

RECRYSTALLIZATION OF METALLIC MATERIALS

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**RECRYSTALLIZATION
OF
METALLIC MATERIALS**

Edited by

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Preface to second edition

Numerous papers have been published on the subject of recrystallization since the first edition was published some 7 years ago. The great interest shown in this field is reflected by the many national and international conferences held during this period. In the light of this the second edition has been critically reviewed. More than half of the chapters have been considerably extended and supplemented by including new data and knowledge; some have even been completely rewritten. A chapter on "experimental techniques", by M. S. Masteller and C. L. Bauer, is a new addition to the book. A subject and metals index have also been added. Compared with the first edition, the second edition has increased by 90% in bulk.

The comparatively low price of the book was only achieved by the sponsorship of the firms DEGUSSA AG, Frankfurt; Kabel- und Metallwerke Gutehoffnungshütte AG, Hannover; and W. C. Heraeus GmbH, Hanau.

I should be very pleased if the book by way of its reasonable price and updated content may be considered a worthwhile purchase by undergraduates and graduates alike. Last but not least I wish to thank Mrs. G. Sieber who assisted in reading the proofs.

Braunschweig, May 1978

FRANK HAESSNER

Preface to first edition

The phenomenon of recrystallization has been utilized extensively in metallurgical processing for some thousand years. In spite of this considerable practical importance, the systematic study of this phenomenon only set in extensively about 50 years ago. Today the scientific and practical knowledge has reached a level which, in a number of cases, permits the planned development of materials by suitable recrystallization treatments. The conceptions and formulations originally developed for the recrystallization of metals have in the meantime been applied successfully to other crystalline materials as well, such as, for example, ceramics.

It therefore appeared desirable to treat the topic recrystallization in a special seminar. Such a seminar was organized jointly by the Institut für Metallkunde of the University of Stuttgart and by the Max-Planck-Institut für Metallforschung, Stuttgart for advanced students, scientists and participants from industry. The lectures presented in the course of this seminar appear in revised form in this book. They represent a survey of our current knowledge and of the opinions and open questions in the most significant subfields of the topic of recrystallization of metals. The individual chapters consist of contributions which, in spite of their self-contained form, are matched to complement each other. Internal cross-references, the use of uniform symbols and a detailed introduction by the editor will help to tie the various contributions together. The treatment of experimental techniques, recrystallization of strongly disturbed thin films and recrystallization of non-metallic crystalline materials has been omitted for lack of space. It is not intended that this book should form a complete text-book on recrystallization. Rather, it is hoped that the most important subjects of interest have been discussed in a way which should enable the reader to gain a simple access to the current problems.

Stuttgart, March 1971

FRANK HAESSNER

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SYSTEMATIC SURVEY AND BASIC PROBLEMS OF RECRYSTALLIZATION

BY F. HAESSNER

The terminology used in describing the phenomena of recrystallization has with the passing of time repeatedly been revised as a result of increasing understanding. Hence, it is not surprising that the term *recrystallization* frequently denotes a phenomenon that may differ in detail from case to case. In this way misinterpretation may occur on critical comparison of the literature. Consequently, in the following presentation that is intended as an introduction to the individual chapters and as a type of guide for the book, an attempt will first of all be made to classify the entirety of the recrystallization phenomena of metals in a scheme which facilitates a survey. Subsequently, attention is turned to those categories of questions that always reappear in one way or another in connection with recrystallization, namely, the *basic problems*.

1. SYSTEMATIC SURVEY

1.1. Decrease in (Gibbs) Free Energy and Reaction of Lattice Defects

The essential processes occurring during recrystallization may be discussed reasonably well by considering a plastically deformed crystalline material. For the sake of simplicity, it is first assumed that the material does not undergo a phase transition in the temperature range considered. As a result of its increased content of physical defects, such a material is in a thermodynamically unstable state. Upon increasing the temperature the material can lower its free energy* by the reduction and rearrangement of the lattice defects. In particular cases, this may occur in quite different ways. For a systematic classification, it has proved appropriate and useful to describe the multitude of phenomena that occur below the melting point in terms of five fundamental structural processes. These fundamental processes, whereby a pure metal can approach a state of equilibrium, are essentially the following:

- 1) Reactions of point defects and point defect agglomerates; in particular the annihilation of these defects.
- 2) The annihilation of dislocations of opposed sign and the shrinking of dislocation loops.
- 3) The rearrangement of dislocations to form energetically more favourable configurations.
- 4) The absorption of point defects and dislocations by grain boundaries migrating through the metal.
- 5) The reduction in the total grain boundary area.

* In a solid material the difference between the Gibbs free energy and the Helmholtz free energy is normally negligibly small. Therefore the word free energy is often used instead of Gibbs free energy.

Generally a deformed polycrystalline metal does not attain the thermodynamically more stable state by a single process. Instead, all of the above-mentioned reactions proceed in turn and may overlap locally and chronologically. If the state of increased free energy is brought about in some other way than by plastic deformation, e.g. by quenching of a single crystal from high temperatures or by particle bombardment, then processes 1) and 2) only will essentially be observed upon annealing. In any particular case, the behaviour will be governed by the type, concentration and arrangement of the lattice defects introduced into the metal by the treatment. These *structural details* determine the decrease in the free energy in the course of each process, i.e. they determine the driving force (change in free energy per unit volume) and the rate of reaction associated with the process under consideration. In pure metals, the changes in the free energy are relatively small, i.e. not more than some few per cent of the latent heat of fusion (c.f. chapter 2). Regarding the rates of reaction, all that can generally be said is that they obey exponential time-laws with activation enthalpies which usually lie between those of lattice and grain-boundary self-diffusion.

In a collective description, processes 1) and 2) are termed *recovery* and processes 4) and 5) *recrystallization* (migration of large-angle boundaries). If the intermediate process 3) contributes to the formation of large-angle boundaries, then to that extent it is also considered as a part of recrystallization. The rearrangement of dislocations leading to the formation of small-angle boundaries (subgrain formation) is sometimes called *polygonization*. In other words, recrystallization is looked upon as the formation and migration of large angle boundaries*. This definition is quite general and incorporates in particular manyphase systems as well. On the other hand, the formation of a crystalline structure from an amorphous phase would not be considered as recrystallization, since the definition of the term large-angle boundary between these two phases would be meaningless.

It was assumed so far that the unstable state is formed prior to annealing. If, however, the unstable state is formed during annealing, e.g. by plastic deformation at sufficiently high temperatures, then one is dealing with the case of *dynamic recovery* or *dynamic recrystallization* (see chapter 9).

The understanding of the processes occurring under such complicated conditions is as yet so incomplete that a systematic classification in the framework of the fundamental processes is not possible.

1.2. Primary Recrystallization

Normally, in the course of the annealing of a sufficiently strongly cold-worked metal, this being the most important case in practice, discretely located nuclei are formed which grow, thereby consuming the deformed structure, replacing it with a new strainfree structure. This process is called *primary recrystallization*. The nucleus comprises a lattice region (of the order of ca. $1\ \mu\text{m}$) that is capable of growth at least in one direction. This region contains a much lower density of lattice defects, in particular dislocations, than the deformed matrix. Such a nucleus is separated from its environment by a newly formed large-angle boundary. In other words, nucleation and primary growth of grains during primary recrystallization correspond to the fundamental processes 3) and 4). The driving force for primary growth of grains results essentially from the difference between the dislocation densities in the nucleus and the matrix (see chapter 2).

Deviations from the normal case of primary recrystallization may be observed after either very strong or very weak cold-working: After strong deformation, the dislocations occasionally

* Occasionally, the formation of large-angle boundaries, i.e. the *nucleation*, is omitted intentionally in the definition of *recrystallization*, since some recrystallization processes occur without nucleation.

rearrange to such an extent during annealing that small regions of low dislocation density which are divided by large-angle boundaries are formed everywhere. A new structure is thus formed in the absence of large-angle boundary migration. Thereby, the texture of the material is essentially retained. This process is called *recrystallization in situ*. In a structural, atomistic sense, this behaviour corresponds to process 3). The phenomenon of the formation of a new structure in the absence of large-angle boundary migration can be studied particularly well in some cold-worked materials that are capable of precipitation. In analogy to the terminology of precipitation phenomena, this special type of recrystallization has been called *continuous* recrystallization, as opposed to *discontinuous* recrystallization, during which *reaction fronts*, i.e. large-angle boundaries, pass through the deformed structure (c.f. chapter 8). After weak deformation, large-angle boundaries that are present from the beginning of annealing may be displaced, leaving a less defect region behind. This process is called *strain-induced grain-boundary migration* and corresponds to process 4) (see chapter 3).

The over-all kinetics of primary recrystallization (in an isothermal experiment) can be described by the recrystallized volume fraction $X(t)$ (t : time). After an initial incubation period, this volume fraction increases sigmoidally as the isothermal annealing time is increased. The time dependence of the recrystallized fraction can often be described by an "Avrami" relationship $X(t) = 1 - \exp(-At^K)$. If a model is assumed for the primary recrystallization, then the *macroscopic* quantities A and K may be described by the *microscopic* parameters of the recrystallization process consistent with the model, such as the nucleation rate and the growth rate. Since the correlation between the respective microscopic parameters and the macroscopic quantities is in general rather complex, analysis of the recrystallized fraction does not normally yield explicit evidence of the fundamental processes involved. Thus the recrystallized fraction and all quantities derived therefrom are suitable in a very limited sense only for a detailed analysis of primary recrystallization.

1.3. Grain Growth

If annealing is continued after the completion of primary recrystallization i.e. $X(t) = 1$, the grain boundaries migrate further through the recrystallized structure, thereby increasing the average grain diameter. These processes are summarized by the term *grain growth*. In a structural, atomistic sense, they correspond to process 5). Phenomenologically, a distinction is made between *normal or continuous* grain growth and *discontinuous* grain growth*. During normal grain growth, the sizes of the individual grains are relatively uniform, whereas in the case of discontinuous grain growth the differences in individual sizes increase as a result of some grains growing more rapidly (c.f. chapter 5, Fig. 3). In other words, the distribution of grain size, and/or shape, remains nearly constant during the first process, whereas in the second case the topological parameters are not invariant. Since in discontinuous grain growth a small number of grains grow at the expense of all others until these are consumed, this case bears a strong resemblance in external appearance to that of primary recrystallization, it is therefore frequently referred to as *secondary recrystallization*. The driving force for grain growth stems from the specific boundary energy of the grains, it is about three orders of magnitude smaller than that for primary recrystallization (c.f. chapter 2). Consequently, grain growth phenomena proceed much more slowly. Furthermore, they can easily be influenced by external manipulations.

The integral kinetics (isothermal experimental conditions) of normal grain growth may be stated without difficulty, if a fixed distribution of grain shapes and relative sizes is assumed

* The terminology used for these subphenomena varies from case to case. The terms *discontinuous grain growth*, *abnormal grain growth*, *grain coarsening*, *exaggerated grain growth*, *discontinuous grain coarsening* and *secondary recrystallization* are used synonymously. The employment of the conventional expression *grain growth* to describe the behaviour defined by process 5) is unfortunate, because it may give rise to confusion with the process of primary growth of grains. The expression *grain coarsening* is a better characterization of the actual state of affairs.

and if furthermore the growth rate of the average grain diameter \bar{D} is assumed to be proportional to the previously mentioned driving force (c. f. chapters 2 and 5). When the average specific grain boundary energy $\bar{\gamma}$ is independent of time, $\bar{D}^2 - \bar{D}_0^2 = K \cdot \bar{\gamma} \cdot t$ is obtained (\bar{D}_0 is the average grain size after complete primary recrystallization; the constant K depends on temperature).

This time-law has been found experimentally in the case of extremely pure metals. Frequently, however, a smaller increase of mean grain diameter with annealing time is observed. There are numerous explanations for this. The *main* reason for this probably is the obstruction of grain-boundary migration by impurity atoms. The hindrance to normal grain growth can reach such an extent that the boundaries can move at a very small number of places only. This is the case of discontinuous grain growth. The reason for such a strong hindrance may be the existence of a very pronounced texture, e.g. the cube texture in f.c.c. metals, or, in the case of alloys, the precipitation of a second phase (see chapter 5). Secondary recrystallization proceeds in a sigmoidal manner with respect to time. Quantitatively the process may be described by an Avrami relation, in analogy to the case of primary recrystallization.

It is occasionally observed that the initial (primary recrystallized) microstructure, on annealing further, first shows typical features of *discontinuous* grain growth. At a later stage *normal* grain growth then takes place. This appears to be the case if the initial size distribution is rather broad. Thus, discontinuous grain growth may often be a necessary intermediate stage in the development towards normal grain growth.

Generally, normal grain growth ceases, when the mean grain diameter has reached a value of the order of the specimen dimensions, because after that the grain boundaries start to become pinned at the surface by thermal grooving (see chapters 2 and 5). If this anchoring effect is small, however, then migration of grain boundaries at the surface can be observed in exceptional cases. The corresponding driving force results from the difference in the surface energy of grains of different orientation. This process is called *tertiary recrystallization*.

1.4. The Influence of Foreign Atoms

In the presence of foreign atoms, the processes occurring during annealing become very complicated in general as a result of the diversity of possible interactions between physical and chemical defects. This has twofold consequences. Firstly, the initially unstable state prior to annealing is not a superposition of the influences of the two defect types. An example for this is, in the case of f.c.c. metals, the change of the deformed structure, when the stacking fault energy is lowered by foreign atoms in solution. In the case of extremely small dispersed particles these may increase the dislocation density, and the homogeneity of the dislocation distribution, compared with the particle-free material. Secondly, foreign atoms modify the fundamental processes listed earlier. Moreover, one must distinguish here between systems which, at the beginning of annealing, contain the foreign atoms either in solid solution, in supersaturation or in the form of precipitates (see chapters 7 and 8). In the latter two cases, the fact that during annealing precipitation and solution processes may occur before, at the same time or after the structural basic processes has to be taken into account as well (see chapter 8).

A partial classification of this variety of combinations is possible in terms of the modification of the driving force due to foreign atoms. Such a procedure is relatively straightforward in the case of the fundamental process 4). The details of this modification depend, of course, on the particular situation: If, for example, during grain boundary migration precipitation occurs exclusively at the boundaries, then an additional driving force of chemical origin results (c.f. chapters 2 and 8). If, on the other hand, thermodynamically stable precipitates already exist in front of the migrating grain boundaries, then a friction force resulting from the particles will act on the boundaries. However, a more complicated situation occurs if a metastable phase is

precipitated out in front of the moving boundaries. Thus the metastable particles can be transformed into the stable equilibrium phase by the migrating boundaries. In this case, the expression for the driving force is supplemented by a transformation term.

With the aid of the time-dependence of the individual driving forces the kinetics of the processes can be classified as well (see chapter 8). The fundamental processes 3) and 5) can also be discussed analogously. With regard to the problem of nucleation, however, this treatment is of a largely speculative nature at the present time, since even in the case of pure metals little details are known about the process of nucleation.

2. BASIC PROBLEMS

2.1. Unstable Initial State

The physical and chemical structure of the initial state determines the recrystallization process. For this reason the starting-point of any analysis of recrystallization phenomena is the question concerning the structure of the unstable initial state, in particular with respect to the structural details relevant to the subphenomena. The answer to this question is generally very unsatisfactory, the underlying reason being that in most cases only mean values characterizing the initial state are known, such as the dislocation density, the texture, the stored energy, the composition and similar quantities. In addition to this, however, a local description of the initial state is required in order to clarify the relevant details. Three simple experimental observations may illustrate this for the sub-phenomenon of nucleation (c.f. chapter 3):

- a) Strained zinc single crystals in which only one glide system has been operated merely show a recovery upon annealing. On the other hand, single crystals deformed by an equivalent amount in multiple slip exhibit recovery and recrystallization upon annealing.
- b) In weakly rolled, polycrystalline aluminium, colonies of nuclei, originating from some intersection lines of the grain surfaces of the initial state, grow into the deformed grains upon annealing.
- c) In a deformed titan-molybdenum alloy the first recrystallization nuclei are observed at the intersections of deformation bands.

From these and related optical microscope observations it was already concluded at an early stage that *local* misorientations in the deformed lattice are a prerequisite for the formation of large-angle boundaries.

The great significance of a local description may be demonstrated for the sub-phenomenon of grain boundary migration on two very recent results (c.f. chapter 4):

- a) By direct and continuous observation of the grain boundary migration of individual boundaries, using the scanning electron microscope, it was discovered that in rolled massive copper single crystals the movement occurs in jerks over distances of some μm .
- b) With the aid of transmission electron microscopy it was shown that the passage of small ordered groups of atoms through a moving grain boundary is very different from site to site.

One of the reasons for these observations is the extremely inhomogeneous distribution of \vee defects in the lattice as well as in the grain boundaries.

In order to elucidate the processes which take place on recrystallization, an accurate microstructural analysis of the initial state is an essential prerequisite; it is for this reason that transmission electron microscopy is the customary technique employed. The great expectations

placed on this experimental technique have not, however, been fully realized. The essential reason is that, with the electron microscopes commonly used so far, only very thin foils could be penetrated with the electron beam. Recrystallization processes in such thin foils, however, differ from those in bulk material (the kinetics are at least slower and probably also qualitatively different). For example, at 100 kV accelerating voltage of the transmission electron microscope, the foil thickness is comparable with the smallest grain diameters. It is therefore rather problematic, if not impossible, to carry out the in-situ investigation of recrystallization processes with this kind of instrument since, for instance, grain boundaries are pinned by thermal grooving. In order to obtain reliable results, a bulk specimen must be annealed from which a thin foil is subsequently prepared. This means, however, that two states of one and the same region (in the bulk material) can never be observed. Therefore, the hitherto employed investigation technique provides at best statistical correlations. This unsatisfactory situation has been remedied by the application of the recently available high-voltage microscopes. It should also be possible to study the nucleation stage of the recrystallization using these instruments.

In spite of these fundamental limitations, many studies of recrystallization have been performed with the aid of the ordinary transmission electron microscopy technique in recent years on materials, ranging from specifically deformed single crystals to complicated multiphase, polycrystalline materials. Still, the question concerning the relevant details in each individual case has seldom been solved unambiguously. In particular, it remains to be decided to what extent the experimental data, frequently interpreted in a different sense by the individual authors, can be generalized. This problem is a consequence of the fact that, among other things, for high magnification only very small sections can be observed. As a result, the critical regions of the material are extremely difficult to find or may even be completely overlooked.

Finally, it should be borne in mind that a good microstructural analysis must also include information on the local foreign atom distribution, perpendicular to the surface for instance, from grain to grain or near dislocation tangles. The various methods of modern local trace analysis, such as Auger spectroscopy, atom probe field-ion spectroscopy and electron energy loss analysis are in principle suitable for this purpose. Although concentrated research is being done in this field, the unstable initial state has as yet hardly been examined more closely using these powerful methods.

2.2. Formation of Large-Angle Boundaries (Nucleation)

The cardinal question of nucleation is: How can a perfect lattice region of another orientation that is capable of growth be formed in a defect lattice? In the attempt to answer this question, the fundamental difficulty is encountered that the experimental data are always obtained on individual objects which are transferred by some means into the range of *perceptibility*. In other words, measurement always registers the *perceptible nucleation* which, however, may be related to a larger or to a smaller extent to growth processes. Experimentally, a distinct separation between the so-called *process of nucleation* and the growth of the nucleus is probably never possible. In the evaluation of the information on nucleation that is contained in the experimental data, this aspect must be taken into account critically at all times, especially when comparison is made with theoretical predictions.

From the observation of the connection between deformed structures and recrystallized regions, the conclusion is that recrystallization nuclei occur preferably in those areas where high stored energy, steep spatial gradient of stored energy and strong lattice curvature exist. Any nucleation model must be able to account for this most important experimental fact.

In the course of time two by nature quite different conceptions of nucleation have been developed. The first kind of model is based on the idea that nucleation is a spontaneous, self-