THE EFFECTS OF PARTICLES AND SOLUTES ON GRAIN BOUNDARY MIGRATION AND GRAIN GROWTH

B. Ralph, K.B. Shim, Z. Huda, J. Furley and M. Edirisinghe

Department of Materials Technology, Brunel, The University
of West London, Uxbridge, Middlesex UB8 3PH, UK

ABSTRACT

This presentation begins by considering the mechanisms of grain boundary migration and how grain boundaries may be separated into "special" and "general" classes in terms of their migration characteristics. The influence of solutes and particles on the migration process is then discussed. From this initial discussion, the development broadens to consider mechanisms of grain growth and how these are influenced by the presence of solutes, precipitates and other microstructural features. A survey is also given of the factors influencing the breakdown from normal to abnormal (or anomalous) grain growth. The survey of these processes, and factors which influence them, encompasses both engineering alloys and technical ceramics.

INTRODUCTION

Whilst the structure of static grain boundaries has received very wide attention and there is considerable synergism in the theoretical treatments and experimental studies (e.g.[1,2]), the same cannot be said for the structure of a moving/migrating grain boundary. Some studies suggest that elements of the structure of static boundaries are retained in the structure as a boundary migrates but these observations are, at best, indirect (e.g.[3]). Further, the properties (and perhaps therefore the structure) of migrating boundaries appear to vary with the velocity of migration; rapidly migrating boundaries having solubilities and diffusivities some orders of magnitude greater than when they are at rest. Much evidence on this comes from investigations of diffusion induced grain boundary migration (see ref. e.g.[4]). Rapidly migrating boundaries tend to be associated with the recrystallisation stage of an annealing treatment where the driving force for migration is orders of magnitude greater than that associated with grain growth.

From the standpoint of fundamental investigations of grain boundary migration, recrystallisation treatments are less desirable since the crystallographic parameters of the boundary will alter during migration under these conditions. The most acceptable type of study of grain boundary migration is one which is performed on especially prepared bicrystals (see e.g. [4]). A variety of geometries for such bicrystals is possible, each of which

is associated with advantages and disadvantages. Such experiments are difficult to perform and, as a result, only limited studies of this type have been made. In particular, where it has been possible to study the migration of boundaries in a number of pure metals as a function of their geometry, no systematic studies of the influence of solutes and particles have been made by this form of investigation.

This last statement is of extreme importance since solutes and particles are used routinely to control the level of grain growth in many crystalline engineering materials. Thus, at best, the correlation which can be established between fundamental experiments on grain boundary migration in bicrystals and studies of grain growth in practical engineering materials, is low.

Notwithstanding this, the technological importance of grain growth is such that attempts to understand the mechanisms of its control are of extreme importance. A very significant literature exists which develops a wide range of models that attempts to describe various aspects of the grain growth process (e.g.[5-11]). Many of these treatments consider in varying amounts of detail, the influences of solutes (e.g.[12-15]) and dispersed phases of various types (e.g.[16-26]). One particularly useful development in the modelling process has been the use of high-speed computing techniques which allow a great many material variables to be incorporated and tested (e.g.[27-30]). Much current interest centres upon the influences of and changes to the texture during grain growth and how these may be modelled (e.g.[9,10]). Other papers in this symposium look at these aspects in greater detail.

At one stage, the main interest in grain growth technologically was to find ways of stopping or reducing it in order to optimise low temperature mechanical properties or to produce a microstructure amenable to superplastic forming. Whilst this remains an active area of research, there is now an increasing interest in using grain growth to increase grain size; particularly for the case of thin films and to generate coarse grained or single crystal materials for high temperature usage.

GRAIN BOUNDARY MIGRATION

It is becoming common to divide grain boundaries into two broad clases, "special" and "general", depending upon the properties associated with them (such as energy), their structure and the crystallographic parameters which describe them (e.g.[1,2]). *Special* boundaries include low angle grain boundaries and those that are at or near to a high density coincidence structure and accordingly have a short repeat structure in the boundary plane. *General*, or *random*, boundaries make up the rest and there is no real agreement as to the limits (in periodicity or deviations in misorientation) as to how to classify a boundary as *special* or *general/random*.

One property that reflects the degree of "specialness" of a grain boundary is its migration rate. Random boundaries tend to be associated with a higher migration rate than those which are special (as loosely defined above) (e.g.[3]). However, a complicating factor appears in this argument in that boundaries at an exact high density coincidence relationship have higher mobilities than random boundaries in the presence of solutes; the reverse is true when the material is of zone-refined purity. A self-consistent explanation for this has been offered from the pioneering work of Aust and Rutter [12].

For low angle grain boundaries and boundaries close to a high density coincidence, where one aspect of their structure is a periodic array of

intrinsic grain boundary dislocations, their mobilities are more sensitive to the presence of a dispersed phase. This observation arises from a detailed study of boundary mobilities and pinning forces as a function of grain boundary geometry in samples of aluminium doped with low volume fractions of fine alumina particles [31]. Here the finding was that such boundaries were much more strongly pinned than their "random" counterparts in the same microstructure during grain growth heat treatments. The explanation offered for this behaviour was that the pinning was stronger where arrays of grain boundary dislocations made up a part of the grain boundary structure. In attempting to migrate such a boundary past a particle, or array of particles, the network of grain boundary dislocations becomes "caught up" at the interface between the particles and matrix.

One further point to make regarding migration of grain boundaries in the presence of particles, is to acknowledge the much stronger binding by coherent particles. The pinning is so strong that usually grain growth is inhibited. The use of Al_3Zr in high-performance aluminium alloys is but one example where the strong pinning by coherent particles is used to restrict the post-recrystallised grain size and discourage grain growth. The low value for the coherent interfacial energy when compared with the grain boundary energy leads to a driving force for change in shape, from say spherical, of the coherent particles when they encounter a grain boundary [32]. Figure 1 shows the various interfacial energies which determine the shape of a coherent precipitate when it is situated at a grain boundary. The dihedral angles and interfacial energies are related:

$$\frac{\sin \theta_1}{\gamma_{coh}} = \frac{\sin \theta_3}{\gamma_{incoh}} = \frac{\sin \theta_2}{\gamma_{gb}} \tag{1}$$

where γ_{coh}, γ_{incoh} and γ_{gb} are coherent particle/matrix, incoherent particle/ matrix and grain boundary energies respectively. Because $\gamma_{coh} \ll \gamma_{incoh} \simeq \gamma_{gb}$, $\theta_1 \simeq 180°$ and the precipitate morphology approximates to a hemispherical cap. Clearly, it is the ratio of the interfacial and grain boundary energies which is important and thus "random" and "special" boundaries, with different energies, lead to different shapes for particles which are on them (figure 1). In the "random" case, Doherty [23] has shown that $r_{hs} \simeq 1.26 r_s$ where r_{hs} and r_s are respectively the radii of the intergranular and intragranular particles. An immediate consequence of this is to expect and find a coarsening of the particles at grain boundaries with a concomitant dissolution of the matrix particles near to the boundary. This results in an increase in the volume fraction, f, of the grain boundary precipitates and thus even stronger pinning (since as seen below, f/r is the critical pinning parameter). Experimental evidence for this comes from a study of post recrystallisation annealing just below the γ' solvus of Nimonic PE16, [32]. Here, using stereological analysis the volume fraction, f, of grain boundary precipitates was found to increase more rapidly than the size, r, of these precipitates; essentially suppressing grain growth.
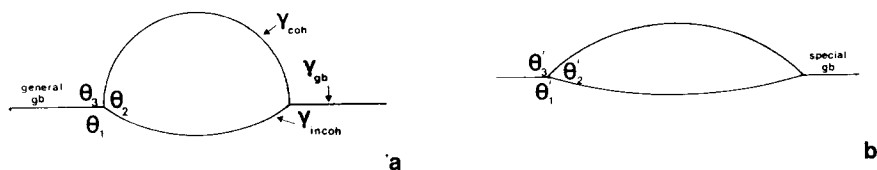

Figure 1. Interplay of interfacial energies for a coherent precipitate on a grain boundary.

GRAIN GROWTH

It is customary to make broad divisions of the grain growth process into normal and abnormal (anomalous or secondary recrystallisation). For the normal case the initial grain size distribution (which is usually log normal in shape) stays the same shape but moves to larger sizes. An extremely large number of models and simulations has been offered for this process (e.g.[5-11], [16-30]); some topographical studies stressing the importance of the shrinkage of small grains (e.g.[22,33]). For the case of abnormal grain growth a small fraction of the grains grow abnormally large whilst the remainder grow much more slowly (i.e. normally) or not at all.

The processes which govern a mechanistic change from normal to abnormal grain growth have attracted very strong interest. One early suggestion was that grains with an initial size advantage (above 1.8 times the average [6]) might be expected to be those which form the basis of the grains which grow anomalously large. However, modelling within a computer makes it clear that a size advantage, as such, is an insufficient criterion, [27]. Rather, a grain which is to appear in the larger of the two "humps" in the bimodal size distribution which characterizes an intermediate stage of abnormal grain growth, must have other advantages. In the bulk, these may include differences in energy/migration characteristics of its boundary (where solutes and particles may play a key rôle (e.g.[7]). Clearly this also suggests a difference in local texture (confirmed in some key experiments (e.g.[34])) and possibly also in macrotexture. The influence of macrotextural components has attracted very considerable interest (e.g.[9,10]); the predictions that result from these studies, from the computer modelling (e.g.[29]) and from measurements of local texture (e.g.[35-38]) are beginning to be seen as growing towards a common picture. One particular experimental study has used neutron diffraction to monitor the changes in macrotexture continuously, electron diffraction to observe changes in local texture in parallel with measurements of grain size distributions, [34]. Grains near to the surfaces of samples will usually exhibit effects which are dominated by the presence of the surfaces.

Under isothermal annealing conditions, normal grain growth tends to follow a relationship of the type:

$$d - d_o = kt^n \qquad (2)$$

where, d, is the average grain diameter at any instant during grain growth, d_o, is the as-recrystallised grain size, t, is the annealing time and n, and k, are constants. The constant k, exhibits an Arrhenius temperature dependence, expressed usually in equations which express the rate of growth, G, in terms of an activation energy, Q_g (e.g.[39]):

$$\ln G = \ln G_o - (Q_g/RT) \qquad (3)$$

here G_o, and R, are constants and T, is the absolute temperature.

The Table gives some examples of n, and Q_g, culled from the literature for ceramic materials. Similar data for metallic materials may be found for example in references [7,8]. In general terms, the maximum value of n, is 0.5 and this is only achieved for ultra-high-purity metals at temperatures approaching their melting points, [60]. At one time, departures from n = 0.5 were taken to indicate effects due to solute drag, particle interactions or textural influences (e.g. [7]). However, results from computer modelling suggest that n < 0.5 is inherent in the growth process, [61].

Table. Experimental values of n and Q for grain growth of some ceramic materials. References are indicated in brackets.

| Material	Additions (pure if not mentioned)	n	Ref.	Q (kJ mol^{-1})	Remarks
ZnO	–	1/3	[40]	224	
	0.5 – 4.0wt.%Bi$_2$O$_3$	1/5	[40]	150	
UO$_2$	–	1/3	[41]	–	
MgO	–	1/2	[42]	–	
	–	1/3	[43]	–	
	0.1 – 0.48wt.%Fe$_2$O$_3$	1/4	[43]	–	
CdO	–	1/3	[44]	–	
CaO	in a synthetic hydroxide-derived	1/5	[45]	333	simultaneous sintering
MgO	dolomite	1/6	[45]	437	
CaO	in Fe$_2$O$_3$-containing dolomite	1/3	[46]	310	simultaneous sintering
MgO		1/4	[46]	330	
CaCO$_3$	0.02 – 10.0 vol.%Al$_2$O$_3$	1/3	[47]	–	
PLZT	–	1/2	[48]	–	
	–	1/3	[49]	361	
AlN	–	1/2	[50]	–	
	CaO,Y$_2$O$_3$	1/2	[50]	–	
CeO$_2$	–	1/2	[51]	–	
	TiO$_2$	1/3	[51]	–	
Yttrium Iron Garnet	–	1/5	[52]	320 308	in oxygen atm in air
MgAl$_2$O$_4$	–	1/2	[53]	462	
Ca$_{0.16}$Zr$_{0.84}$O$_{1.84}$	–	2/5	[54]	–	
YBa$_2$Cu$_3$O$_{7-x}$	–	1/5	[55]	–	at 925, 950°C
	–	1/3	[55]	–	at 970°C
Y$_2$O$_3$ stabilised ZrO$_2$	–	1/3	[56]	550	
CaO stabilised ZrO$_2$	7.5wt.%CaO	1/3	[57]	–	
ZrO$_2$ toughened Al$_2$O$_3$	–	1/4	[58]	–	
TiO$_2$ nucleated β-spodumene	–	1/3	[59]	–	
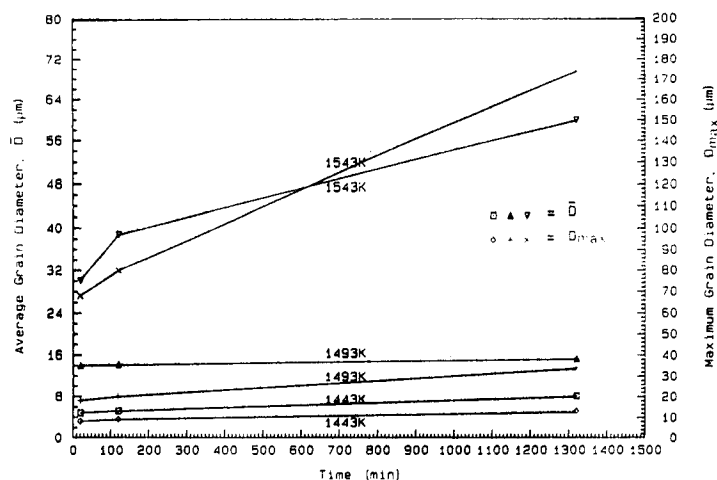
Figure 2. Plots showing the variation of grain sizes with time and temperature for a powder-formed superalloy.

Figure 2 shows some kinetic data from grain growth experiments on powder formed samples of a nickel base superalloy, IN792. The behaviour here is characterized by extremely low n values (*viz.* at 1443K, n = 0.12; at 1493K, n = 0.02; and at 1543K, n = 0.16). It is clear from this figure, and the micrographs on which it is based, that at 1543K (just above the y' solvus for this alloy) the growth process tends to an anomalous form. Essentially in systems like this, the growth process below the y' solvus is extremely slow (in part for the reasons outlined in the discussions to figure 1) and that it is really a duplex growth process involving the coarsening of the y and y' phases. Above the y' solvus (i.e. at 1543K) there is a tendency for the single phase microstructue to coarsen anomalously, at least initially, which is to be encouraged where this material is chosen for engine components to run under conditions where creep is a major concern.

The quoted activation energies for grain growth are usually taken to be consistent with either volume or grain boundary diffusion (e.g.[62,63]). There remains considerable uncertainty over this. Some authors have divided the grain growth processes into four stages in an effort to resolve this problem, [60]. An alternative approach separates normal grain growth into grain drift and grain diffusion processes, [64-66].

The data included in the Table refers to ceramic materials. The increased interest in technical ceramics for engineering applications has led to a considerable expansion in studies of the forming processes of ceramics. In general, sintering tends to accompany grain growth and there is often an interest in reducing the rate of grain growth whilst sintering continues in order to maintain a fine grain size (e.g.[67,68]).

In general, pores and voids will tend to reduce the migration rate of grain boundaries, rather in the same manner as particles. Where porosity is linked by grain boundaries, its elimination may be accelerated by the high-diffusion paths offered by grain boundaries (e.g.[69,70]).

One feature of the ceramic case, not usually met in metallic materials, is the use of liquid-phase sintering. In essence, this is an extreme case of

the influence of solutes which are here added deliberately in order to accelerate the densification of the compact by eliminating its porosity. In this process, the grain boundary regions become highly "alloyed" and, at the sintering temperature, a liquid phase replaces all grain boundaries. At lower temperatures, this solidifies to a glass (e.g.[71]). What is really needed then, in terms of optimizing mechanical properties is to find a way of eliminating this glassy film.

EFFECTS OF PARTICLES

In technological practice, it is common to incorporate dispersed phases or particles to control grain growth. Whilst many models (e.g.[7,8,11,16-26]) and a large number of experimental studies (e.g.[31,32,72]) have been made of the pinning influences of various types of particle, much uncertainty still exists over the interactions between migrating grain boundaries and various types of particle.

Earlier in this text, consideration was given to the strongest form of pinning, namely that achieved with coherent particles (see discussion of figure 1 and refs. [7,8,18,23,32,73]). More usually, it is common to begin a discussion of the influence of particles on grain growth by considering the case of a dispersed phase which has zero solubility in the matrix. This is the case first considered by Zener and cited by Smith [16]. In this treatment, and a whole gamut of variants of it (e.g.[17-26]), the particles are considered to be fixed with boundaries moving through the structure. However, there is an alternative case where the particles may be "dragged" by the boundary (e.g. [74]); this situation is often particularly relevant where sintering accompanies grain growth.

Treatments of the type given by Zener suggest that the pinning force per particle, F, has the form:

$$F = \pi r \, \gamma_{gb} \tag{4}$$

whilst the pinning force per unit area of grain boundary, P, is given by

$$P = \frac{3}{2} \gamma_{gb} \frac{f}{r} \tag{5}$$

where f, and r, are the volume fraction and radii of the particles and γ_{gb} is the grain boundary energy.

Equations of this type may be used to predict a limiting grain size, r_{lim}, at which grain growth will cease:

$$r_{lim} = C \frac{r}{f} \tag{6}$$

where C, is a constant which has been evaluated many different ways (e.g. [6, 7,17,22]). It is generally agreed that equation (6) only applies when f, is small. Computer modelling of the bypass process has suggested an $f^{-\frac{1}{2}}$ dependence for r_{lim} with an explanation of the difference between this and equation (6) relying on the topological connectivity of the migrating grain boundary assembly [75].

The original model due to Zener, and many subsequent modifications to it, ignored many aspects of the boundary/particle interaction; for instance, whether the particle was soluble or insoluble, its shape and the effects due to local boundary curvature near to the particle. Ashby and co-workers [18] and more recently Ringer and co-workers [26] have modelled the bypass process for a

range of situations using soap films. These models take into account the nature of particle/boundary interactions and, whilst confirming the basic ideas of the Zener approach, have an added value in permitting measurements to be made on particle/boundary interactions from which pinning forces may be determined. In one particular case, this has permitted the differences in pinning force between "special" and "random" boundaries to be determined [31].

Another factor has to be allowed for when the particles are precipitates of limited equilibrium during the annealing treatment. Here there is a distinct possibility that the precipitates will either preferentially dissolve (often leading to the onset of anomalous growth) or coarsen. In the latter case, stronger pinning will often result as the particles in contact with the boundary will tend to coarsen differentially at the expense of those in the nearby matrix and the critical f/r parameter will increase as shown in a detailed study of annealing treatments of Nimonic PE16 [32].

OTHER CONSIDERATIONS

Post recrystallisation anneals should be expected to lead to microstructures with a lower internal energy. If one takes a view that the energy of all grain boundaries is the same then it is logical to assume that the grain boundary area will decrease; leading to grain coarsening. However, such a simple picture ignores any variations of grain boundary properties (such as energy and migration rate) with grain boundary structure. Evidence to support the need to consider the properties of grain boundaries as an input into post recrystallisation processes arises from a number of sources. For instance, computer modelling experiments, where the boundary energy is an input variable , show evidence for the onset of an anomalous process [30]. Further, the influence of initial and developing macrotexture also clearly demonstrates, albeit indirectly, the significant influence which the properties of individual boundaries have on the grain growth process (e.g. [9,10]).

Clarification of this in a more boundary-specific manner comes from a series of annealing studies on Nimonic PE16 [35,73]. Where the annealing was performed just above the γ' solvus, it was found that the percentage of "random" type boundaries increased as the grains grew. This was established using electron back scattered diffraction (EBSD) in a scanning electron microscope which allowed the axes of misorientation/angles of misorientation to be determined for grain boundary populations before and after an annealing treatment [35]. The interpretation of this result is straightforward: here the differential migration rates of random over special boundaries are seen and there is a tendency for the microstructure to exhibit an anomalous grain size distribution. It should be appreciated that whilst the overall energy of the system is lowered due to grain growth, the mean energy per unit area of the grain boundaries which remain is actually rising.

In the other case, very long-term annealing treatments were given to the same alloy, below the γ' solvus and no increase in grain size was detected. However, using the same means of measuring the local texture (i.e. EBSD) a considerable increase in the proportion of "special" grain boundaries was detected. The explanation for this comes from the lower energies of special boundaries, compared with the random type. The mechanism for this has to involve a co-operative sliding of boundaries which leads to the larger percentage of boundaries of special type [73].

A current study perhaps demonstrates further complications which will need to be considered in the modelling of post recrystallisation annealing processes. Figure 3 arises from a study of grain growth at 1700°C in samples
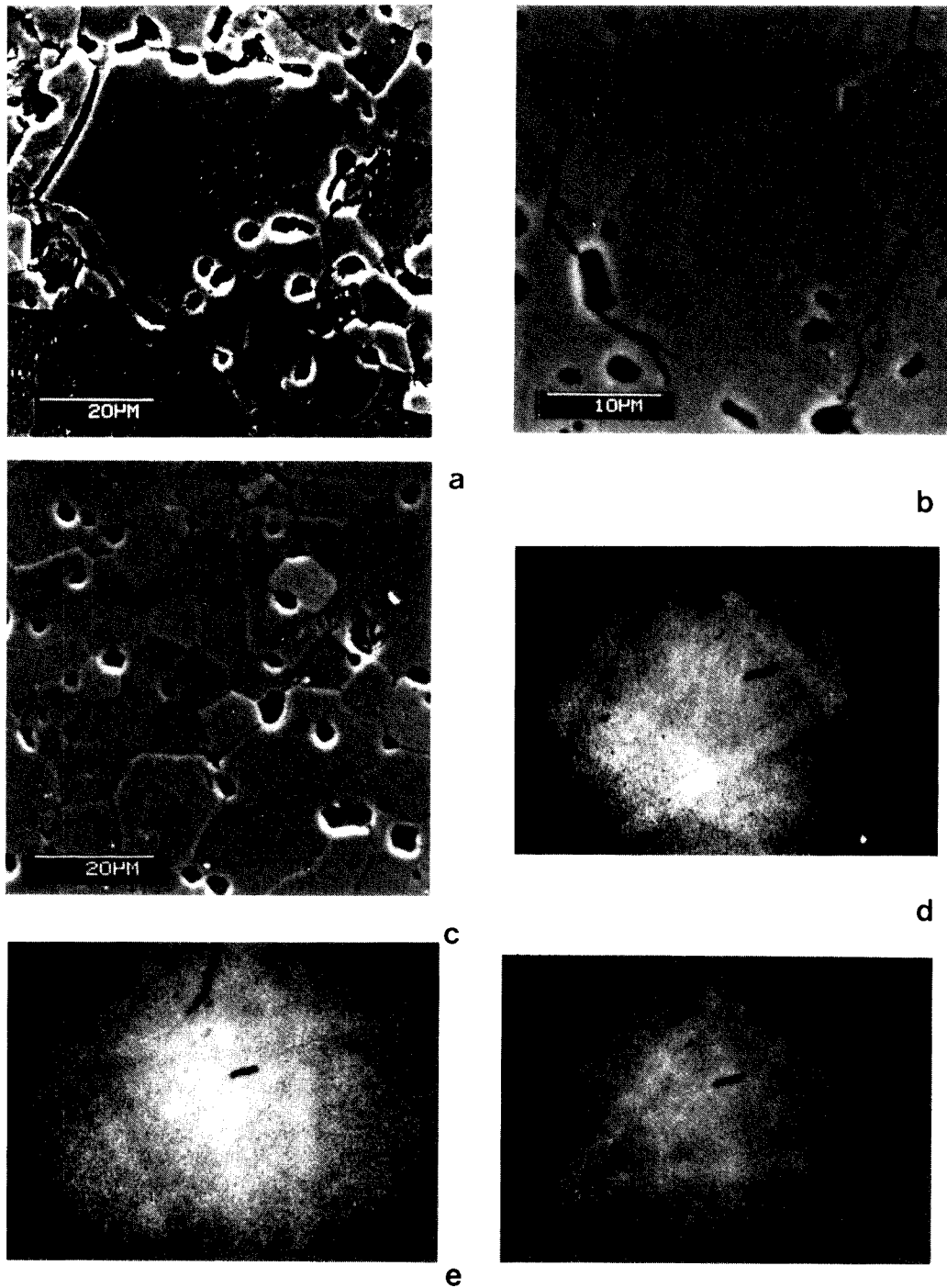
Figure 3. Data from an SEM study of grain growth in TiB_2 at 1700°C:
(a) 0 hours; (b) 2 hours; (c) 10 hours; (d) to (f) are EBSD patterns from
the points marked 1 to 3 on the same grain in (b) establishing poly-
gonization in the bulk.

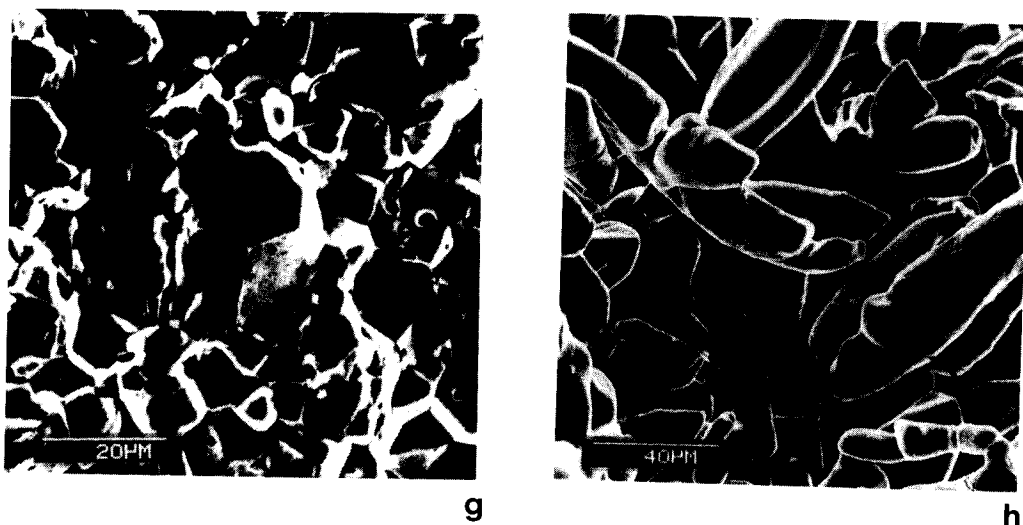g   h

Figure 3 *continued*. SEMs of surface grains after 0 hours (g); and after 10 hours (h) at 1700°C. Subgrain boundaries are seen in the 10-hour treated specimen (h).

of titanium diboride. Light microscopy has been used to establish a very limited pattern of growth (initially $\bar{D}$ = 21 μm, 22 μm after 2 hours and 24 μm after 10 hours). However, scanning electron microscopy using EBSD reveals an added complication in that the grains are becoming polygonized during the grain growth process (figures 3d-f). These polygon boundries are imaged directly on surface grains (compare figures 3g and 3h) whilst it is the EBSD patterns which reveal the polygonization in the bulk. One explanation, at this early stage in the study, might well invoke some grain boundary sliding on certain boundaries to lower energy configurations. Since the material is not fully dense, it may well be that the topological connectivity is insufficiently developed and that the sub-boundaries merely accommodate the misorientation change for one contacting grain boundary as it alters its geometry to a lower energy, special configuration.

OVERALL DISCUSSION

Despite a great deal of theoretical and computer modelling and many significant experimental studies, uncertainties remain concerning the grain growth process. Whilst we know the broad influences of the main microstructural variables (e.g. solutes, particles, texture, presence of surfaces, etc.), our knowledge is far from predictive. In part, at least, this is because an understanding of the grain boundary migration process is far from complete, even in pure materials.

It could perhaps be said that the data base of experience is relatively complete for metals and alloys. However, the complexity of processing ceramics (with the complications of simultaneous sintering, etc.), remains as an area where still more investigations will be needed.

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