.

*Complete recrystallization was not observed with certainty at this temperature in 8 hr. annealing cycle.



Time of Anneal (Plotting based on Seconds).

FIG. 5. GRAIN SIZE AND HARDNESS vs. TIME



Time of Anneal (Plotting Based on Seconds)

Temperatures are those at which the specimens were annealed, (deg. C.)
 Per cent deformation is the degree of prior cold deformation.
 o-Complete recrystallization first observed

FIG. 5. GRAIN SIZE AND HARDNESS vs. TIME

amination proved these specimens were not completely recrystallized for the experimental conditions. When complete recrystallization was established, the grain size was determined and the increase in grain size was followed for the entire annealing cycle.

VI. DISCUSSION OF RESULTS

14. *Dependence of Grain Size and Hardness Upon Time of Anneal.*—Figure 5 shows the average grain diameters in millimeters and the Rockwell H hardness numbers plotted against the time of anneal (logarithmic scale) for a given degree of deformation and a given temperature of anneal. The circles on these curves indicate the time at which complete recrystallization was first observed.

The hardness curves indicate a gradual loss in hardness with time, for a given degree of deformation and time of anneal, until recrystallization commences. This initial loss in hardness represents the period of recovery and is sometimes spoken of as the "incubation period." When recrystallization commences, the hardness decreases rapidly until the time at which recrystallization is complete, and the curve then flattens out and assumes a straight line relation. The loss in hardness after recrystallization is complete represents the effect of increased grain size, due to coalescence, upon the hardness number. For a given degree of deformation the hardness number at the point where recrystallization is just complete is constant, regardless of the time and temperature relations necessary to establish complete recrystallization. The hardness number, when recrystallization is just complete, increases with an increase in deformation, due to a decrease in the recrystallized grain size. The hardness number after eight hours at temperature is approximately the same for all deformations annealed at a given temperature, provided recrystallization is complete and appreciable coalescence has taken place.

The grain size curves (Fig. 5) represent a family of curves having the same general shape, and clearly demonstrate the dependence of grain growth by coalescence upon both time and temperature. For a given deformation the grain size representing complete recrystallization, without coalescence, is constant, and independent of the time and temperature necessary to establish complete recrystallization. The higher the annealing temperature, the shorter the time required to produce complete recrystallization for a given degree of cold deformation. It should be noted that recrystallization was complete, for some of the deformation-temperature conditions, before the material had

reached the temperature of annealing. The grain size produced after eight hours' annealing was practically constant for a given temperature of anneal, regardless of the degree of prior deformation, so long as appreciable coalescence had taken place. The grain size is less, the greater the prior deformation, when recrystallization is just complete, and is independent of both time and temperature.

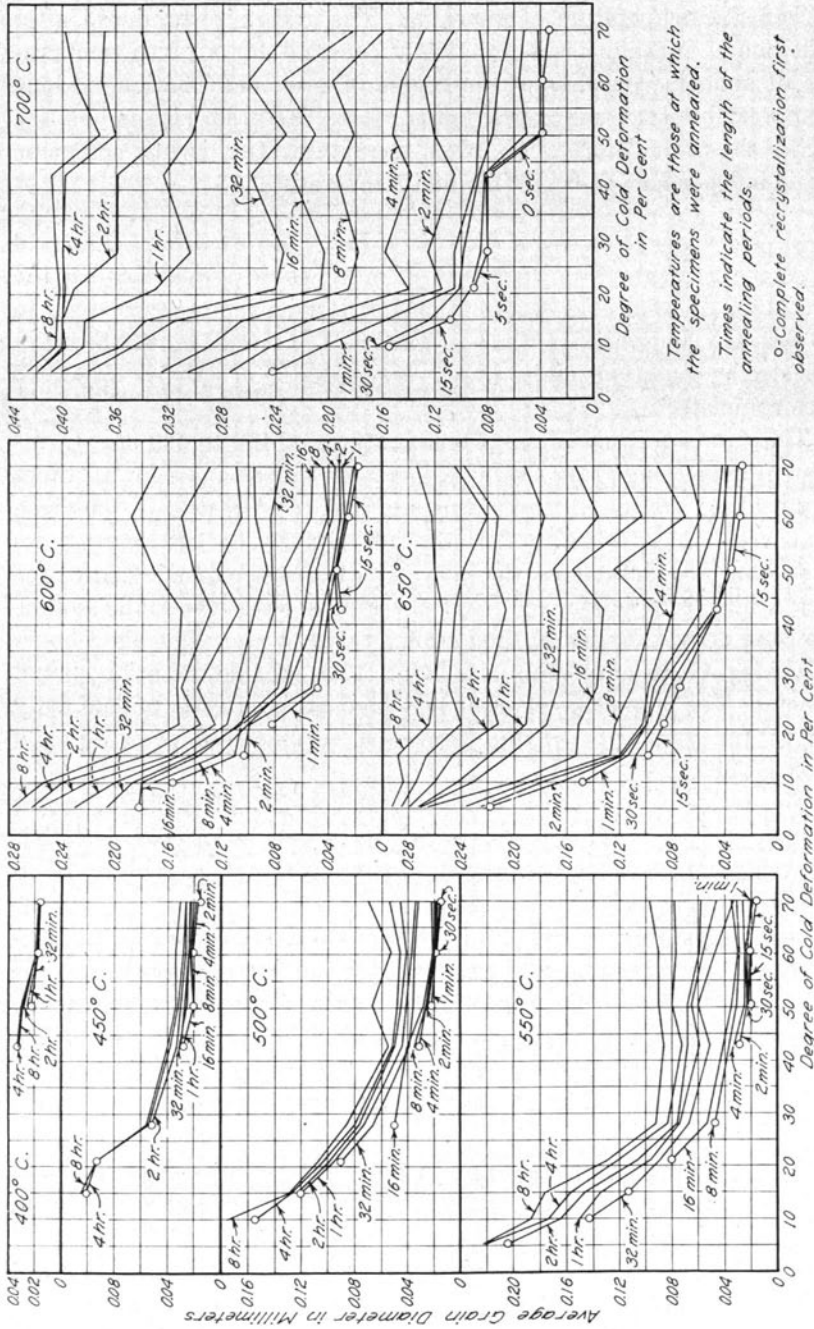
15. *Dependence of Grain Size Upon Prior Deformation.*—Figure 6 shows the average grain diameters in millimeters plotted against the percentage of prior cold deformation for a given temperature (400 to 700 deg. C.) and variable times of anneal. The circles on these curves indicate the conditions under which recrystallization was first observed to be complete.

The curves for annealing at temperatures of 400 to 600 deg. C. indicate a decreasing grain size with increased deformation for all times of anneal. At annealing temperatures of 650 and 700 deg. C. the grain size curves are considerably flattened, particularly for the longer times of anneal. The flatness of the curves indicates that coalescence was approaching completion, and that the grains were essentially stable. The close approximation of final grain size, after annealing eight hours at 700 deg. C., to prior grain size before final cold deformation should be noted. The similarity of prior and final grain sizes is undoubtedly due to the similarity of the annealing temperature prior to cold deformation.

Theoretically, the most stable metal consists of a single grain; however, polycrystalline metals which have been subjected to rather high temperatures for great lengths of time have not been observed to grow into single crystals by coalescence.

Single crystals of aluminum have been produced by a slight deformation of polycrystalline aluminum followed by annealing. In this case the resulting single crystal is the result of true recrystallization. In other words, a single nucleus is established in the slightly deformed aluminum, and grain growth results from the unstrained nucleus growing at the expense of its strained surroundings, and does not result from coalescence.

The failure of polycrystalline metals to grow into single crystals by a process of coalescence can be explained as follows: Coalescence of metallic grains takes place by a decrease in the surface energy of individual grains as the ratio of surface area to volume is decreased by increased grain volume through grain growth. There is apparently an equilibrium value for the surface energy of individual grains and



Temperatures are those at which the specimens were annealed.
 Times indicate the length of the annealing periods.
 o-Complete recrystallization first observed.

FIG. 6. GRAIN SIZE VS. DEFORMATION—VARIABLE TIME

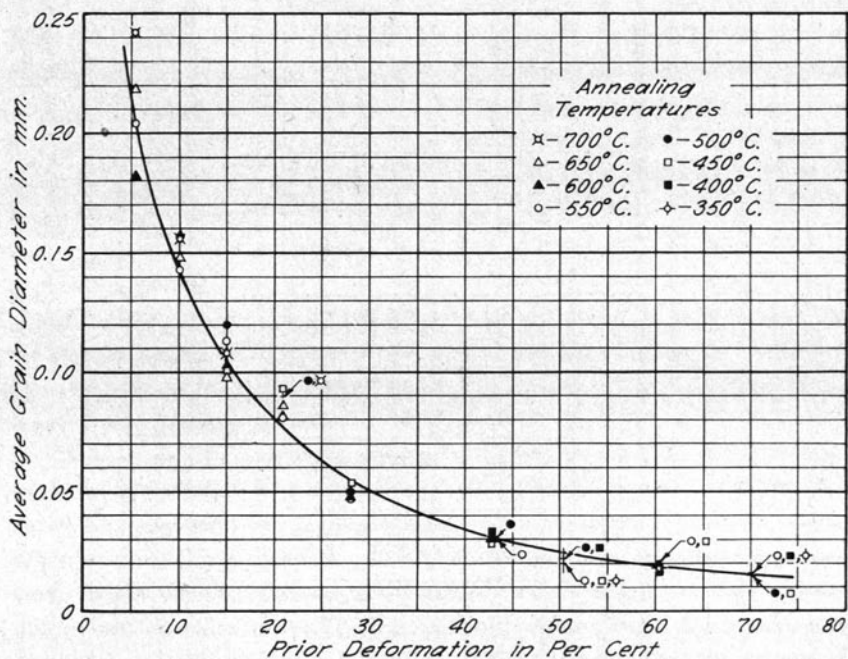


FIG. 7. GRAIN SIZE vs. DEFORMATION AT COMPLETE RECRYSTALLIZATION; NO APPRECIABLE COALESCENCE

the thermal mobility of atoms within the grains for a given temperature. If the temperature of annealing is increased, then the thermal mobility of the atoms is increased, and a further decrease in surface energy may take place through grain growth. This would indicate that there is a comparatively stable grain size, for a given temperature of annealing, provided a sufficient amount of coalescence has taken place and that no appreciable grain size contrast exists.

In Fig. 7 is summarized the grain size as a function of the percentage of prior deformation. The grain sizes represented are those first observed when recrystallization was just complete. These grain sizes were produced by varying the time and temperature of the anneal to produce complete recrystallization without appreciable coalescence of the recrystallized grains. Within the accuracy of the experiment the grain sizes may be said to be constant for a given prior deformation. This constancy of grain size indicates that the grain size is independent of both time and temperature, and is a function of the

TABLE 10
 TIME-TEMPERATURE-DEFORMATION CONDITIONS WHEN RECRYSTALLIZATION WAS FIRST OBSERVED
 TO BE COMPLETE UNDER MICROSCOPE

Percentage Deformation	700 deg. C.		650 deg. C.		600 deg. C.		550 deg. C.		500 deg. C.		450 deg. C.		400 deg. C.		350 deg. C.	
	Time	GS	Time	GS	Time	GS	Time	GS	Time	GS	Time	GS	Time	GS	Time	GS
5.3	60s	0.242	2m	0.218	16m	0.182	120m	0.204	240m	0.154	240m	0.101	240m	0.033	240m	0.020
10.0	15s	0.155	1m	0.148	4m	0.157	32m	0.143	60m	0.120	240m	0.093	240m	0.017	32m	0.018
15.0	15s	0.108	15s	0.098	2m	0.103	32m	0.113	32m	0.090	240m	0.052	240m	0.015	32m	0.016
20.9	5s	0.090	15s	0.086	1m	0.082	16m	0.080	16m	0.050	120m	0.028	120m	0.017	32m	0.018
28.0	5s	0.080	15s	0.074	1m	0.048	8m	0.047	16m	0.031	16m	0.028	16m	0.017	32m	0.018
42.8	0s	0.078	15s	0.046	15s	0.034	2m	0.029	4m	0.022	8m	0.021	8m	0.017	32m	0.018
50.4	0s	0.038	15s	0.029	15s	0.025	15s	0.021	30s	0.019	4m	0.015	4m	0.017	32m	0.018
60.4	0s	0.038	15s	0.027	15s	0.018	15s	0.016	30s	0.015	2m	0.015	2m	0.016	120m	0.016
70.0	0s	0.033	15s	0.027	15s	0.018	15s	0.016	30s	0.015	2m	0.015	2m	0.016	120m	0.016

GS = Grain size in millimeters diameter.
 1s = 1 second at temperature of anneal.
 1m = 1 minute at temperature of anneal.
 = Incomplete recrystallization for conditions.

prior deformation only (for a given metal). The temperatures of annealing are indicated in the legend of Fig. 7. The data from which Fig. 7 was plotted are tabulated in Table 10.

Jeffries and Archer¹², in writing of grain growth and recrystallization, state that "certain combinations of non-uniformity of structure or temperature give rise to what may be termed 'exaggerated grain growth'—that is, the production of grains of abnormally large size. Any case of the development of abnormally large grains may be called germination. The temperature range within which the abnormally large grains are formed is called the germination temperature range." In the discussion of germination and germination temperature range, the experiments and conclusions of Sauveur¹³, Chappell¹⁴ and Ruder¹⁵ were given as examples of the phenomenon.

The conception of germination whereby an exceptionally large grain size resulted after annealing in the germination temperature range has confused the real mechanism of recrystallization for a number of years. Germination implies that a range of annealing temperatures exists which produces an extremely large grain but, for annealing temperatures above the germination temperature, the grain size produced is smaller than with the same degree of strain annealed within the germination temperature. The results of this experiment fail to show the existence of germination as originally proposed. For the range of temperatures and deformations of this experiment, the resultant grain sizes produced by recrystallization form a smooth curve. This curve (Fig. 7) indicates that grain size at complete recrystallization is independent of both time and temperature of anneal and is dependent only upon the degree of strain. Eastwood, Bousu, and Eddy¹⁶ in a series of experiments with 89-11 and 66-34 brass strain gradient bars and temperature gradients failed to show the presence of germination or germination temperatures. The results of their experiments also indicate that the grain size produced at complete recrystallization is independent of both time and temperature of anneal. R. S. French¹⁷ has reported a series of experiments upon the grain sizes produced upon recrystallization of cold-rolled cartridge brass. French's results are similar to the results reported in this bulletin and indicate that grain size produced at complete recrystallization is independent of both time and temperature.

Figure 8 shows the relation between recrystallized grain size (no appreciable coalescence) and percentage of cold deformation, when the grain size is plotted on the logarithmic scale against the square root

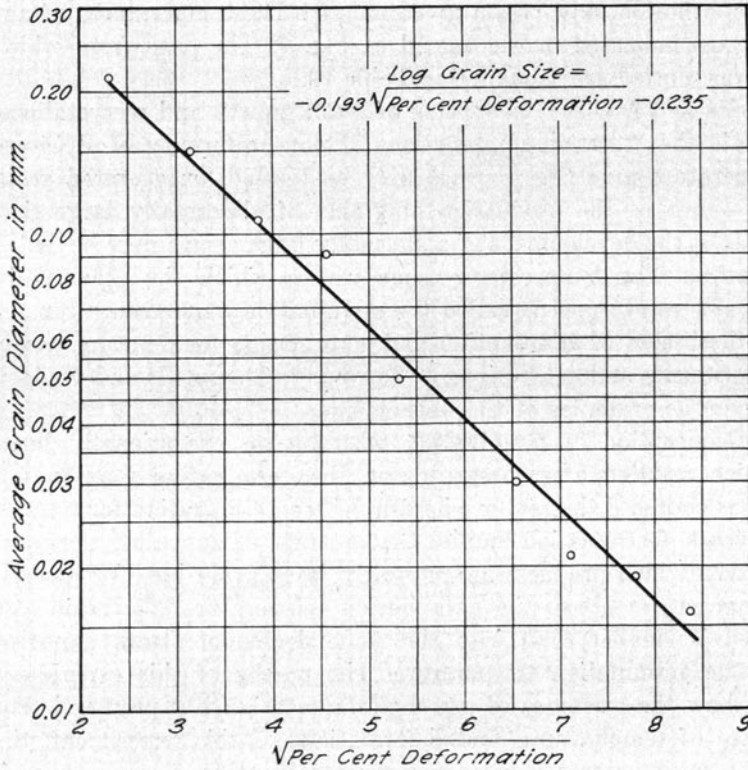


FIG. 8. DEPENDENCE OF RECRYSTALLIZED GRAIN SIZE UPON PRIOR DEFORMATION

of the percentage deformation. A straight line is produced of the general exponential type:

$$Y = mX^n \text{ or } Y = m^{10nx}$$

$$\text{or } \log y = \log m + nx \log 10 = \log m + nx$$

$$\text{log grain size} = \log m + n \sqrt{\text{percentage deformation.}}$$

If the curve is extrapolated to zero per cent deformation, then the log grain size at zero deformation equals $\log m$, a constant. The equation for the curve shown becomes:

$$\text{log grain size} = -0.193 \sqrt{\text{percentage deformation}} - 0.235. \quad (1)$$

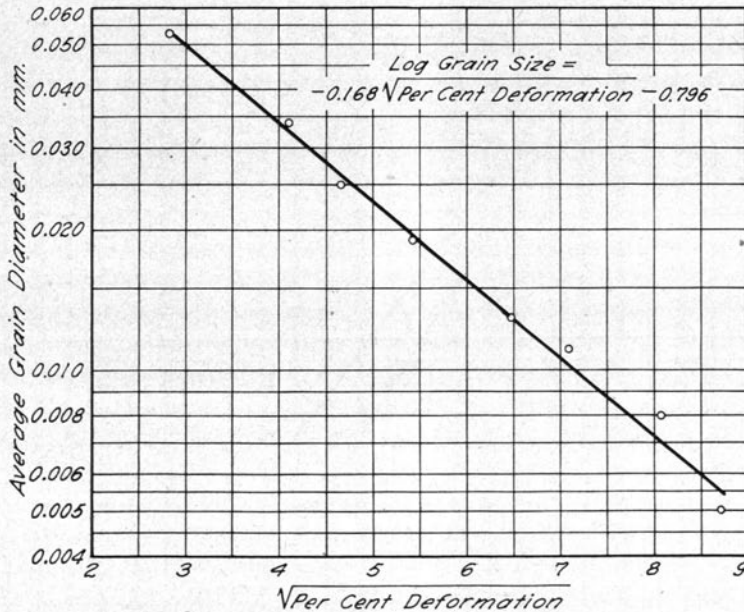


FIG. 9. DEPENDENCE OF RECRYSTALLIZED GRAIN SIZE UPON PRIOR DEFORMATION; DATA FROM FRENCH¹⁷

Figure 9 shows the data of French¹⁷ plotted in the same manner as Fig. 8. The equation for the data in Fig. 9 has been found to be

$$\log \text{ grain size} = -0.168 \sqrt{\text{percentage deformation}} - 0.796. \quad (2)$$

These two curves are of the same type, and the slopes of the two curves are very nearly equal; in fact, they may very well lie within the experimental error of the two sets of data. The value of the constant, $\log m$, is very clearly a function of the average grain diameter prior to the final cold reduction. In the experiments of French the average grain size prior to final cold reduction was 0.053 mm. In this experiment the average grain size prior to final cold reduction was 0.48-0.57 mm., or approximately 10 times the average diameter of the grains in the brass used by French. Thus there is very definitely established a dependence of the grain size when recrystallization is just complete upon the grain size prior to final cold working.

No physical significance has been established for the equation derived, and it remains an empirical equation representing the observed data.

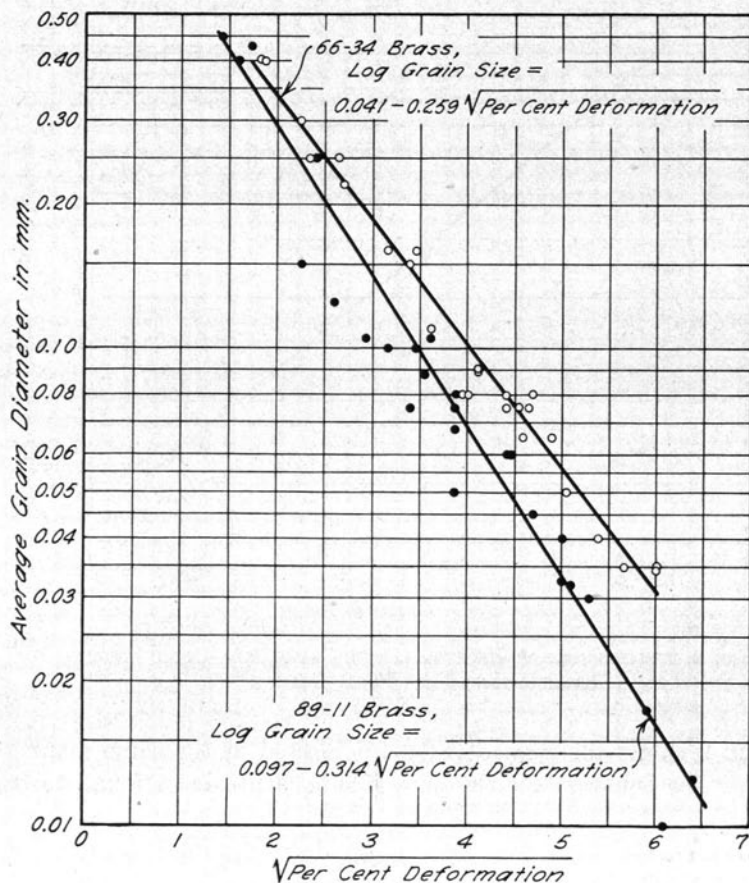


FIG. 10. DEPENDENCE OF RECRYSTALLIZED GRAIN SIZE UPON PRIOR DEFORMATION;
DATA FROM EASTWOOD-BOUSU-EDDY¹⁶

The data for Eastwood, Bousu, and Eddy have also been plotted and are shown in Fig. 10. The calculated equations for the two curves are:

$$\begin{aligned} &89-11 \text{ Brass; log grain size} = \\ &0.097 - 0.314 \sqrt{\text{percentage deformation}} \end{aligned} \quad (3)$$

$$\begin{aligned} &66-34 \text{ Brass; log grain size} = \\ &0.041 - 0.259 \sqrt{\text{percentage deformation}}. \end{aligned} \quad (4)$$

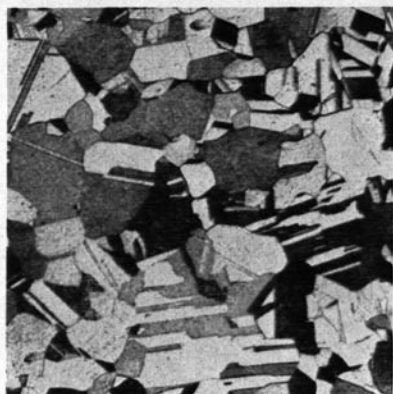
In the latter experiment there was not only a difference in composition but also in grain size and method of cold working. The average prior



(a) 2 hr. at 450 deg. C.
Not completely recrystallized.



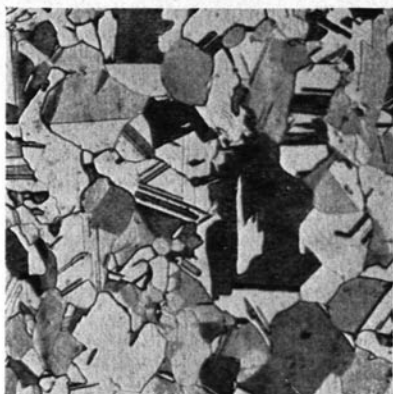
(b) 4 hr. at 450 deg. C.
Av. G.S. = 0.093 mm.



(c) 32 min. at 500 deg. C.
Av. G.S. = 0.090 mm.



(d) 16 min. at 550 deg. C.
Av. G.S. = 0.080 mm.



(e) 1 min. at 600 deg. C.
Av. G.S. = 0.092 mm.

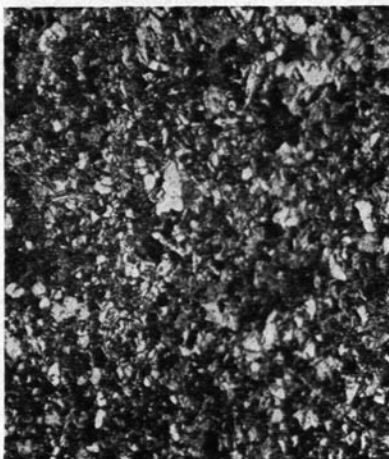


(f) 15 sec. at 650 deg. C.
Av. G.S. = 0.086 mm.

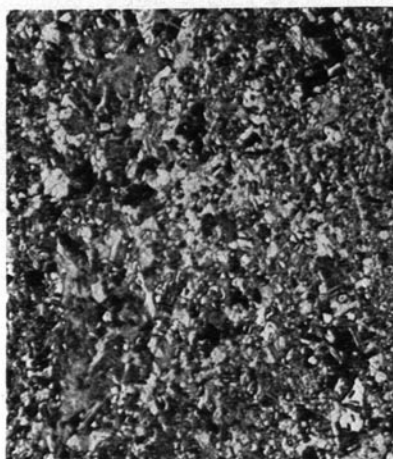
FIG. 11. MICROSTRUCTURE AT COMPLETE RECRYSTALLIZATION FOR 20.9 PER CENT COLD DEFORMATION—VARIABLE TIME AND TEMPERATURE OF ANNEALING—MAG. 75X



(a) 16 min. at 400 deg. C.
Av. G.S. = 0.016 mm.



(b) 2 min. at 450 deg. C.
Av. G.S. = 0.015 mm.



(c) 30 sec. at 500 deg. C.
Av. G.S. = 0.015 mm.



(d) 15 sec. at 650 deg. C.
Av. G.S. = 0.027 mm.
Considerable coalescence.

FIG. 12. MICROSTRUCTURE AT COMPLETE RECRYSTALLIZATION FOR 70 PER CENT COLD DEFORMATION—VARIABLE TIME AND TEMPERATURE OF ANNEALING—MAG. 225 \times

grain size was 0.60 mm. The 66-34 brass was machined to tapered bars and strained in tension to produce a strain gradient bar. The 89-11 brass was machined to tapered wedges and rolled to strain gradient bars. The results are therefore subject to differences in (1) compo-

sition, (2) prior grain size, (3) strain gradients, and (4) statistical grain count in determining the average grain diameter.

16. *Microstructure of Cold-Worked and Annealed Metal.*—The very great number of experimental annealing schedules does not permit illustrations of all the structures produced. The structure of the cold-worked metal is shown in Fig. 1. To illustrate the dependence of grain size at complete recrystallization upon the prior plastic deformation, and the independence of time and temperature of anneal, there are shown in Figs. 11 and 12 the microstructures for 21 per cent and 70 per cent deformation, for a series of anneals at different times and temperatures. Figure 13 shows the progress of recrystallization and coalescence for cartridge brass with a prior deformation of 28 per cent.

VII. SUMMARY

17. *Summary of Conclusions.*—On the basis of experimental data obtained in this experiment the following features governing grain growth are presented:

(1) Metal grains in the solid state may grow at the expense of cold-deformed metal (recrystallization) or at the expense of unstrained metal (coalescence).

(2) The grain size at just complete recrystallization for cartridge brass is independent of time and temperature, and is dependent only upon the degree of deformation and upon the composition and history of the metal prior to deformation.

(3) There is no indication of a germinant temperature for the production of abnormally large grains, since an increase in temperature for a given deformation and time of anneal produces a coarser grain rather than finer grain.

(4) Coarser grains are produced in the just completely recrystallized metal by a decrease in the degree of cold deformation.

(5) Grain size increases with time and temperature and obeys the rules of normal grain growth, whether the growth be at the expense of cold-deformed metal (recrystallization) or at the expense of newly formed and unstrained grains (coalescence).

(6) The number of nuclei that serve as recrystallization centers is independent of temperature, but is dependent upon the degree of cold deformation.

(7) The rules of normal grain growth account for all instances of the growth and production of large grains.



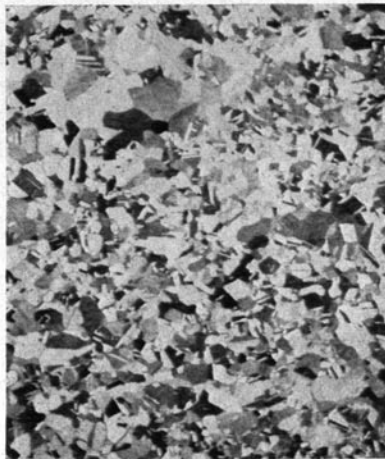
(a) 15 sec. at 500 deg. C.
Recrystallization has just begun.



(b) 4 hr. at 400 deg. C.
Partial recrystallization.

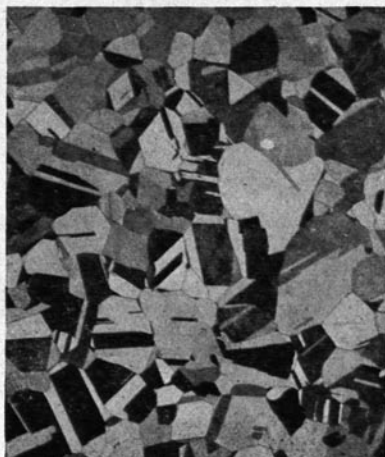


(c) 8 min. at 500 deg. C.
Recrystallization almost complete.



(d) 5 sec. at 700 deg. C.
Recrystallization just completed.

FIG. 13. PROGRESS OF RECRYSTALLIZATION AND COALESCENCE FOR A PRIOR COLD DEFORMATION OF 28.0 PER CENT—VARIABLE TIME AND TEMPERATURE OF ANNEALING—MAG. 30X



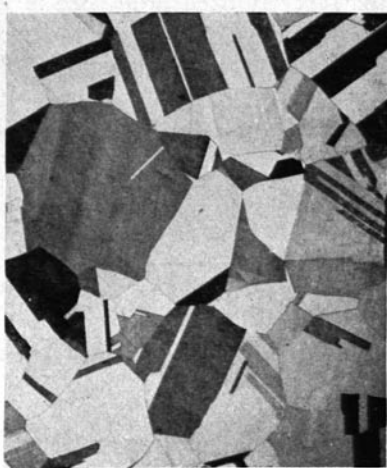
(e) 8 min. at 700 deg. C.
Coalescence.



(f) 32 min. at 700 deg. C.
Coalescence.



(g) 1 hr. at 700 deg. C.
Coalescence.



(h) 8 hr. at 700 deg. C.
Coalescence.

FIG. 13. PROGRESS OF RECRYSTALLIZATION AND COALESCENCE FOR A PRIOR COLD DEFORMATION OF 28.0 PER CENT—VARIABLE TIME AND TEMPERATURE OF ANNEALING—MAG. 30×

(8) It has been shown that the grain size at complete recrystallization (without appreciable coalescence) is a function of the prior cold deformation, and may be expressed by the equation:

$$\text{Log grain size} = -n \sqrt{\text{percentage deformation}} \pm b.$$

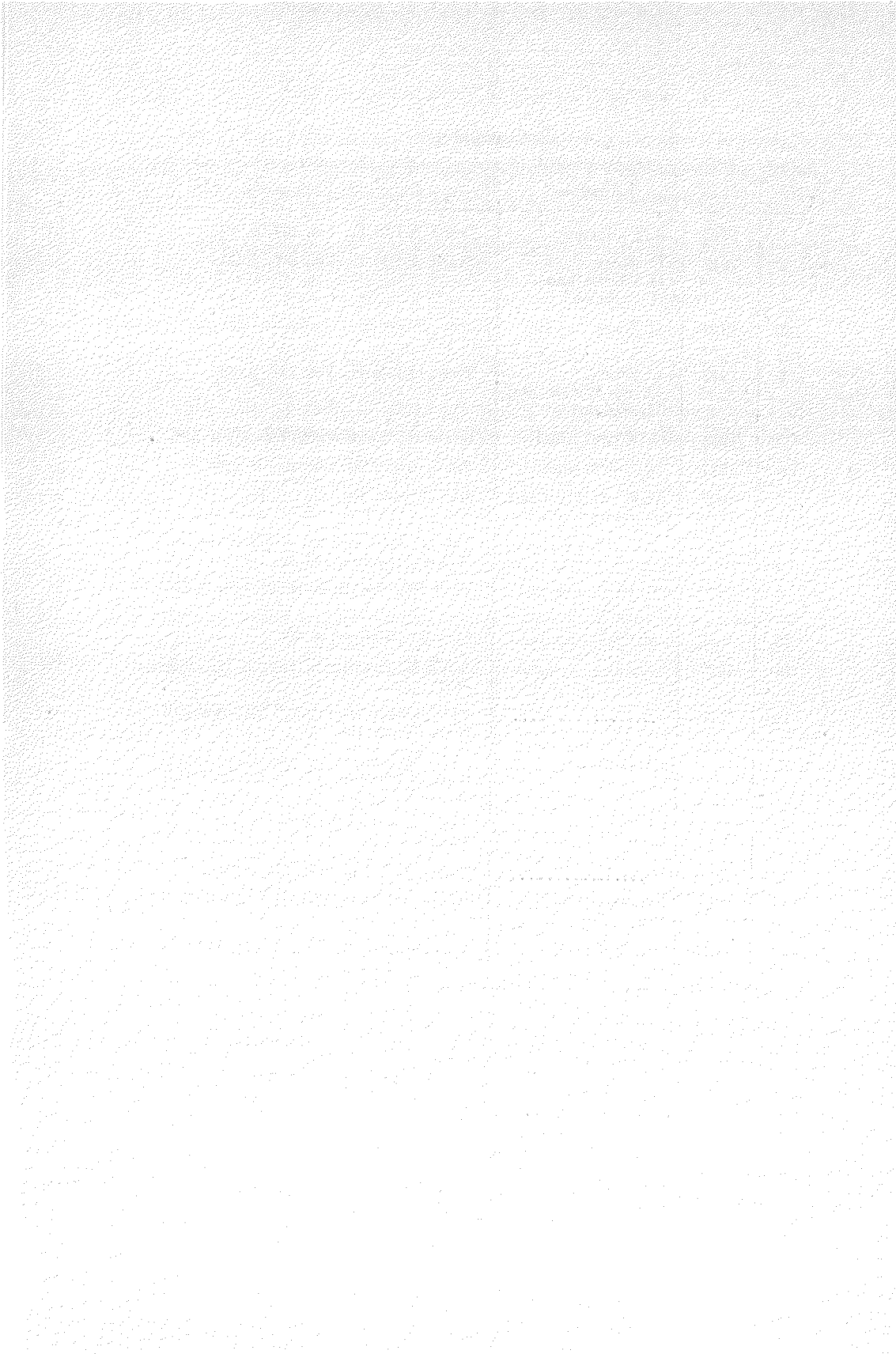
(9) For a given metal or alloy the value of the constant b in the equation is a function of the grain size prior to final deformation. The slope of the curve is probably dependent upon the composition of the metal or alloy.

(10) The velocity of grain growth by coalescence is lowered with a decreased temperature of annealing for recrystallization. The lower the temperature of annealing, the longer will be the time required to produce recrystallization. For a given percentage of cold deformation the coalesced grain size is more easily controlled, the lower the annealing temperature.

(11) The equation expressing the grain size, at complete recrystallization, as a function of prior deformation, may be used to predict the recrystallized grain size of a given brass, provided the grain size is determined for three different and widely separated degrees of cold deformation, and the values of the constants determined therefrom.

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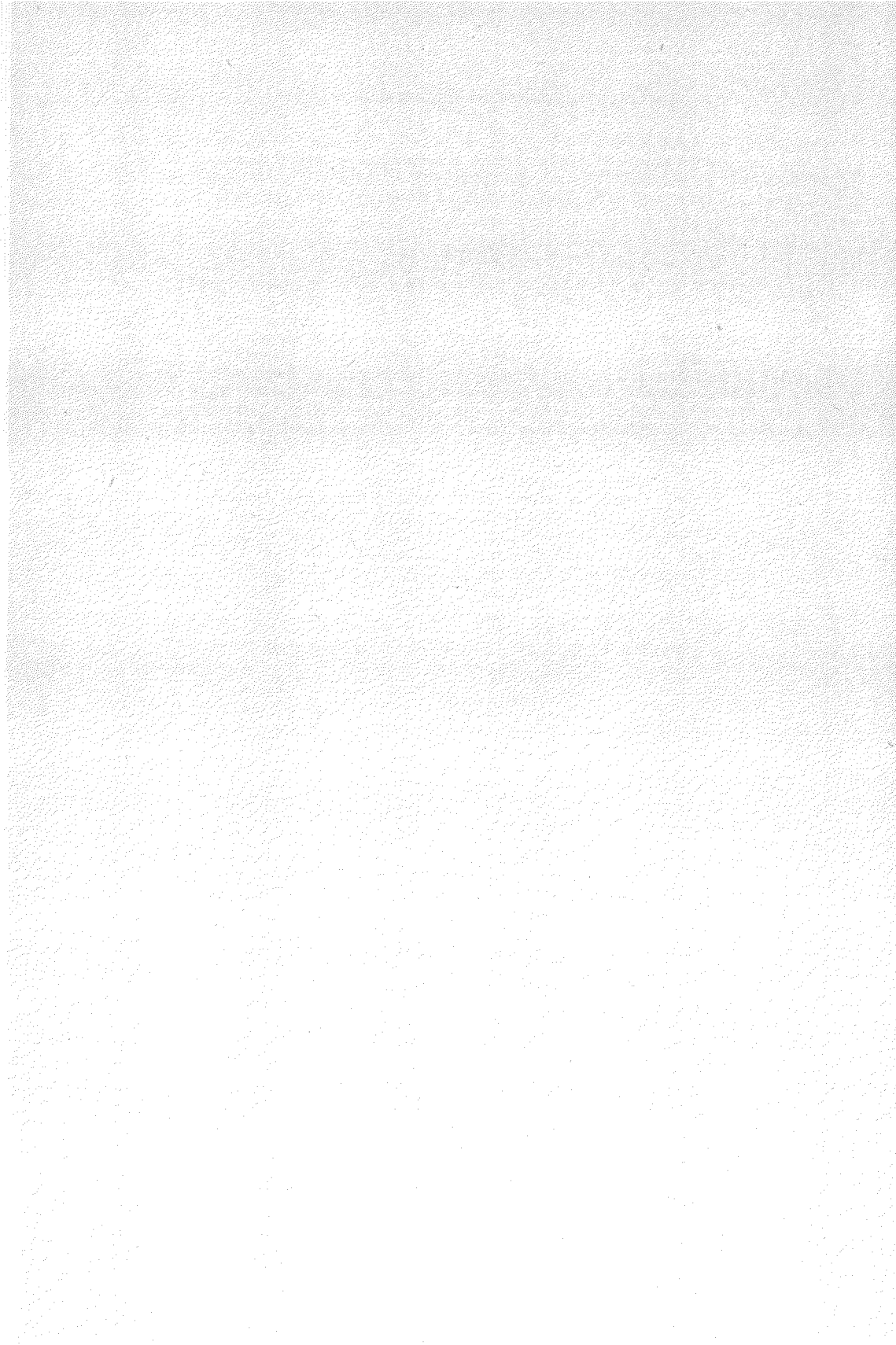
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