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# Kinetics of abnormal grain growth in pure iron

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Grain growth after primary recrystallization in unstrained and strained specimens of pure iron is examined during isothermal anneals at three different temperatures (664, 680, 690° C). Undeformed specimens undergo continuous grain growth, while the deformed ones show a stage of rapid discontinuous growth. The peculiar characteristics of the abnormal growth kinetics, and in particular the presence of three well-defined stages of growth, are brought out by plotting, versus annealing time, the ratio between the mean grain diameters of deformed and undeformed samples. The parameters $n$ (exponent of the kinetic equation) and $Q$ (apparent activation energy) are determined.

## 1. Introduction

The processes by which grain size increases in metals after completion of the primary recrystallization may be classified according to two different patterns: (a) *normal (or continuous) growth*, consisting of a relatively uniform growth of all the crystals; (b) *abnormal (or discontinuous) growth*, during which only some larger grains are allowed to grow at the expense of the other smaller ones.

The second pattern, also called *secondary recrystallization*, takes place when particular conditions, such as impurities or slight plastic deformations, interfere with the simpler process of normal growth. In fact, chemical and structural conditions which direct secondary growth processes along either of the two patterns are well established [1–4], as also is the nature of their driving force [2, 5, 6]. However, the statistical features peculiar to normal and abnormal growth are known rather as qualitative hypotheses [1, 2] than as experimentally defined data. Lastly, the kinetics of the process is sufficiently known, both from an experimental [1, 7, 8] and a theoretical point of view [9, 10]; the fitting of data, however, is unsatisfactory and correct results refer specifically to the kinetics of normal growth.

The present work extends the results exposed in a preceding paper [11] giving data obtained during isothermal treatments at three different temperatures. Some conclusions are drawn as concerns the kinetic characterization of the two different modes of growth. Particular attention is paid to the discussion of the kinetics of the discontinuous stage of growth and to its dependence on temperature.

## 2. Materials and methods

Completely recrystallized samples of high-purity iron (> 99.998%) were prepared in the form of 50 mm-long square rods (5 mm × 5 mm cross-section) with a mean grain size of about 50 $\mu$m.

Grain growth was investigated both on samples deformed by tension to about 2, 5 and 10% elongation* and on undeformed ones, during annealing at 664, 680 and 690° C; details on experimental methods are reported elsewhere [11].

Mean grain sizes after each anneal were obtained by measuring the diameter of the equivalent area of the individual grains, using a semi-automatic apparatus (Leitz TGZ3).

## 3. Continuous and discontinuous growth

In Fig. 1 the mean grain diameter of all the

*Precisely: 1.79, 4.99 and 10.66%.

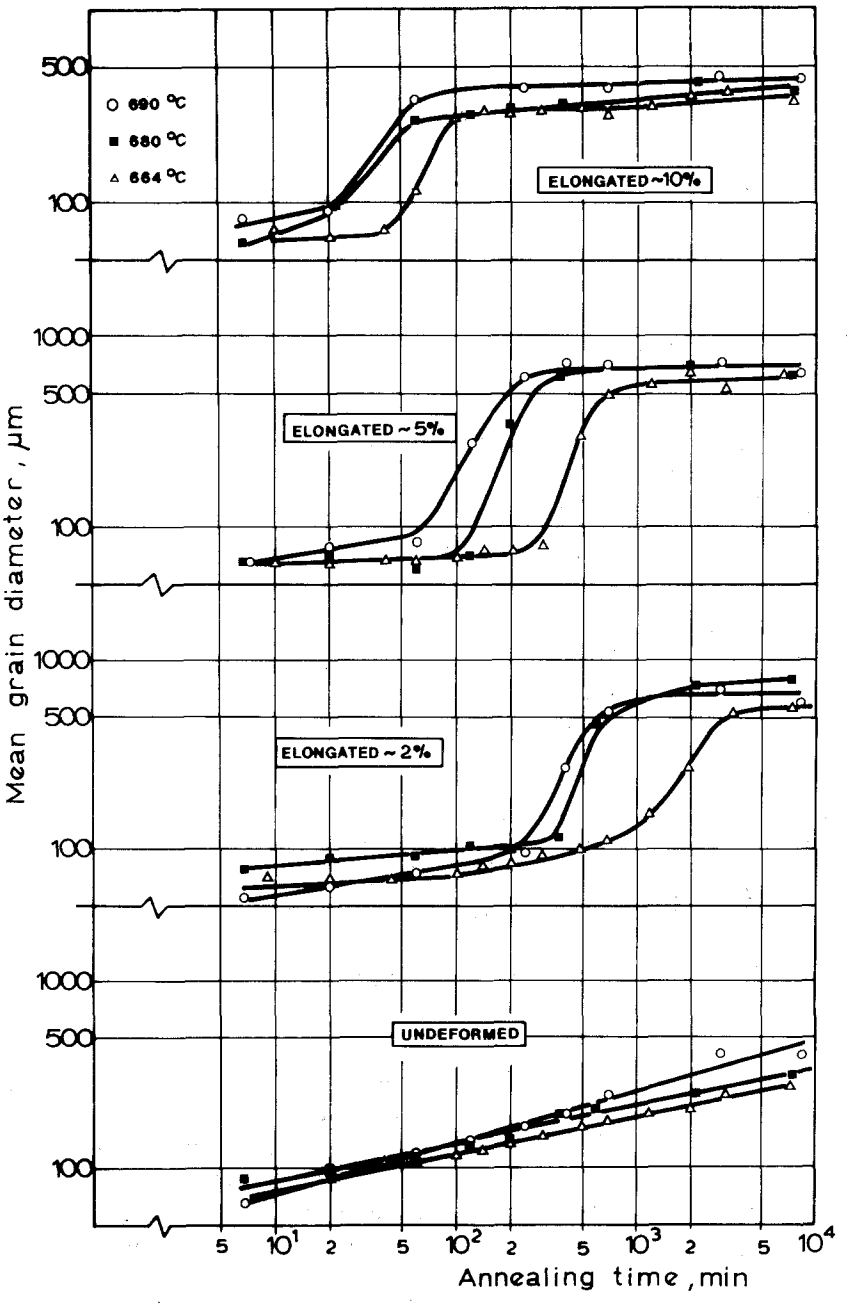


*Figure 1* Mean grain diameter of deformed and undeformed samples as a function of annealing time, during isothermal treatments at 664, 680 and 690° C.

samples for three isothermal treatments is reported, on a logarithmic scale, as a function of the annealing time. Three stages of growth are evidenced for all the deformed specimens: (1) a first period during which a slight overall increase of grain dimensions takes place; (2) an abrupt variation of grain size corresponding to the onset of the abnormal growth for a few grains, while the remaining ones practically maintain their dimensions unchanged, and (3) a final stage where growth is almost blocked, up to the longest annealing times.

The undeformed samples, on the other hand, undergo a continuous, normal growth.

This picture confirms the data previously obtained [11] as regards some typical features of grain growth in slightly deformed specimens after primary recrystallization: (a) whatever the amount of deformation, a stage of abnormal growth is observed; (b) the higher the applied

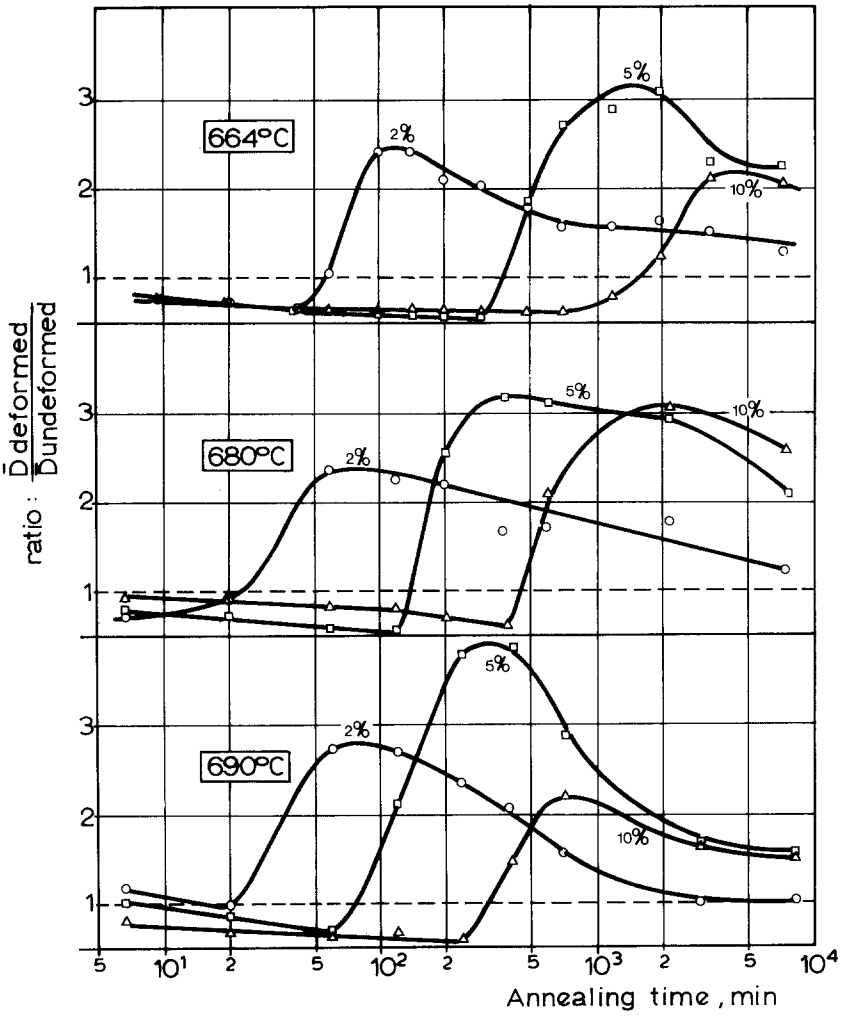


*Figure 2* Ratio between mean grain size of deformed and undeformed samples at the same annealing time during isothermal anneals, as a function of annealing time.

strain, the shorter the time at which abnormal growth begins; (c) for higher strain (10%), lower values of maximum grain size are attained at the end of the process.

The peculiar features characterizing the process of abnormal growth are clearly pointed out in the diagrams of Fig. 2, in which the behaviour of strained and unstrained specimens is compared at the same annealing temperature. The diagrams show the evolution, against annealing time, of the ratio between the mean grain size of each strained sample and the mean grain size of the unstrained specimen at the same annealing time and temperature.

The division into three stages is emphasized here, and Stages 1 and 3 show actually decreasing plots, thus evidencing that, except during the rapid growth of Stage 2, the process of discontinuous growth goes through stages in which rates of growth are slowed down or even stopped.

This fact, as concerns Stage 1, may easily be explained as being due to an inhibition of boundary motion rate due to the formation of a polygonized substructure induced by slight deformation; such an inhibition might be the main factor governing the overall kinetics of the abnormal growth process [4, 13].

The slowing down of growth during the last stage of discontinuous growth is also well established; one can remark that, at higher temperatures, the ratio reported in Fig. 2 tends to reach the value 1 over a long period, i.e. mean grain sizes in specimens grown normally and abnormally tend to coincide again when the process of secondary growth comes to an end.

The stage of rapid growth ends in fact when abnormally grown grains come into contact, smaller grains having practically disappeared, and the mean grain size in the specimen has become ten or more times greater than at the be-

ginning. It is possible that the drastic fall of the driving force (inversely proportional to grain size) below a critical value at which boundary motion can be maintained at an appreciable rate may be responsible for the nearly complete blocking of the growth at Stage 3. Furthermore, the mean grain size attained at the end of Stage 2 reaches the order of magnitude of the thickness of the specimen, thus becoming another important growth-inhibiting factor.

## 4. Kinetics of abnormal growth

An analysis of the commonly accepted relationship between instantaneous mean grain size, $\bar{D}$, time $t$ and temperature $T$ of an isothermal grain growth treatment may start from the assumption that the velocity $V = \mathrm{d}\bar{D}/\mathrm{d}t$ of the process is proportional to the driving force, $p$, through a boundary mobility coefficient $m$ dependent on the absolute temperature

$$V = mp \tag{1}$$

and that $p$ is inversely proportional to $\bar{D}$ through a coefficient $\alpha$ depending on interface energy and on form factors, thus giving

$$\mathrm{d}\bar{D}/\mathrm{d}t = \alpha m \bar{D}^{-1} \tag{2}$$

Integration of Equation 2 leads to

$$\bar{D}^2 - \bar{D}_0^2 = k(T)t \tag{3}$$

where $\bar{D}_0$ is the initial mean grain size, and $k(T) = 2\alpha m$ has the normal Arrhenius-type dependence on temperature

$$k(T) = k_0 \exp(-Q/RT).$$

Experimental checking of the expression (Equation 3), usually made by neglecting $\bar{D}_0$ (which is correct only when growth reaches an advanced stage), leads to the empirical correction of the expression as follows:

$$\bar{D} = k'(T)t^n \tag{4}$$

with $n = 0.5$, and $k'(T) = [k(T)]^n$.

Values of $n \approx 0.25$ are generally obtained for normal grain growth [11–13] and the present work confirms this range of values, even if a certain measure of temperature dependence is indicated ($n = 0.28$ at 690° C, $n = 0.20$ at 664° C for the undeformed samples).

It must be emphasized [14] that only a value of $n = 0.5$ and the use of the Equations 1, 2 and 3 in their correct form enable one to assume a simple mechanism of growth, leading to a meaningful value of the activation energy of the process; this should correspond to the fact that the rate-determining parameter, $p$, would be the *reduction of interfacial energy*, this latter being inversely proportional to the instantaneous mean grain diameter.

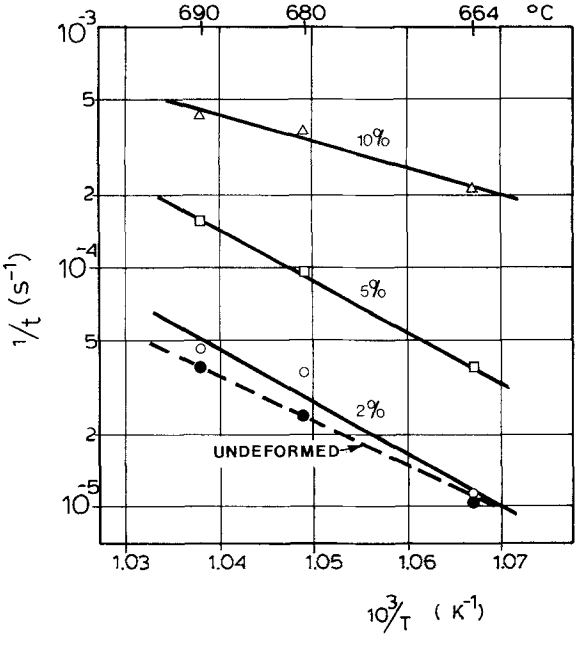


*Figure 3* Arrhenius plot for deformed and undeformed samples at $\bar{D} = 200\,\mu\mathrm{m}$.

TABLE I Apparent activation energy $Q$(kcal mol$^{-1}$) for grain growth in strained and unstrained samples

| Deformation | $Q$ |
|---|---|
| ~ 2% | 101.5 |
| ~ 5% | 96.4 |
| ~ 10% | 50.8 |
| undeformed | 84.8 |

Values of $n$ different from 0.5, which however match experimental data, lead only to apparent activation energy, thus only giving information regarding the temperature dependence of the phenomenon.

On the other hand, the present authors [11] have suggested that a value of $n = 1$ could be regarded as a value with a not merely empirical significance for the stage of rapid growth in the discontinuous process, involving an independence of the driving force of the mean grain size. Values of $n$ ranging from about 0.9 to about 2 have been obtained in the present work (with a mean value of about 1.6) for the stage of rapid growth. Owing to the abrupt character of Stage 2, the spread of the values can be considered acceptable.

Starting from Equation 4 and plotting, against $1/T$, the logarithm of the reciprocal of the time at which a given value of $\bar{D}$ is attained (Fig. 3), one can obtain activation energy data for the process. As the curves are not strictly isokinetic, the limits of validity of the results obtained have been critically mentioned above.

In Table I the values of the apparent activation energy obtained are reported. Two ranges of values may be singled out for the samples undergoing secondary growth: (a) about 90 and 100 kcal mol$^{-1}$ for the unstrained sample and the specimens deformed 5% and 2%, and (b) about 50 kcal mol$^{-1}$ for the more deformed sample (10%). These values seem to indicate stronger temperature dependence for the process of secondary growth compared to the case (10% deformation) in which, as already observed [4, 11], a partial superposition of primary recrystallization may be hypothesized.

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