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A statistical investigation of normal and abnormal grain growth in iron

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The evolution of the statistical distribution of linear grain sizes during isothermal grain growth in unstrained or slightly strained pure iron specimens was investigated. A log normal distribution was confirmed to fit the data well for normal growth, and was shown to be applicable in the initial and final stages of abnormal growth. Analysis of statistical parameters of the size distributions proves that the coefficient of variation $\sigma/\bar{d}$ significantly distinguishes between abnormal and normal growth processes.

1. Introduction

The statistical distribution of grain sizes in metals, and its evolution during secondary grain growth, has received some attention since quantitative metallographic methods have been developed. A certain number of models and theoretical hypotheses on topological, statistical and structural features of growth processes have been advanced [1–6], particularly concerning normal growth, and there has been little experimental work completed [5–11].

Grain size distribution in a massive metal specimen cannot be directly determined since only planar sections of the sample can be examined. At present it is not possible to obtain a spatially correct distribution for metal grains from a planar one; this is only possible for particles (e.g. precipitates) in a matrix, where a spherical shape and no interaction among particles may be assumed, whilst neither of these two conditions can be applied to the grains in polycrystalline materials [9, 12].

Considering only two-dimensional sections of the real grains is of course unsatisfactory, but the size distribution on the section plane can be regarded as significant if the investigation mainly concerns the evolution of grain size distribution during a growth process.

It is known that after completion of primary recrystallization the mean grain size may increase on heating, following two alternative paths (normal or abnormal growth) showing different kinetics [5, 6, 13, 14]. The present work studies the statistical features of grain size distribution during secondary growth, in order to point out parameters characterizing the two different patterns of growth from a statistical point of view.

2. Materials and methods

High purity iron specimens (Koch-Light 99.998%) were prepared in order to achieve a 50 μm average grain size, and then elongated by tensions of 2, 5 and 10%; a series of samples was kept unstrained [13]. Isothermal anneals were then carried out at 664, 680 and 690° C, for increasing times up to 7500 min. Micrographs were taken of each specimen after each annealing. The grain size was determined by means of a semi-automatic image analyser Zeiss TGZ 3, as the diameter of the equivalent circle area. The diameters were grouped in 48 classes (corresponding to the counters of the analyser) according to a logarithmic scale. Some of the grain diameters exceeded the upper size limit of the 48th counter of the analyser and their area was measured with a planimeter. These grains were grouped in extra classes, the limits of which were computed using the same logarithmic scale ratio.

The data so obtained (magnification, class limits, central points and number of events for each class) were used as input information for a computer program which executes the operations reported

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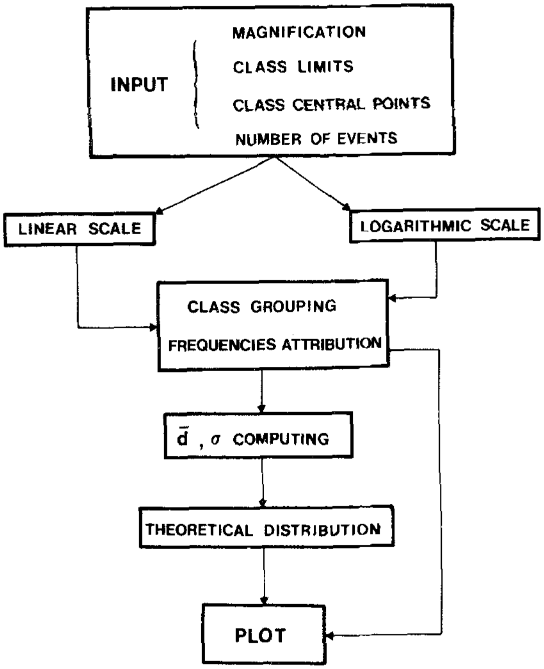

Figure 1 Block diagram for the computing program.

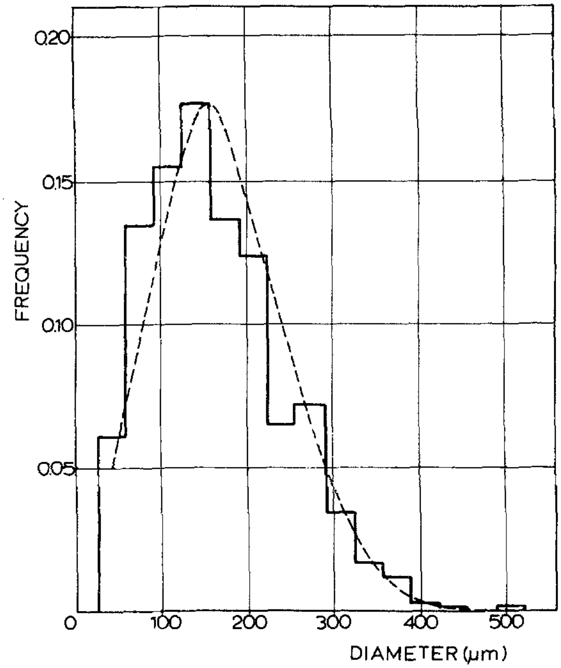

Figure 2 Histogram of grain size frequencies. Unstrained specimen, annealed for 240 min. Total number of grain: 807. Dashed line: the corresponding lognormal function obtained with experimental σ and $\bar{d}$

in the block diagram of Fig. 1. The total number of classes, ranging from 48 to 58, was reduced by grouping the data in 15 to 20 classes according both to a linear and to a logarithmic scale of grain size. The central points of the new classes were computed, and the grouped frequencies assigned to them. The reduction of the number of classes was performed in order to smooth the distribution curves without loss of information.

The mean grain size, $\bar{d}$, and the standard deviation of the distribution, σ, were then computed. Theoretical functions, to fit the experimental frequencies at the central points of the classes, were built up from these parameters. Experimental and computed distributions were then plotted together.

The results pertaining to isothermal treatments at 690° C are reported in the following section. The data obtained at 664° C are 680° C are very similar.

3. Results and discussion

In previous papers [13, 14] the present authors pointed out that the abnormal growth of grains taking place in slightly deformed specimens presents three-stage kinetics: (1) a period of very slight increase in the mean grain size; (2) a rapid increase in the mean grain diameter due to a sudden growth of a few grains (while the dimensions of the remaining grains are comparatively unchanged); (3) a stage of almost blocked up growth. The grains of an undeformed specimen, heated for the same time, grow normally with a regular increase of their mean size.

Grain size distribution data, obtained in the present work after each anneal, and subjected to the statistical analysis outlined above, show asymmetrical right-skewed distribution curves, on a linear scale of abscissae, as can be seen in Fig. 2.

The evolution of frequency curves during secondary growth may be followed in Fig. 3 and 4, showing successive distribution curves obtained at different annealing times. Quite distinct behaviours are evidenced for normal and abnormal growth processes.

In normal growth (Fig. 3) the mean grain size increases while the frequency of the modal value diminishes regularly, thus indicating the continuity of the lowering of the total number of grains. When frequencies are displayed against $d/\bar{d}$, practically all the curves are seen to coincide (Fig. 5).

On the other hand, the evolution of the distribution curves during anomalous growth (see Fig. 4) follows a different pattern: in the first incubation stage the shape of the curves does not

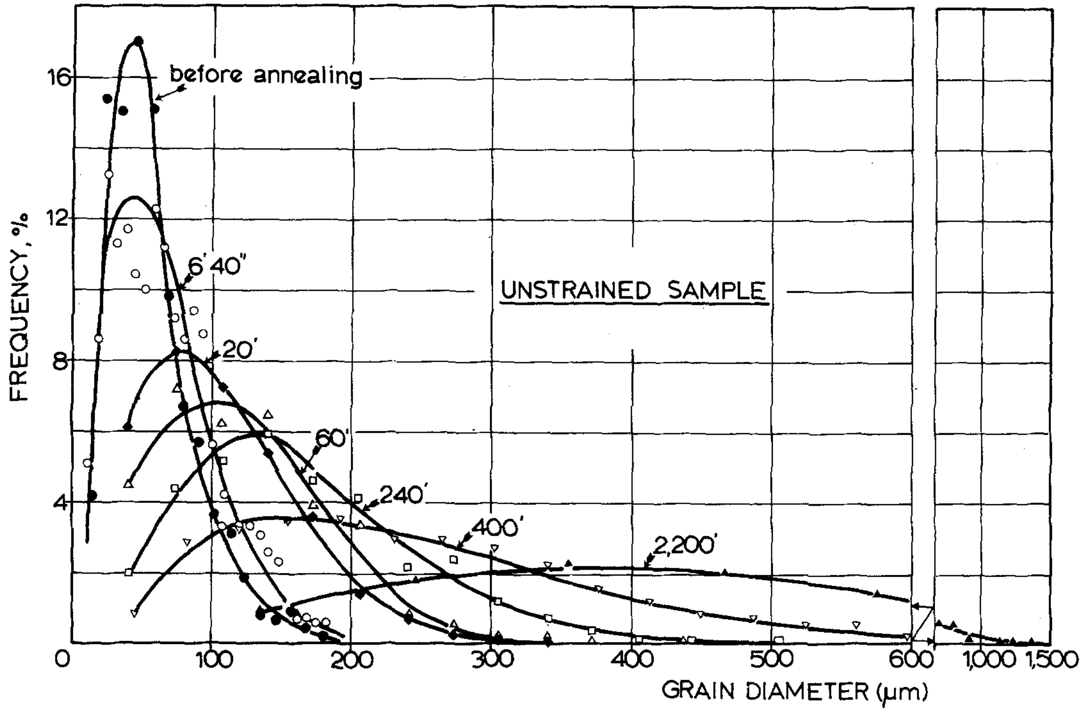

Figure 3 Evolution of grain size distribution at different stages of annealing for the unstrained sample. The points refer to the central values of each class. Total number of grains: from 1400 to 200.

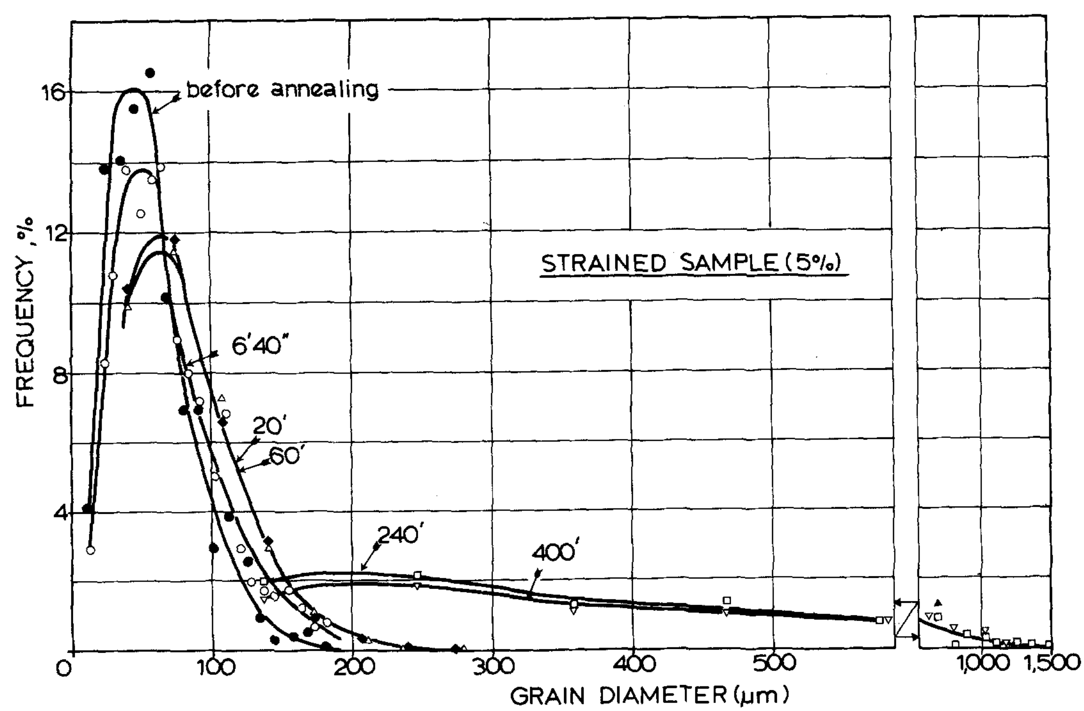

Figure 4 Evolution of grain size distribution at different stages of annealing for the sample strained 5%. The points refer to the central values of each class. Total number of grains: from 1500 to 120.

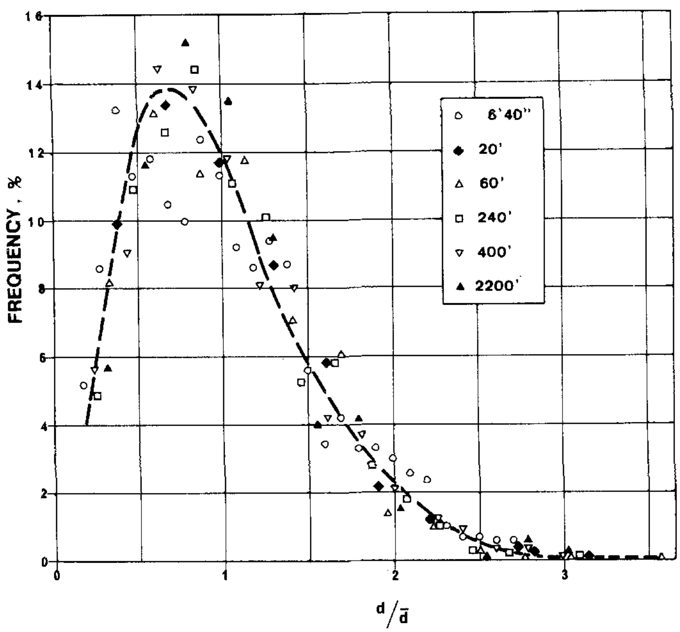

Figure 5 Frequency of grains as a function of $d/\bar{d}$ for the unstrained specimen with different annealing times. Frequencies of Fig. 3 are here normalized to have a constant total area.

substantially change either in relative position or in height. An abrupt shift of the curve in the direction of larger dimensions, together with a fall in the modal value frequency and the appearance of a tail due to larger grains is then observed. When this process is completed, a situation similar to that of the first stage, i.e. normal growth, is again obtained.

The empirical distribution describing the normal growth is found to be lognormal. The fitting of the function

$$f(d) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left(\frac{-[\ln(d-\bar{d})]^2}{2\sigma^2}\right)$$

to data presented in this work confirms previous observations and hypotheses, [2, 9], dealing with normal growth, and allows the extension of the

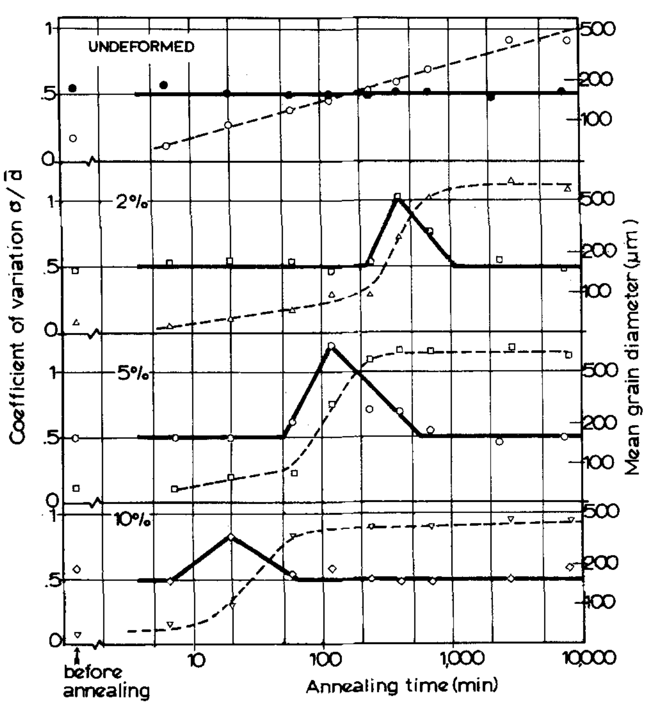

Figure 6 Coefficient of variation ($\sigma/\bar{d}$) of grain size distributions as a function of annealing time for the underformed specimen and those with different amounts of deformation. For comparison, the evolution of mean grain sizes for the same specimens are superimposed (dashed lines).

applicability of this distribution to the first and third stages of abnormal growth. Fig. 2 shows the matching of the experimental points with the function.

A more detailed discussion of the second stage of abnormal growth is necessary. The experimental frequencies are well fitted by the log normal curve over a large number of points, but the extreme frequency values are not in agreement with the proposed functions. It is reasonable to assume that the log normally fitted points refer to the non-growing grains, while the right-skew of the curve is to be ascribed to the rapidly growing grains [15] (although the possibility of it corresponding to the second peak of a bimodal distribution cannot be excluded). Our data are not sufficient to verify such an hypothesis; in fact, since the ratio of the number of the two types of grains ranges between 1:1000 and 1:100, it would be necessary to evaluate the size of between 10^5 and 10^6 grains in order to express the whole range of size distribution correctly.

A more striking quantitative statistical characterization of the two alternative paths of secondary growth may be shown by following the evolution of the coefficient of variation, $\sigma/\bar{d}$, as a function of annealing time. Since σ is the absolute dispersion about the mean value, the coefficient of variation is a measure of the relative dispersion, independent of the units in which the variable (i.e. the diameter after each anneal) is expressed.

Fig. 6 shows that for the undeformed material, undergoing normal growth, the value of $\sigma/\bar{d}$ remains practically constant throughout. In addition, the analysis of frequencies shows that the percentage of grains with diameters higher than $(\bar{d}+2\sigma)$ remains fairly constant ($\sim 3\%$) for different annealing times. This means that the shape of the distribution curve does not change as the mean grain size increases, and indicates that the regular process of grain growth is dependent on a simple mechanism and a unique type of driving force. Moreover, the fairly constant value that can deduced for the coefficient of variation from Fig. 6

$$\frac{\sigma}{\bar{d}} \sim 0.5 \qquad (1)$$

shows a feature of secondary growth that

$$\sigma^2 = \frac{\Sigma_i (d_i - \bar{d})^2}{N-1} \qquad (2)$$

and $d_i^2 = (4/\pi)\, a_i$ (where a_i is the equivalent circle area of the ith grain). It is found, if $N \gg 1$, that

$$\sigma^2 = \frac{4}{\pi} \cdot \frac{A}{N} - (\bar{d})^2 \qquad (3)$$

where $A = \Sigma_i a_i$ is the total area examined on the micrograph.

Combining (1) and (3) gives

$$\bar{d}^2 \sim \frac{16}{5\pi} \times \frac{A}{N} = \alpha \frac{A}{N} \qquad (4)$$

The coefficient of proportionality, α, is a numerical constant, the value of which depends on the way of collecting data; in this case, as equivalent circle areas.

The strained specimens show, Fig. 6, a different evolution of the coefficient of variation $\sigma/\bar{d}$. During the first stage of the process the same value (0.5) as for normal growth is obtained, but, at the onset of the stage of rapid growth, $\sigma/\bar{d}$ starts increasing and reaches a maximum value of ~ 1, then falls back to its normal value after this stage has come to an end.

In this case it is found that the relation (4) becomes

$$\bar{d}^2 = \alpha \frac{A}{N}, \quad \alpha \sim \frac{2}{\pi} \qquad (5)$$

characterizing the stage of discontinuous growth. The different value obtained for α is considered significant, as same technique was used to collect both sets of data. In particular, the lower value of α for the discontinuous process is to be correlated with the different shape of the grain size distribution. In fact the few very large grains on the right side of the distribution have only a slight influence on the mean diameter $\bar{d}$, but represent a substantial contribution to the total area A, thus producing a lowering of the ratio $\bar{d}/A$ for discontinuous growth with respect to the continuous process.

It is remarkable that the same difference in the distribution shape is shown by the different percentage of grains with linear size higher than $(\bar{d}+2\sigma)$: 7% for discontinuous growth, as opposed to 3% for the continuous process.

4. Conclusion

A statistical analysis of grain size frequency evolution during normal (continuous) and abnormal (discontinuous) secondary grain growth in pure iron points out the following main results:

(1) Grain size frequency histograms correspond to asymmetric right-skewed partition curves. A log normal distribution is confirmed to fit well the data for continuous normal growth. It is also valid

for the first and third stages of discontinuous growth. Present results do not verify, or exclude, the hypothesis of, a bimodal distribution for the second stage of abnormal growth.

(2) Grain size frequency curves evolve differently according to whether a normal or abnormal growth process is followed.

(3) The coefficient of variation $\sigma/\bar{d}$, measuring the relative dispersion of grain size distributions, proves to be a statistical parameter able to discriminate between the two alternative paths of secondary growth.

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