ACTA STEREOL 1991; 10/2: 181-189
STERMAT '90
ORIGINAL SCIENTIFIC PAPER

# TECHNIQUES FOR THE MEASUREMENT OF GRAIN SIZE DISTRIBUTIONS DURING GRAIN GROWTH

Brian Ralph
Department of Materials Technology
Brunel, The University of West London
Uxbridge, Middlesex UB8 3PH, U.K.

## ABSTRACT

An overview is given of the techniques and analytical procedures to be preferred for making size distribution measurements. It is demonstrated that detecting the onset of anomalous grain growth requires some sophistication in the data collection and processing procedures.

**Key words: grain size, grain growth, size distribution, thermomechanical processing.**

## INTRODUCTION

The control of grain growth during thermomechanical processing is of very considerable technological importance (e.g. Cotterill and Mould, 1976; Ralph 1990). In most cases, particularly for low temperature strength, the aim is to reduce the rate of grain growth and maintain a fine grain size. However, where the material is to be exposed to the conjoint effects of stress and high temperature (creep conditions) a large grain size is to be preferred.

The process of grain growth also attracts considerable attention from computer modellers (e.g. Anderson, 1986; Doherty *et al.*, 1989) and from theoreticians (e.g. Feltham, 1957; Hillert, 1965; Gladman, 1966; Haroun, 1980). One way of looking at grain growth is that it reflects, albeit in a rather complex way except in bicrystals, the processes occurring during grain boundary migration (e.g. Grant *et al.*, 1984). Further, it is found experimentally, and confirmed by theoretical treatments, that the presence of solutes, precipitates and other dispersoids has a major influence on the grain growth process (e.g. Randle *et al.*, 1986). In addition, there are considerable effects on grain growth from the presence of textural components left over from the initial recrystallisation (e.g. Abbruzzese and Lücke, 1976). Much interest now centres on the relative importance of micro-textural components and the more local orientation space in which a particular grain finds itself (e.g. Randle and Ralph, 1988).

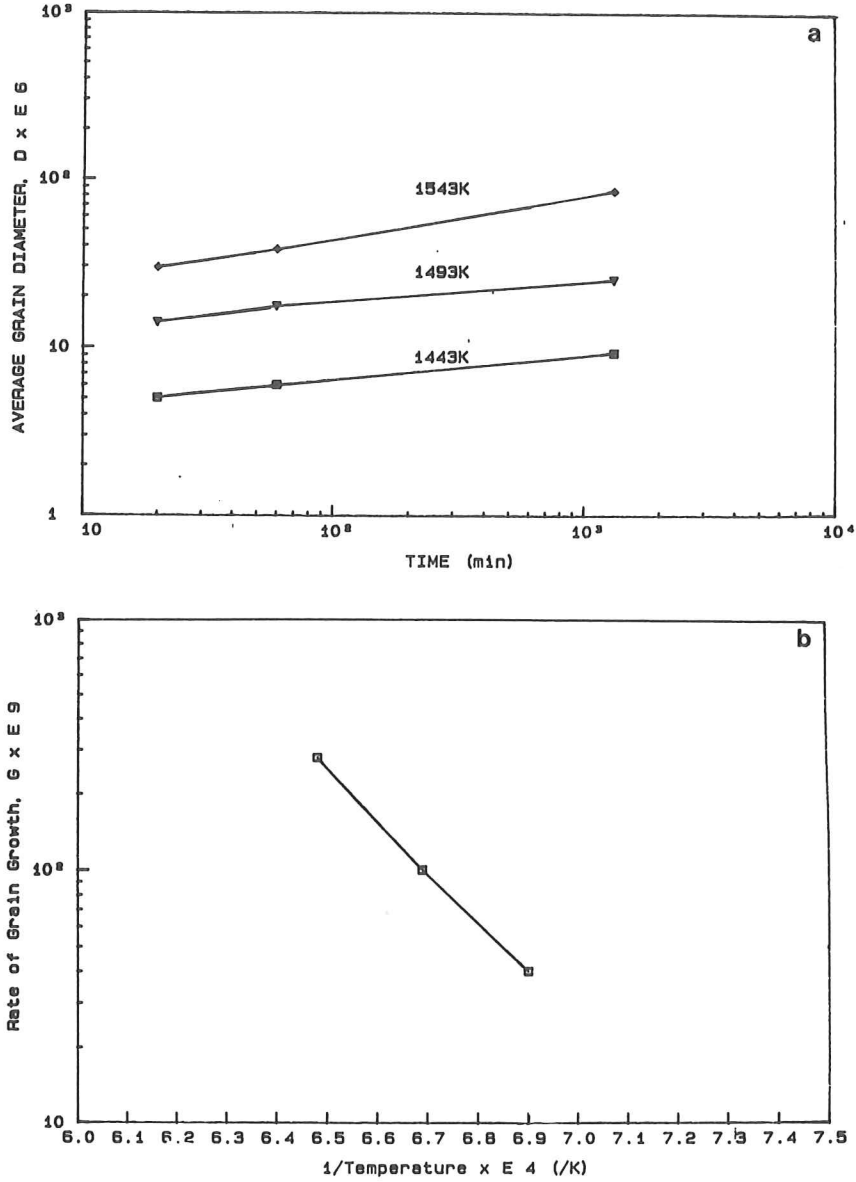


Figure 1. Derived kinetic grain growth data from samples of a powder-formed nickel-base superalloy (APK6) (from Huda and Ralph, 1990).

(a) gives the plots from which the growth exponent has been determined to be ~0.16; (b) gives the Arrenhius plot from which an activation energy of ~ 375 kJ $mole^{-1}$ has been determined.

Grain growth as a process may be investigated experimentally in a number of ways. For instance the process can be monitored by following the topographical changes which occur (e.g. Rhines and Craig, 1974). In other cases, usually to acquire kinetic data, it is often sufficient to follow the evolution of the mean or maximum grain size. This approach may be used to evaluate the growth exponent and the apparent activation energy of the process (see figure 1 and e.g. Huda and Ralph, 1990).

However, much interest centres on the factors which affect the breakdown from normal grain growth (where the distribution of grain sizes is maintained in shape but shifts to larger sizes (see figure 2a)) to anomalous/abnormal grain growth (also often termed *secondary recrystallisation*). In the anomalous/abnormal case an initially unimodal distribution becomes bimodal during the process; that is some grains grow abnormally large and can co-exist or consume the fine grain structure in which they are embedded (see figure 2b).

Whilst an abnormal process can be identified relatively easily by qualitative microscopy, clearly the quantification of this process requires more than the simple measurement of average grain size by mean linear intercept methods. Rather, it is necessary to determine the distribution of grain sizes and how this evolves during the grain growth process. What follows in this short overview is an attempt to present a systematic method of producing the required data. The treatment given leans heavily on the work of Tweed and co-workers (Tweed *et al.*, 1983, 1985).

## GRAIN SIZE MEASUREMENT TECHNIQUES

Conventionally, grain size is measured by some averaging technique which compares against standard graticules (e.g. ASTM system) or makes measurements of some mean sizing parameter such as linear intercept (e.g. DeHoff and Rhines, 1968). In these cases, it is usual either to avoid stereological processing or to use the simplest of transformations.

Where it is deemed necessary to measure the grain size distribution, as in the case around which this presentation is based - that is detecting the onset of anomalous grain growth, very considerable care is needed in all the steps in the data-gathering process. In general these days, to collect statistically significant amounts of data, recourse will be made to automatic image analysis and it is within this framework that the following points are made. The advantages of automatic techniques arise from the ease and accuracy with which large samples of grains may be measured. Further, many parameters may be measured simultaneously, stored within large data bases and then processed in a variety of ways so that different representations of the data may be made (e.g. Tweed *et al.*, 1985).

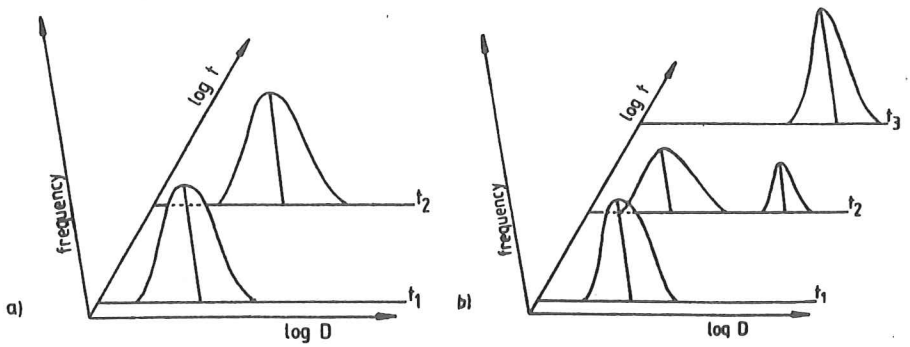


Figure 2. Schematic representation of the change in grain size distribution as a function of annealing time resulting from (a) normal and (b) anomalous grain growth (after Detert (1972) and Tweed (1983)).

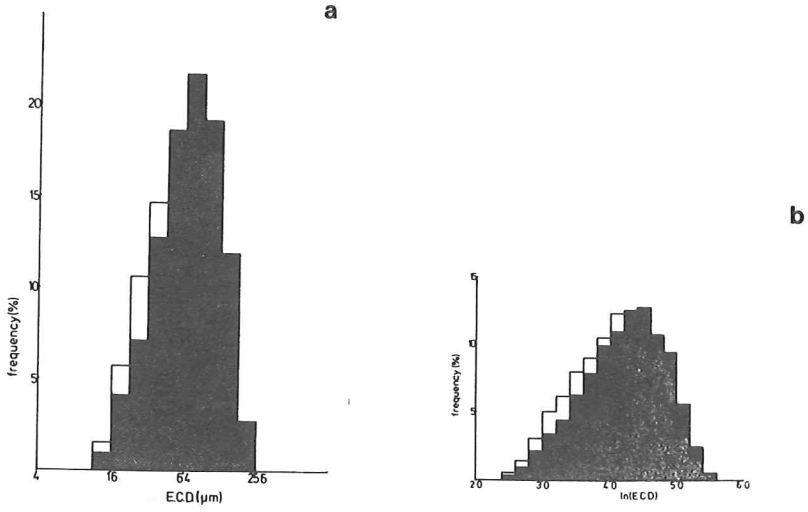


Figure 3. Two-dimensional grain size distribution (plotted as a function of frequency *versus* equivalent circle diameter) for a sample of aluminium containing alumina. In each case, and in figures 4 and 5, the grain size distribution before grain growth is represented by the light region of the histogram and that after grain growth by the dark region. The shaded region represents the region of overlap between the two conditions. These two geometrically scaled plots are of the same data using (a) a scaling modulus of $\sqrt{2}$ for diameter and (b) a scaling modulus of $e^{0.2}$ (from Tweed *et al.*, 1985).

Initially there is a need to choose suitable sample preparation techniques both to ensure an unbiased representation of the parent population (e.g. Exner, 1972) and to produce reliable and systematic contrast. In the case considered by Tweed and co-workers, anodized specimens were viewed in crossed polars by light microscopy to establish grain-to-grain contrast but even then two rotated views were necessary to ensure that each grain was separated. At the end of the day, insufficiently reliable algorithms were available in the image processor used to "detect" all the grain boundaries and a system of tracing the boundaries on to acetate sheets coupled with viewing them on a macroviewer was used (Tweed *et al.*, 1983, 1985). However, improvements in specimen preparation technique combined with ever more refined image processing techniques (using intelligent kernals) may eventually make automatic grain sizing a more reliable possibility.

Another factor which has to be considered is the resolution available within the microscope compared to the smallest feature (grain) which needs to be measured. For instance, in a recent study of grain growth in a powder formed superalloy, it was found necessary to use scanning electron microscopy for the starting populations and light microscopy as the growth process proceeded (Huda and Ralph, 1990).

Initially the data is collected two dimensionally and Exner (1972) provides guidance on the relation between the number of size bins and the number of sizing measurements. Typically in the study made by Tweed and co-workers (1983, 1985) of the order of 5000 grains were measured from each sample. Because the data was collected automatically in these experiments it was possible to compare two-dimensional distributions (and their shifts due to grain growth) of chord size, Ferret diameter and equivalent circle diameter (e.g. Gahm, 1972). In general, the distributions which result are very similar, although there is a preference for the one based on equivalent circle diameter where the grain areas are measured.

Scaling these two-dimensional data presents a number of possibilities. However, in grain growth the major concern is with changes at the extreme ends of the distribution and since a two-dimensional grain size distribution is approximately log-normal (Exner, 1966; Feltham 1957) using arithmetic scaling usually results in loss of detail at one or other of the ends of the distribution. Geometrical scaling is then to be preferred and figure 3 shows a comparison of the scheme recommended by Exner (1972), where a scaling modulus of 2 for diameter was used, with the case of a scaling modulus of $e^{0.2}$ (Tweed *et al.*, 1985). The latter scaling modulus (that is $e^{0.2}$) obviously gives a clearer visual representation of the grain growth process in this particular case.

## STEREOLOGICAL PROCESSING

An impression of the way a grain-size distribution alters during grain growth may be achieved by comparing the two-dimensional distributions before and after growth (as in figure 3b for instance). One advantage of doing this is that the statistical confidence in the data is not degraded by the stereological procedures necessary to generate three-dimensional distributions.

However, much of theoretical development of the process of grain growth is derived for the three-dimensional case. Differences betwee the plane section (2-D) and true spatial distribution (3-D) occur for two reasons (e.g. Exner, 1972):

a) due to the truncation effect - that is a plane section through a grain will normally give a size smaller than its true maximum section;

b) due to the sampling effect - that is the probability of cutting through a particular grain increases as its size increases.

In order to perform a stereological transformation, it is necessary to know or assume a shape for the grains. The interplay of grain size and shape is extremely complex (e.g. Underwood, 1970) and in most cases it is advisable to make an assumption that the grain shape is spherical. A conventional process of using "look-up" tables, essentially "stripping" the distribution back from the largest size class, then gives plots of the form shown in figure 4a. This has been derived from the data in figure 3b, although here, as is quite normal, the bottom two size bins have not been plotted in figure 4a since the calculated frequencies turned out to be negative.

In making comparisons between specimens where from observation it was apparent that in one case normal grain growth had occurred and in the other anomalous growth, it became clear that plots of frequency were inadequate. This is simply because the number (frequency) of large/anomalous grains is very small in a sample exhibiting anomalous grain growth. Accordingly, a better means of data presentation was sought, and found, whereby the volume fraction occupied by a given grain size was plotted instead. Figures 4 and 5 allow these two means of plotting the data to be compared.

## CONCLUDING REMARKS

This overview has concentrated on the techniques adopted to detect the onset and development of the anomalous grain growth process The advantages of collecting the data automatically and thus being able to process and present it in a variety of ways have been demonstrated. In this particular case, it appears that presenting the data in true spatial terms showing the volume fraction of a particular size class is to be preferred.

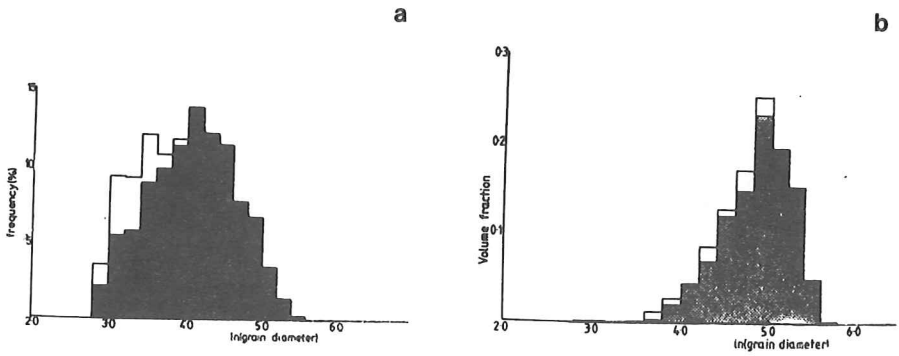


Figure 4. Three-dimensional grain size distributions derived from the data given in figure 3b. Figure 4a gives the spectrum of frequencies of particular grain diameter classes whilst figure 4b gives the same data in volume fraction terms. In this case, normal grain growth is observed to have occurred (from Tweed *et al.*, 1985).

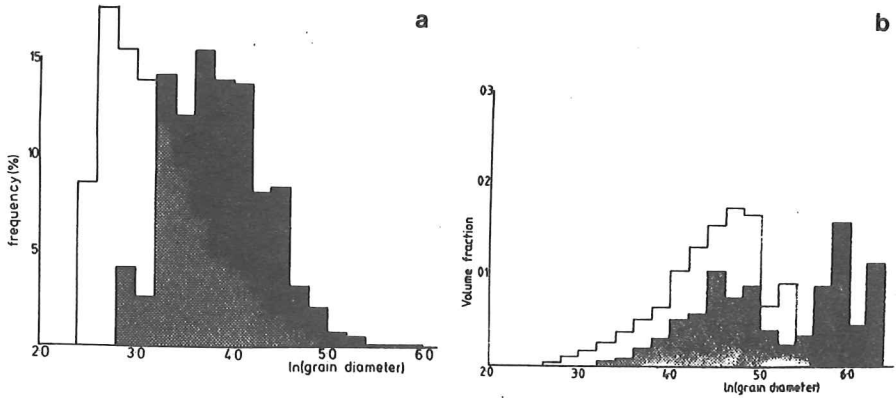


Figure 5. Three-dimensional grain size distributions from a different aluminium/alumina sample where anomalous behaviour was detected. Figure 5a gives the frequency plot of the data where the anomalous behaviour is hard to discern. By contrast, a volume fraction plot of the same data, figure 5b, makes the bimodal nature of the behaviour obvious (from Tweed *et al.*, 1985).

ACKNOWLEDGEMENTS

The author is grateful to Mr. Zainul Huda, Dr. Cherry Tweed and Dr. Niels Hansen for valuable discussions.

REFERENCES

Abbruzzese, G. and Lücke, K. A statistical theory of grain growth including texture and drag effects and its application to texture controlled grain growth. *In*: Proc. 7th Int.Symp. on Met. and Mat.Sci., (eds. N. Hansen *et al.*) Risø Press, Denmark 1976, 1-14.

Anderson, M.P. Simulation of grain growth in two and three dimensions. *In:* Proc. 7th Int.Symp. on Met. and Mat.Sci., (eds. N. Hansen *et al.*) Risø Press, Denmark, 1976, 15-34.

Cotterill, P. and Mould, P.R. Recrystallisation and grain growth in metals. Surrey University Press, London, 1976.

DeHoff, R.T. and Rhines, F.N. Quantitative Metallography. McGraw-Hill, New York, 1968.

Detert, K. Secondary recrystallisation. *In:* Recrystallisation of Metallic Materials (ed. F. Haessner) Dr.Riederer-Verlag GMBH, Stuttgart, 1971; 97-109.

Doherty, R.D., Li K., Kashyup, K., Rollett, A.D. and Anderson, M.P. Computer modelling of particle-limited grain growth and its experimental verification. *In:* Proc. 10th Risø Int.Symp. on Met. and Mat.Sci., (eds. J.B. Bilde-Sørensen *et al.*) Risø Press Denmark, 1989; 31-49.

Exner, H.E. Analyse der Grössenverteilung von Körnern, Poren und Pulverteilchen. Z.Metallkde, 1966; 57: 755-763.

Exner, H.E. Analysis of Grain - and particle - size distributions in metallic materials. Int.Met.Rev., 1972; 17: 25-128.

Feltham, P. Grain growth in metals. Acta Met., 1957; 5: 97-105.

Gahm, J. Stereological measurements with lines, circles and structural standards. J.Miscosc., 1972; 95: 368-373.

Gladman, T. On the theory of the effect of precipitate particles on grain growth in metals. Proc.Roy.Soc. (London), 1966; A294: 298-309.

Grant, E., Proter, A.J. and Ralph, B. Grain boundary migration in single-phase and particle-containing materials. J.Mat.Sci., 1984; 19: 354-3573.

Haroun, N.A. Theory of inclusion controlled grain growth. J.Mat.Sci., 1980; 15: 2816-2822.

Hillert, M. On the theory of normal and abnormal grain growth. Acta Met., 1965; 13: 227-238.

Huda, Z. and Ralph, B. Kinetics of grain growth in powder formed IN-792 - a nickel-base superalloy. Metallography, 1990 (in press).

Ralph, B. Grain growth. Proc. Int.Conf. Microstructure and Mechanical Processing, Institute of Metals, Met.Sci. and Tech., 1990 (in press).

Randle. V. and Ralph, B. Local texture changes associated with grain growth. Proc.Roy.Soc. (London) 1988; A415:239-256.

Randle, V., Ralph, B. and Hansen, N. Grain growth in crystalline materials. *In:* Proc. 7th Int.Symp. on Met. and Mat.Sci., (eds. N. Hansen *et al.*) Risø Press, Denmark, 1976; 123-142.

Rhines, F.N. and Craig, K.R. (with appendix by DeHoff, R.T.) Mechanisms of steady state grain growth in aluminium. Met.Trans., 1974; 5: 413-421.

Tweed, C.J. Grain growth in samples of aluminium containing alumina particles. Ph.D. dissertation, Cambridge University, 1983.

Tweed, C.J., Hansen, N. and Ralph, B. Grain growth in samples of aluminium containing alumina particles. Met.Trans., 1983; 14A: 2235-2240.

Tweed. C.J., Hansen, N. and Ralph, B. Methods of assessing grain-size distributions during grain growth. Metallography, 1985; 18: 115-127.

Underwood, E. Quantitative Stereology. Adison-Wesley, Reading MA, 1970.