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COMBINED *IN SITU* SEM ANNEALING AND EBSD OF DEFORMED MATERIALS

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An SEM heating stage which enables microstructural and crystallographic information to be determined as a deformed specimen is annealed, is described and the application of the technique to recovery, recrystallization and grain growth in aluminium alloys is discussed. It is shown that the development of the recrystallized microstructure and the growth of grains during recrystallization are similar to those occurring in the interior of a specimen. However, the presence of a free surface influences several aspects of annealing, including recovery and grain growth. It is found that annealing twins are formed more frequently at a free surface than in the specimen interior, and the significance of this result is discussed.

KEY WORDS: SEM, annealing, in situ, recrystallieation, grain growth, twins, aluminium.

INTRODUCTION

One of the many contributions which the late Professor Hsun (Bill) Hu made to the subject of recrystallization was the early use of *in situ* annealing experiments in the TEM to observe the recovery and recrystallization of deformed metals (Hu 1962). His results provided experimental support for the recovery mechanism of subgrain coalescence, and have led to extensive subsequent discussion as to the importance of such a mechanism (Humphreys and Hatherly 1995).

Although *in situ* transmission electron microscopy (TEM) or high voltage transmission electron microscopy (HVEM) annealing enables microstructural changes to be observed at high spatial resolution, annealing of very thin specimens has some drawbacks. For example, the presence of two free surfaces is likely to have a substantial affect on recovery, and stress relaxation by bending in thin foils will also influence dislocation behaviour. The advent of good backscattered electron detectors for the SEM and the development of electron backscattered diffraction techniques (EBSD) have meant that imaging and diffraction from deformed and annealed grains and subgrains in an SEM are now straightforward, and although the spatial resolution does not compare with that of a TEM, there are several advantages in using an SEM for *in situ* annealing studies. These include ease of specimen preparation, large areas of specimen, only one free surface, and resistance to specimen buckling. In addition, it is easier to calculate the effects of one free surface on microstructural evolution for an SEM specimen than it is to model the behaviour of a TEM specimen, which is of variable thickness.

Although all *in situ* experiments have drawbacks, their main advantage over the conventional *post mortem* examination of annealed microstructures is that the same area may be examined throughout the experiment and thus the "before" and "after"

microstructures can be uniquely related. The present paper reports some preliminary experiments carried out to assess the applicability of *in situ* annealing in the SEM combined with EBSD.

EXPERIMENTAL PROCEDURE

The heating stage

The experiments were carried out on a JEOL JSM6300 scanning electron microscope. The heating stage was designed such that it should be as far as possible a direct replacement for the standard specimen holder and that is should be capable of operating in either the horizontal configuration or when tilted at up to 70° for EBSD. The construction of the stage was based on a copper block 12 mm diameter and 10 mm deep which is identical to the standard JEOL specimen stub. A 2 mm hole was drilled normal to the axis (see Figure 1), and into this was inserted a non-inductively wound rhenium heating element which was insulated from the block with a mouldable alumina cement. The temperature of the block was measured by a thin chromel-alumel thermocouple inserted into the block above the heating element. In order to minimise electrical interference, heating was achieved by means of a low voltage DC supply. Heat conduction from the block to the microscope holder was minimised by use of a low density ceramic insulator.

The top of the block was recessed so as to hold the specimen, which is in the form of a 3 mm diameter disc of thickness 0.5 to 1 mm, and which is thus similar to the specimen blank from which a TEM specimen is made. A molybdenum spring exerted pressure on the side of the specimen disc so as to ensure good thermal contact with the heating block. All samples were electropolished before use.

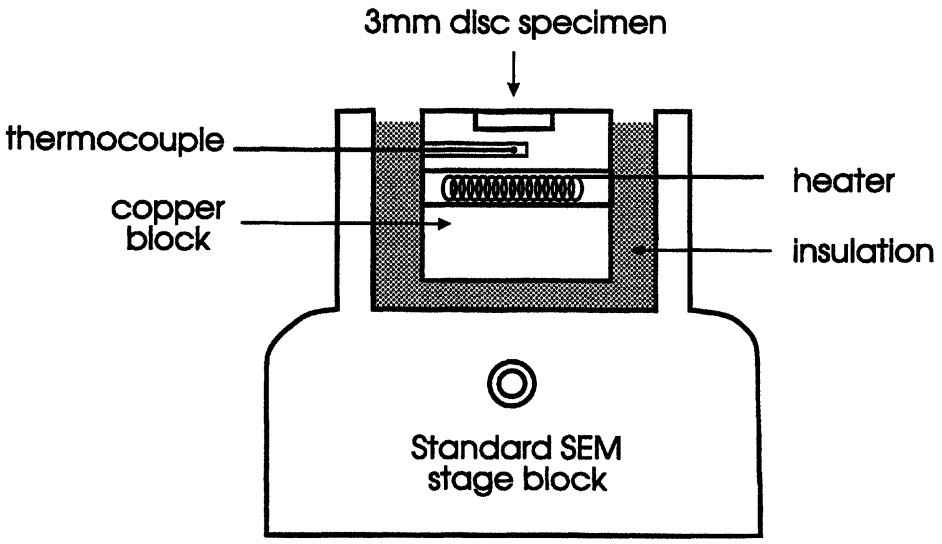


Figure 1 Schematic diagram of the heating stage used for combined annealing and EBSD investigations.

Images were obtained using solid state backscattered (KE Ltd) electron detectors, either a four quadrant detector mounted under the pole piece (for untilted samples) or a 10 mm^2 detector mounted in the forward scattering position (for 70° tilted samples). EBSB patterns were obtained with an ISIT camera (Custom Cameras Ltd) employing frame averaging and background subtraction and the patterns were analyzed automatically on-line with Schmidt (HKL Software) software.

Image recording was carried out either by normal photographic methods or by video recording. A U-matic video recorder was used to record images from the microscope image framstore. Real-time images, which were somewhat noisy, could be obtained with the backscattered detector amplifier operating on low gain. The best backscattered images were obtained by operating at a scan speed of 30 s per frame, although the resultant videotape then contained a sequence of slowly updated frames rather than real-time video. Provision of a sound track enabled the video sequences to be annotated.

Performance

The stage was designed to optimise isothermal annealing experiments, having a sufficient heat capacity to ensure that temperature fluctuations were minimised, being no more than ± 1°. The maximum power was normally kept constant during a run and heating and cooling rates at ~300°C were 1.3°C s^{-1} and 0.4°C s^{-1}.

The total backscattered signal from a particular region of a specimen is a function of both temperature and electron accelerating voltage. As the crystallographic or channelling contrast in a backscattered image is very weak, typically ~5% (Newbury *et al.*, 1986), we have carried out experiments to determine the optimum contrast conditions at elevated temperatures.

In a specimen of recrystallized aluminium, two grains which gave the lightest and darkest contrast were selected and the backscattered signal from small areas within these grains was measured at operating voltages of 10 keV and 20 keV. The specimen was then heated in steps of ~50°C and the measurements repeated. The backscattered electron signal from the two grains, average over several runs, is shown in Figure 2 and is seen to rise sharply above ~300°C. The crystallographic contrast between the two grains, calculated from the backscattered signal is also shown in Figure 2. It may be seen that although the total backscattered electron signal increases with temperature, the increased emission is not sensitive to crystallography, and the useful specimen contrast decreases significantly at higher temperatures.

Very similar results were obtained at both 10 and 20 keV accelerating voltages. However, at high temperatures and lower accelerating voltages, topographic features in the image were less visible and therefore the crystallographic contrast image was much improved as shown in Figure 3. In consequence of this, all results reported in this paper were obtained at an accelerating voltage of 10 keV. It was found that the quality of EBSD patterns was little affected by the temperature of the specimen and good patterns have been obtained at temperatures of up to 400°C on aluminium.

APPLICATIONS

Following preliminary investigation of the performance of the stage, annealing experiments were carried out on deformed copper and various aluminium alloys at temperatures up to 400°C. In this paper, we report only the annealing experiments on

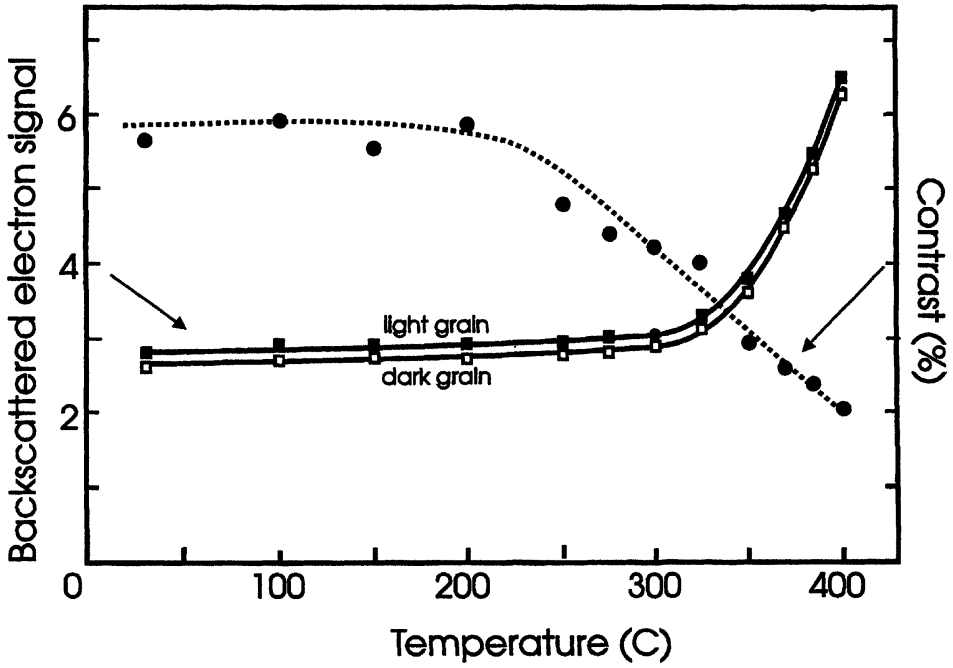


Figure 2 The effect of specimen temperature on the backscattered electron signal and contrast from two grains (10 keV).

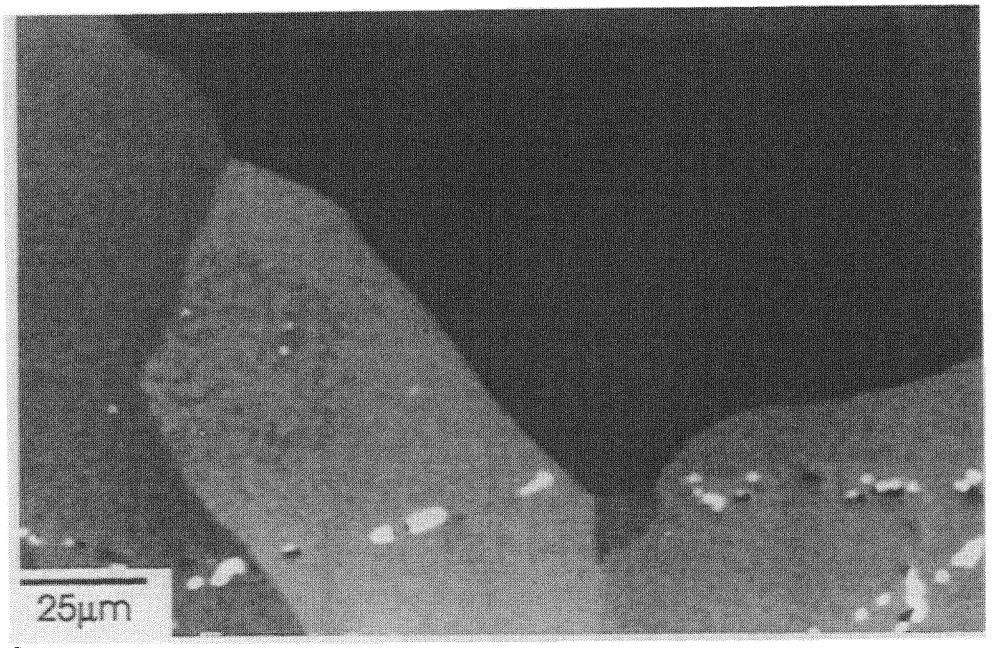
aluminium alloys. As the bulk annealing behaviour of several of these materials has been the subject of research in our laboratory, we have been able to compare the results of the *in situ* annealing with those from bulk annealing.

Recovery experiments

We have recently reported subgrain growth rates in bulk samples of high purity aluminium polycrystals (Humphreys and Humphreys 1994) and in {110} <100> crystals of A1-0.05% Si (Ferry and Humphreys 1995). Using deformed specimens from these investigations we have carried out *in situ* annealing experiments in the temperature range 250°C to 400°C. Preliminary experiments showed that it was very difficult to induce *in situ* subgrain growth in either of the two materials, and it was found that subgrain growth rates were much lower than those in the bulk material. It is suggested that the proximity of a free surface, whose energy is some 5–10 times that of the low angle boundaries (Murr 1975) has a strong influence on subgrain migration, and that the results of such *in situ* recovery experiments should be treated with caution.

Grain growth rates during recrystallization

The technique is ideally suited to studying the effect of orientation on the growth rates of grains during recrystallization. The mobility of grain boundaries during recrystallization is not well understood and is currently a limiting factor in our understanding of annealing (Humphreys and Hatherly 1995). Experiments in which the



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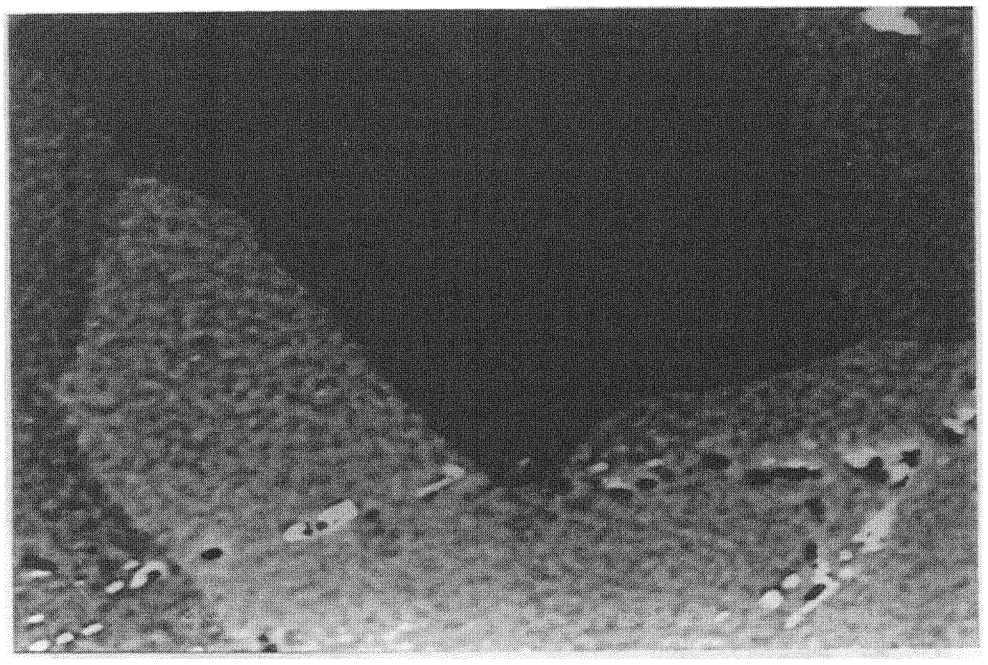


Figure 3 The effect of accelerating voltage on the quality of the backscattered electron image in recrystallized AA1050 at 300°C. a) Accelerating voltage 10 keV. b) The same area at 20 keV with a similar level of crystallographic contrast, showing increased contrast from surface topography.

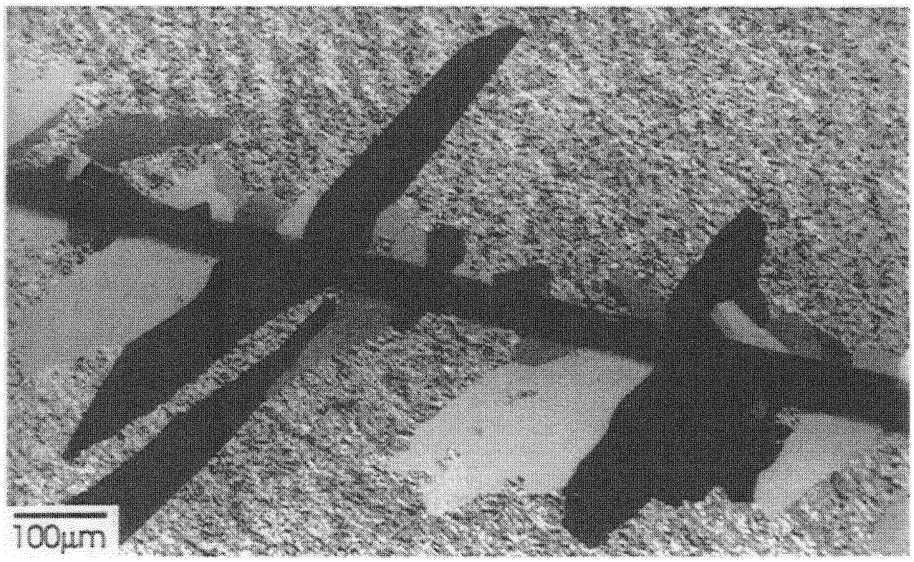
propagation of artificially nucleated grains is measured, have been used for many years [e.g. (Uohara *et al.* 1958, Gottstein *et al.*, 1978) and have led to some understanding of the orientation dependence of boundary mobilities (Humphreys and Hatherly 1995). However, using EBSD and *in situ* annealing, such experiments may be carried out very rapidly, enabling the effects of parameters such as composition and temperature to be investigated. Figure 4a shows a single crystal of Al-0.05% Si of orientation {112}<111> which was deformed to a true strain of 0.7 in plane strain compression. After electropolishing, the surface was scratched and the specimen was then annealed *in situ* in the SEM at 275°C. It may be seen that grains have nucleated only at the scratch and that they have propagated into the deformed material, many of the faster growing grains having a platelike morphology. The orientation of these grains and of the deformed matrix were measured by EBSD and typical results are shown in Figure 4b. As expected from previous work (Gottstein *et al.* 1978), the fastest moving boundaries are tilt boundaries with an orientation relationship of 35–45° about a <111> axis to the deformed matrix. Grains with misorientations of less than 30° about other axes were found to grow very slowly at all temperatures.

The experiment was repeated at temperatures of 250°C and 300°C and the growth rates of plate-shaped $38 \pm 2°$ <111> grains, measured from videotape recordings, are shown in Figure 4c. Analysis of the data for these fast growing grains gives an activation energy of ~140 kJ/mol, which is close to that for self diffusion in aluminium.

Evolution of microstructure during recrystallisation

Annealing of AA1050

Specimens of AA1050 cold rolled 50% and 75% have been annealed in the SEM. Sections parallel to the rolling plane and to the transverse plane have been examined. Specimens deformed 50% were found to start to recrystallize at ~300°C and those deformed 75% at ~260°C. These recrystallization temperatures are similar to those found in bulk annealed samples.



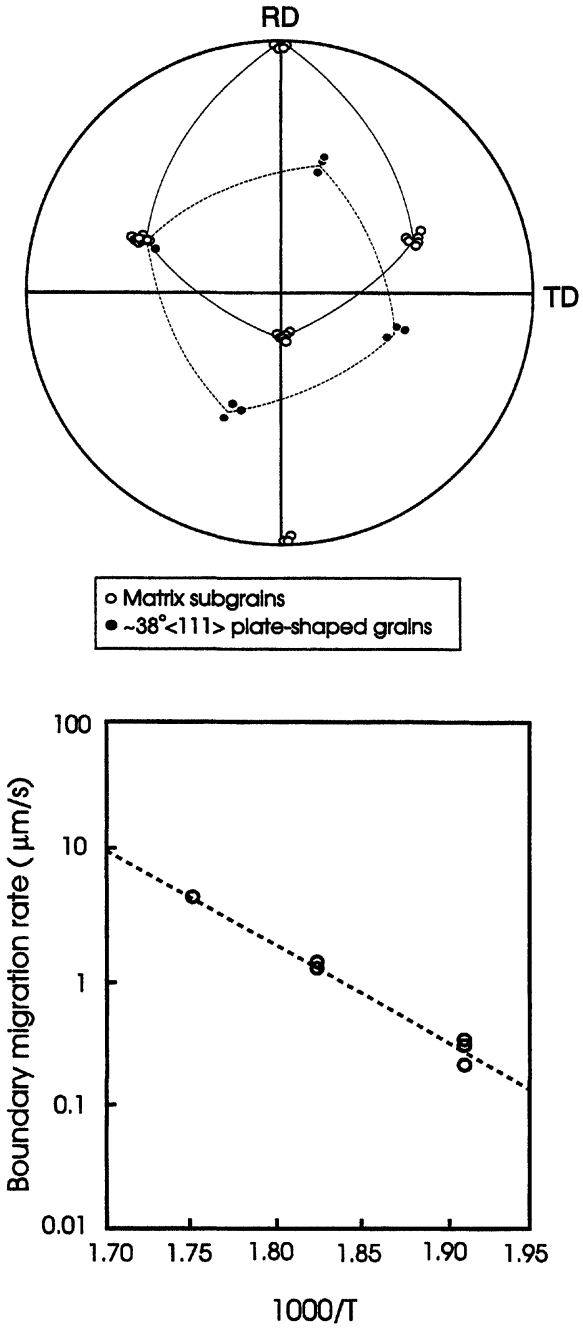


Figure 4 Growth, during *in situ* SEM annealing, of artificially nucleated grains in a {112}<111> Al-0.05%Si crystal deformed to a strain of 0.7 in plane strain compression. a) Backscattered electron micrograph of partly recrystallized specimen, b) Orientations of matrix and fastest growing platelike grains. c) Effect of temperature on the growth rate in the fastest growing direction of 38 ± 2° <111> misoriented plate-shaped grains.

Figure 5 shows part of an annealing sequence for a rolling plane section of a 50% deformed sample. In Figure 5a recrystallization has just started, and in particular we note the large grain A and the smaller grains B, C and D, which are associated with large second-phase particles and which have probably nucleated by particle stimulated nucleation (PSN). Figure 5b shows the specimen when it is some 90% recrystallized. It may be seen that whereas grain A has grown extensively, grains B and C have remained small and grain D has been engulfed by a faster growing grain. Such sequences (see also those in (Humphreys and Ferry 1995) are typical and show the large variation in grain growth rates within a single deformed grain. Limited EBSD results indicates that in many cases the faster growing grains have misorientations with respect to the deformed matrix approximating to a 30–50° rotation about an axis within 10° of <111>.

Annealing of TD section speciments allows a better correlation of the recrystallization with the deformed grains as seen in Figure 6, and in particular, the inhomogeneity of recrystallization is clearly seen. For example, grain A has almost completely recrystallized before grain B has begun to recrystallize. Such observations are consistent with experiments on bulk annealed material (Hjelen *et al.*, 1991) and indicate that the *in situ* annealing reproduces many features of recrystallization in the bulk.

Annealing of a bimodal Al-Si alloy

The recrystallization behaviour of an Al-1.6% Si alloy containing both large and small particles has been investigated. The alloy was heat treated to produce a dispersion of particles of diameter ~10 μm and also a dispersion of particles of initial diameter ~0.1 μm. The heat treatment, particle content and recrystallization behaviour of bulk specimens of this alloy have been previously investigated (Chan and Humphreys 1984). The large particles act as the sites for recrystallization by PSN and the dispersion of small particles retards recrystallization by pinning the boundaries.

On *in situ* annealing a specimen which had been cold rolled 60%, no recrystallization occurred until 375°C. This is some 100°C higher than would be expected for a similar alloy without the small particles. The small particles coarsen as the specimen is annealed, and at ~375°C the particle pinning pressure, which is proportional to F_V/d (Humphreys and Hatherly 1995), is reduced sufficiently to allow recrystallization to start. It is found that although all nuclei apparently originate at the large particles, only a few grains grow rapidly (e.g. A in Figure 7) and that other grains (such as B in Figure 7) grow very slowly. The resulting grain size is very large, and in many cases isolated (island) grains remain. Such microstructures are also characteristic of bulk annealed samples (Chan and Humphreys 1984). It is found that such alloys often develop a strong recrystallization texture (Daaland and Nes 1994) and this has recently been interpreted as an orientation dependence of the particle pinning presence (Humphreys and Ardakani). The *in situ* EBSD results of the present investigation confirm this. For example in Figure 7, grains A and B are growing within the same deformed grain. The slowly growing grain B has an orientation relationship of ~17° <011> to the deformed grain, whereas the fast growing grain A has an orientation relationship of ~42° < 456>, which is close to a high mobility orientation.

Grain growth following recrystallization

Low temperature instability of grain structures

The stability of recrystallized microstructures during *in situ* annealing has been investigated. Specimens of a single-phase Al-0.05% Si alloy were deformed 80% and

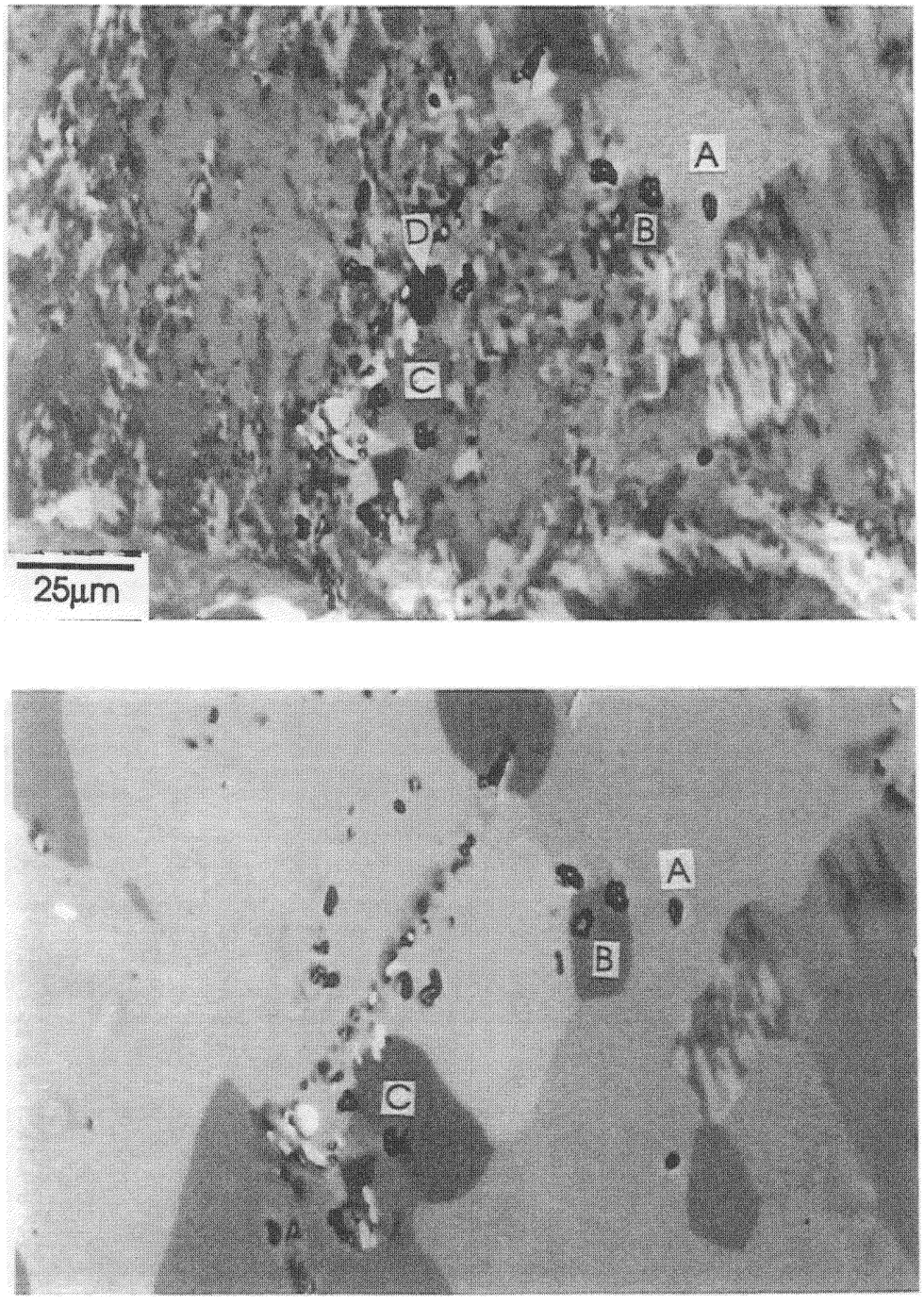


Figure 5 Part of an annealing sequence of AA1050 deformed 50% (rolling plane section) showing non-uniform grain growth.

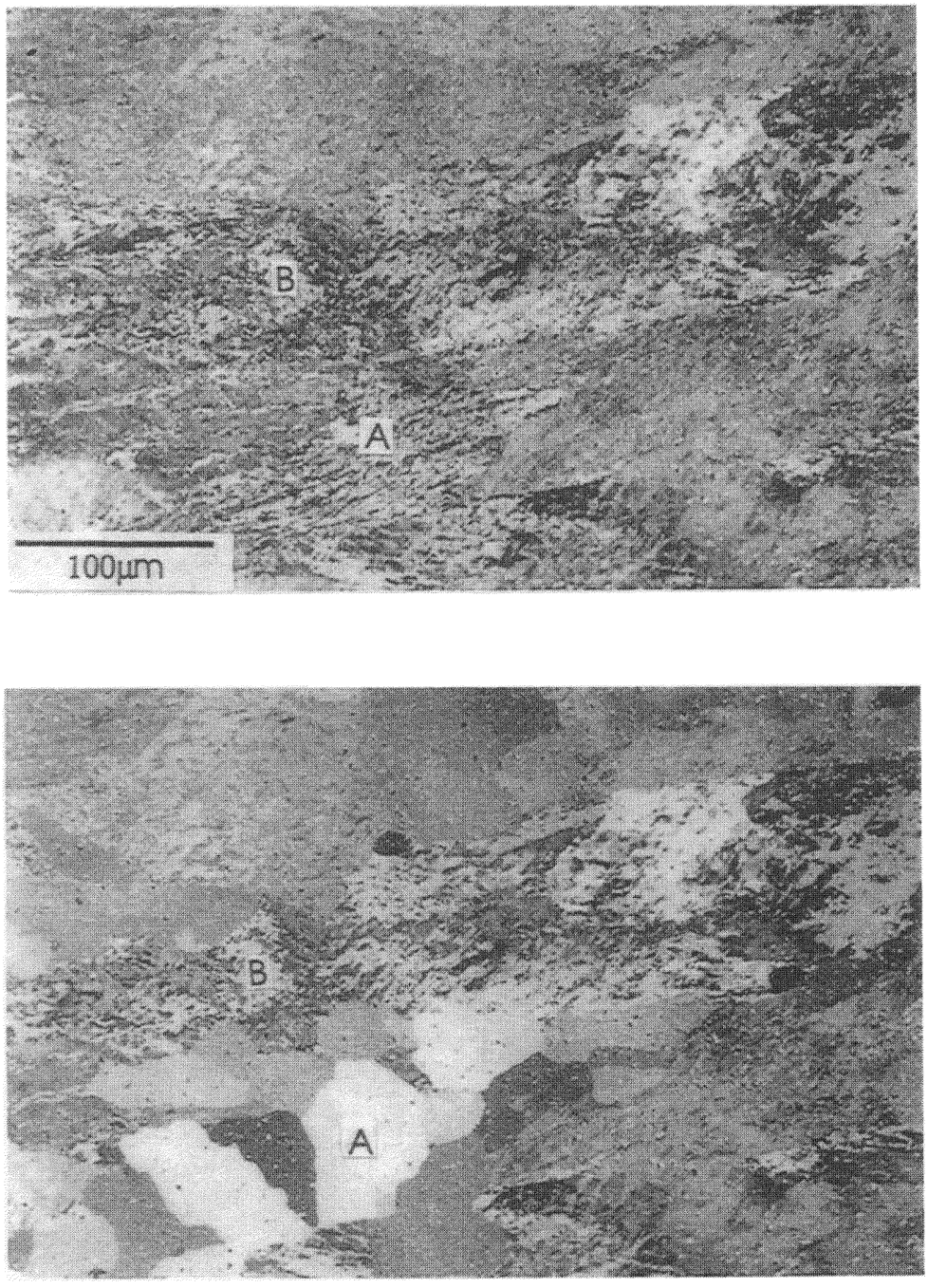


Figure 6 Annealing of the TD section of AA1050 deformed 50% showing the different rates of recrystallization of grains A and B.

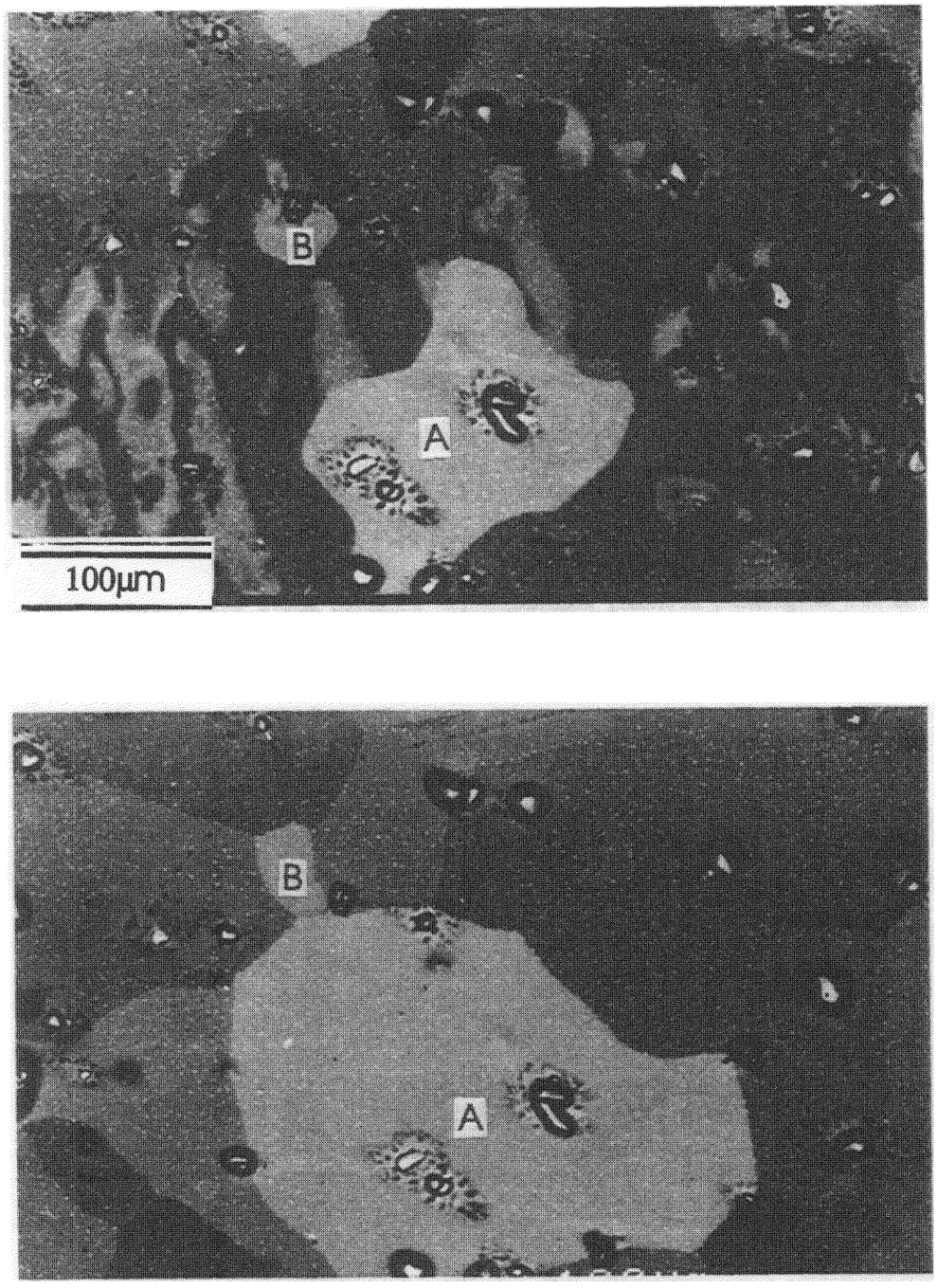
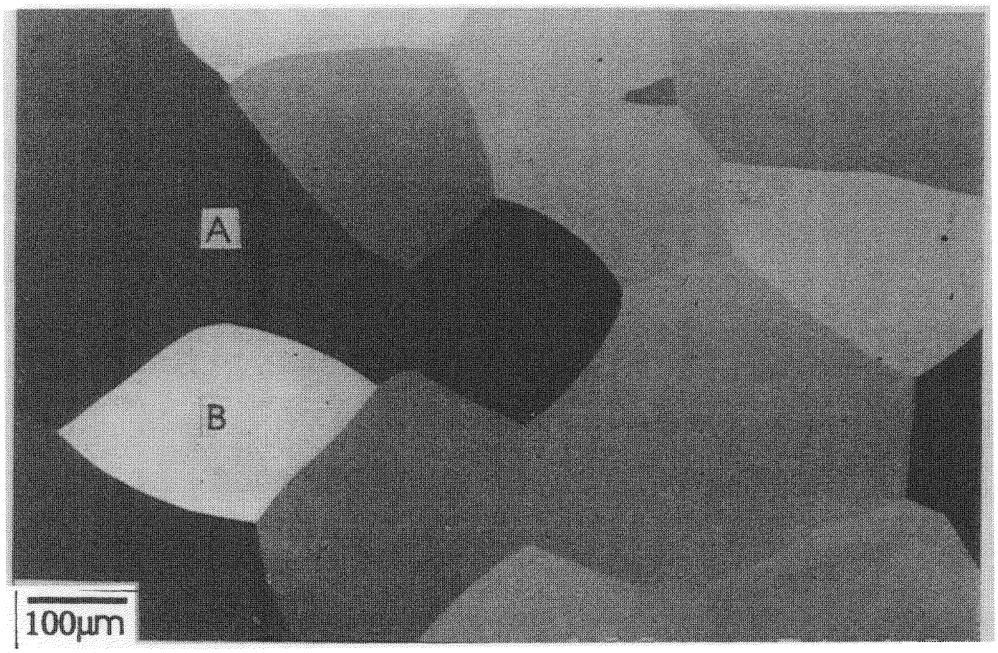


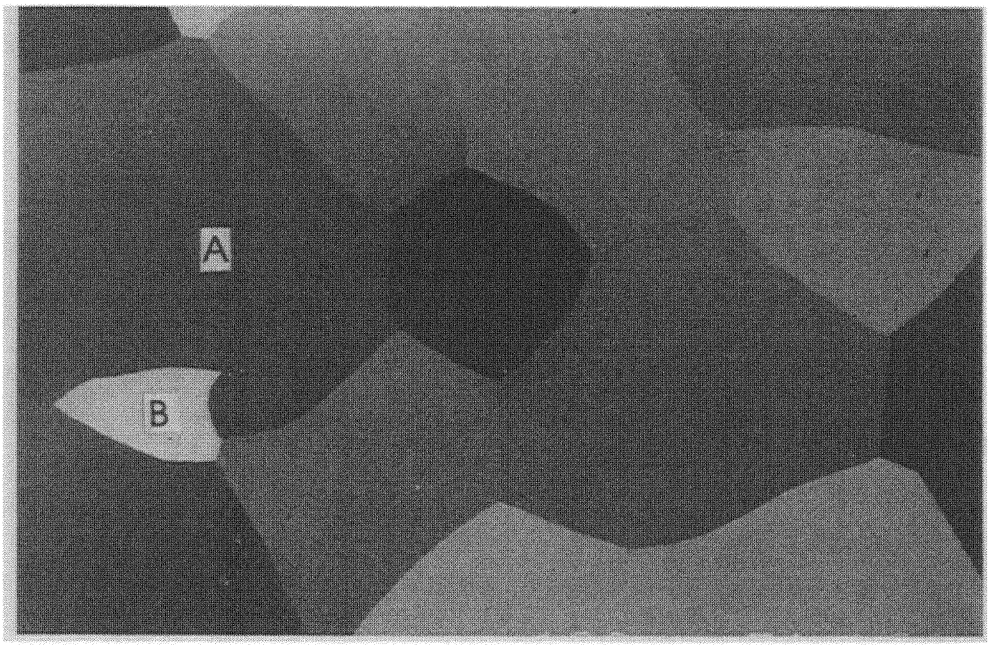
Figure 7 Recrystallization of an Al-1.6% Si alloy containing both large and small particles. a) 5 min at 375°C, b) 5 min at 490°C.

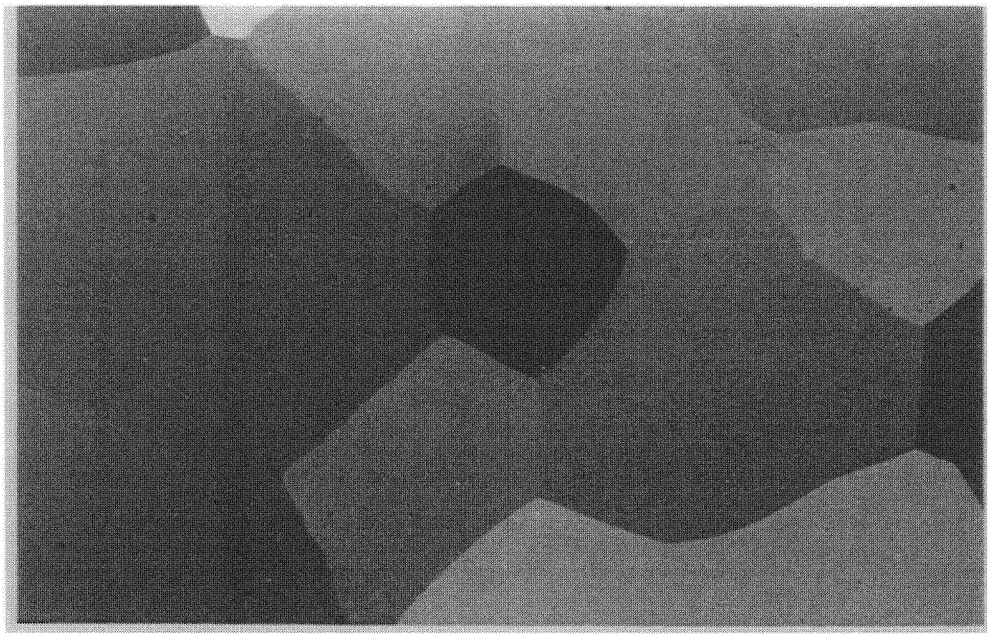
subsequently annealed for 1h at 400°C, producing a fully recrystallized microstructure with a grain size of ~200 μm as shown in Figure 8a. On annealing this specimen in the SEM it was found that rapid boundary migration occurred at 300°C as shown in Figures 8b and c. Grains were found to either grow (A) or shrink (B) and small grains on grain edges always shrank. This process, which occurs at temperatures well below that at which the microstructure was stabilised and whose rate decreases with time is essentially one of **boundary relaxation at a free surface.** A grain structure which is stable within a bulk specimen may not be stable when a free surface is subsequently introduced. This is easily demonstrated for the two-dimensional microstructure in Figure 9. The grain structure in Figure 9a is close to equilibrium. However, if the specimen is section and polished such that the plane AB is now a free surface (which has an energy of around three times that of a high angle boundary (Murr 1975), then boundaries close to AB will tend to migrate under the influence of the changed boundary tensions as shown in Figures 9b and c. For example, grains X and Z will tend to grow, grains Y and W will shrink and grain Y will ultimately disappear. Such boundary rearrangements are thus seen to be an inevitable consequences of the experiment and although they are not representative of grain growth in the specimen interior, they may be relevant to grain growth in thin films or surface-induced grain growth phenomena.

Grain growth at higher temperatures

The low temperature boundary rearrangements discussed above were complete within a few minutes at 300°C and a stable microstructure results (Figure 8c). On further annealing at 400°C and above, further grain growth, which is related to grain growth in the bulk sample occurred as seen in Figures 8d and 8e. The formation at C of an annealing twin (white contrast) of grain D is seen in Figure 8d. This twin then disappears during further grain growth (Figure 8e).




A
B



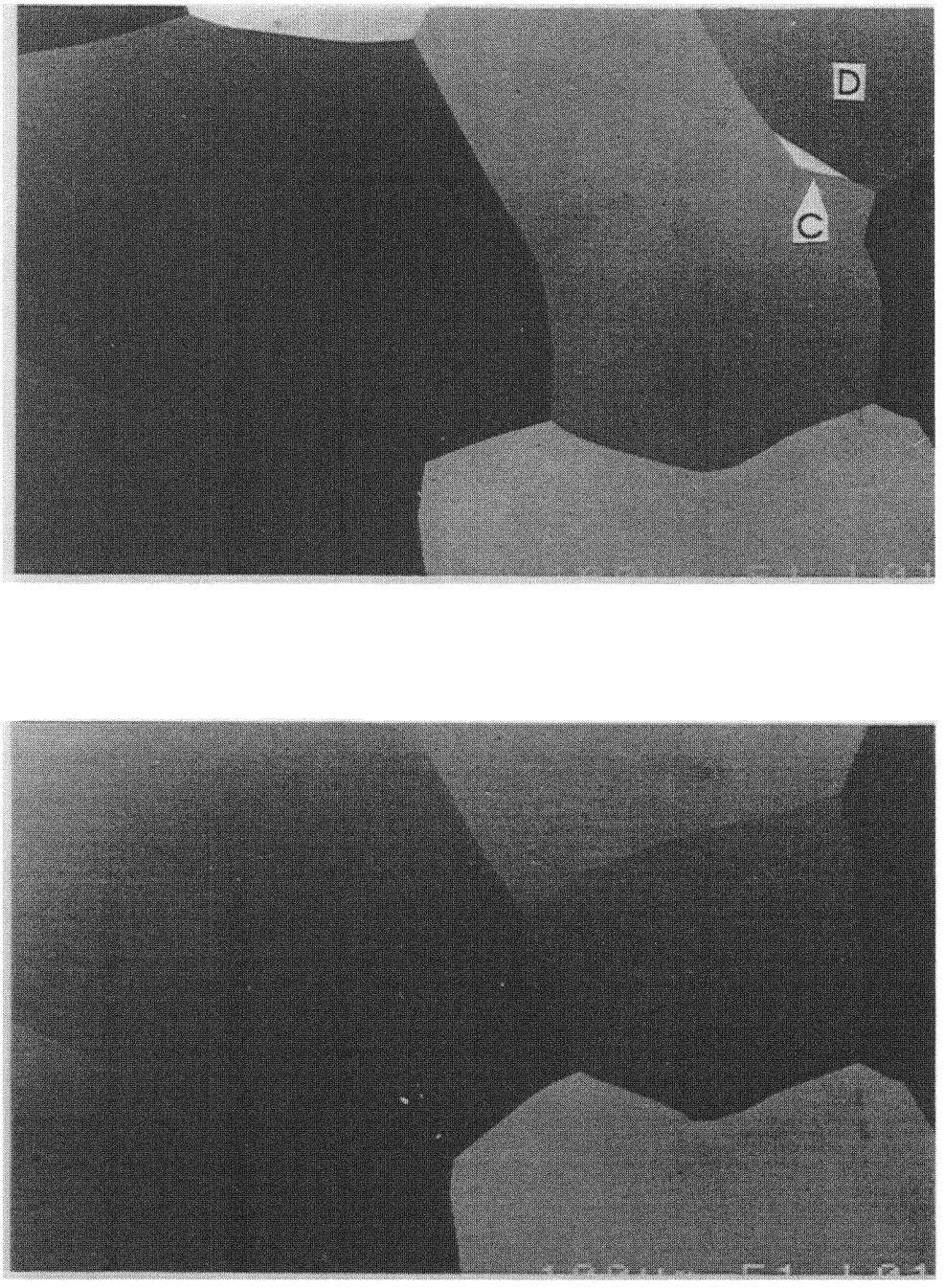


Figure 8 Annealing of a single-phase A1-0.05% Si specimen previously recrystallized at 400°C. a) Initial microstructure, b) 3 m at 300°C, c) 20 m at 300°C, d) 1 m at 400°C, e) 20 m at 400°C.

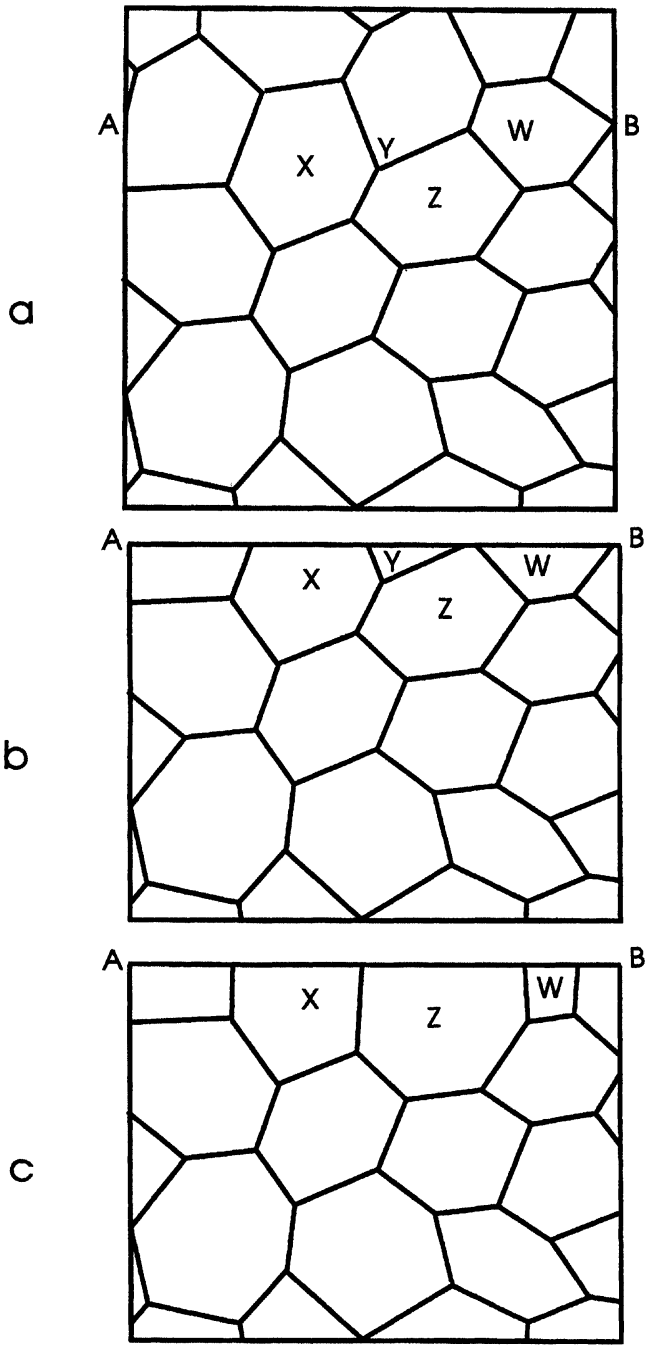


Figure 9 Schematic diagram showing the effect of introducing a free surface. a) Initial stable microstructure, b) Specimen sectioned at plane AB, c) Boundary rearrangements caused by the free surface.

A QUANTITATIVE COMPARISON OF BULK AND *IN SITU* ANNEALING

Recrystallization texture and grain size of AA1050

As part of an investigation of recrystallization textures in particle-containing aluminium alloys (Johnson and Humphreys), a quantitative comparison has been made between the textures and grain size distributions developed in the interior of a sample and those developed at a free surface, such as is found during *in situ* annealing experiments.

An AA1050 alloy with a weak starting texture, was furnace cooled from the homogenisation temperature of 600°C and reduced 50% or 85% by cold rolling. An electropolished section from the specimen mid-plane was recrystallized at 350°C. Without further polishing, the texture of the specimen was measured by x-ray diffraction and the grain size distribution on the free surface was also measured. Approximately 200 μm of material was then removed from the surface and the texture and grain size were then re-measured to determine these parameters for the bulk material.

Textures

The textures are shown in Table 1 as the percentages within 15° of **cube, rolling** components (brass (B_s), S, Cu) and **other** (or random) components.

The results show that the volume fractions of recrystallization texture components at the free surface and within the specimen interior for both the 50% and 85% deformed material are similar, implying that the presence of a free surface has a negligible effect on the recrystallization texture.

Grain size

The experimentally measured grain size distributions for the bulk and surface cases are shown in Figure 10. It is seen that the grain size distribution for the free surface case is somewhat broader than for the bulk case. The difference in the mean grain size was found to be small, the surface recrystallized grains being some 5–10% larger than those in the specimen interior.

It is to be expected that the grain size distribution will be affected by the presence of a free surface. If we consider a plane within a recrystallizing sample, then new grains will intersect this plane from both sides in the bulk material, but from only one side

Table 1 Comparison of the main texture components in recrystallized AA1050 at a free surface and within the bulk.

Material/thermomechanical processing route	*Bulk material vol% and (x random)*			*Free surface vol% and (x random)*		
	Cube	*Rolling (S + Cu + Brass)*	*Random /minor/ other*	*Cube*	*Rolling (S + Cu + (Brass)*	*Random / minor / other*
AA1050 – 50% cold rolled and annealed 30 min at 350°C	10.8 (2.4)	11.4 (0.7)	77.8 (1.0)	12.3 (2.7)	11.2 (0.6)	76.5 (1.0)
AA1050 – 85% cold rolled and annealed 30 min at 350°C	10.0 (2.2)	17.8 (1.0)	72.2 (0.9)	9.9 (2.2)	17.7 (1.0)	72.4 (0.9)

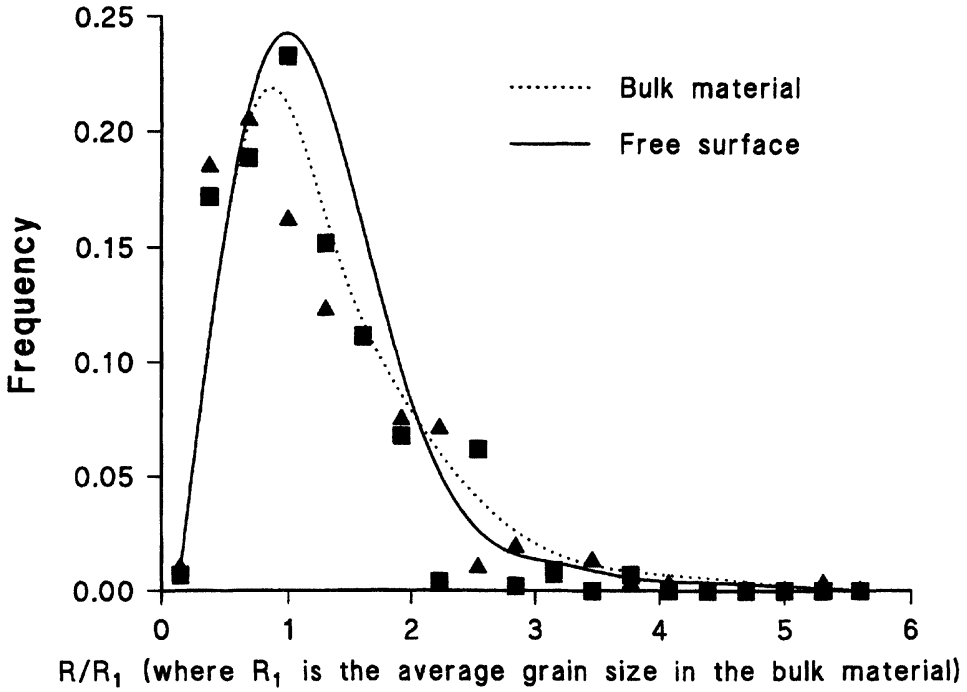


Figure 10 Experimental grain size distributions at a free surface (■) and the specimen interior (▲) in AA1050 recrystallized after 85% cold rolling.

if the plane coincides with a free surface. Therefore we expect a larger grain size at the free surface. We have carried out an Avrami computer simulation of recrystallization (Mahin *et al.*, 1980, Saetre *et al.*, 1986, Furu *et al.*, 1990), and compared the predicted grain size distributions within the material and at the surface as shown in Figure 11.

It may be seen that the computer simulation predicts both a broader spread of grain sizes and a mean grain size which is larger (by ~30%) at the free surface. The experimentally measured grain sizes do not show the predicted large increase in size and this may be due either to the accuracy of the experimental measurements or to the fact that the Avrami simulation does not take into account, either for the bulk or surface case, boundary relaxations which may occur after recrystallization is complete.

Annealing twins

During the *in situ* annealing experiments on aluminium alloys, it was noted that significant numbers of annealing twins were formed both during recrystallization and grain growth. An example of the formation of a small twin at a triple point, and its subsequent annihilation, is seen in Figures 8d and 8e. Further examples of annealing twins formed during *in situ* recrystallization of AA1050 are shown in Figure 12. The twin in Figure 12a is associated with a triple point and that in Figure 12b is associated with a large second-phase particle.

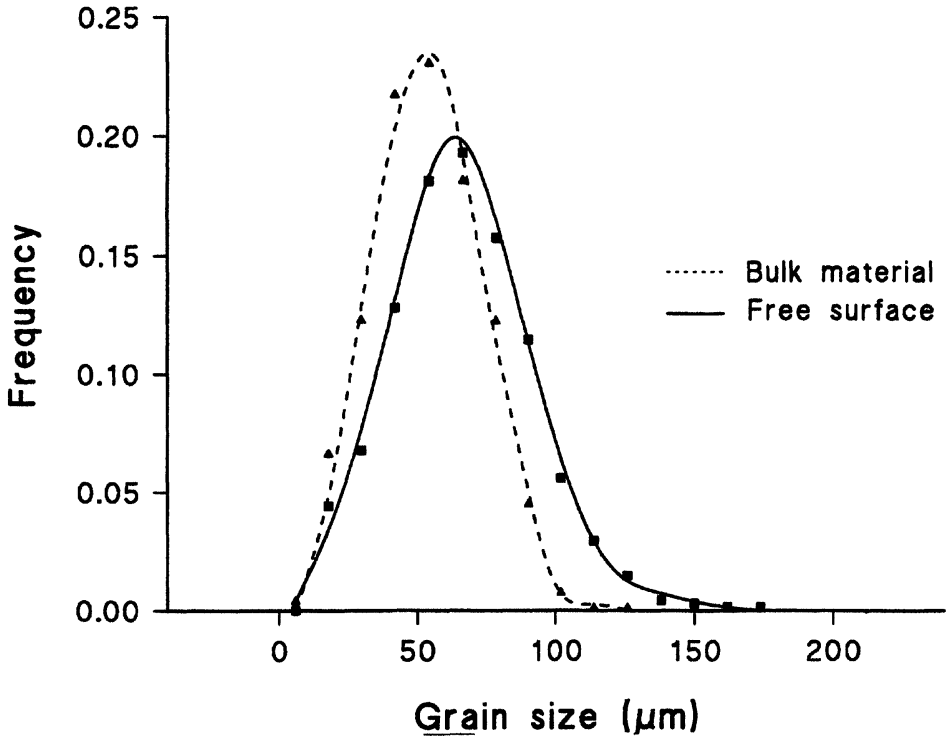


Figure 11 A comparison of the grain size distributions obtained using a 3-d computer Avrami recrystallization simulation, for the free surface (■) and grain interior (▲).

In order to determine whether the frequency of twinning during *in situ* annealing of aluminium differs from that occurring during annealing in the bulk, the following experiment was carried out. Specimens of AA1050 cold rolled 50% were annealed in the SEM as described earlier until fully recrystallized to a grain size of ~50 μm. Areas ~200 × 150 μm, each of which contained approximately 10 grains, were examined in the microscope. The number of grains and grain boundaries and the number of twin boundaries in the selected area were determined. Twins were tentatively identified by their straight coherent twin boundary, and the twin orientation was confirmed by EBSD. Some 25 such areas were measured in each of three *in situ* runs. The number of twin boundaries per grain boundary was found to be 1.3%. The specimens were then ground to a depth of ~400 μm and repolished. As several grain diameters had been removed from the specimen surfaces, the microstructures so revealed can be considered to be representative of the bulk annealed material. These specimens were then examined in the SEM and measurements similar to those described above were made. It was found that the number of twin boundaries per grain boundary in these samples was 0.28%. These experiments convincingly show that annealing twins are five times more likely to be formed at the **free surface** during *in situ* annealing than to be formed in the **interior** of the specimen.

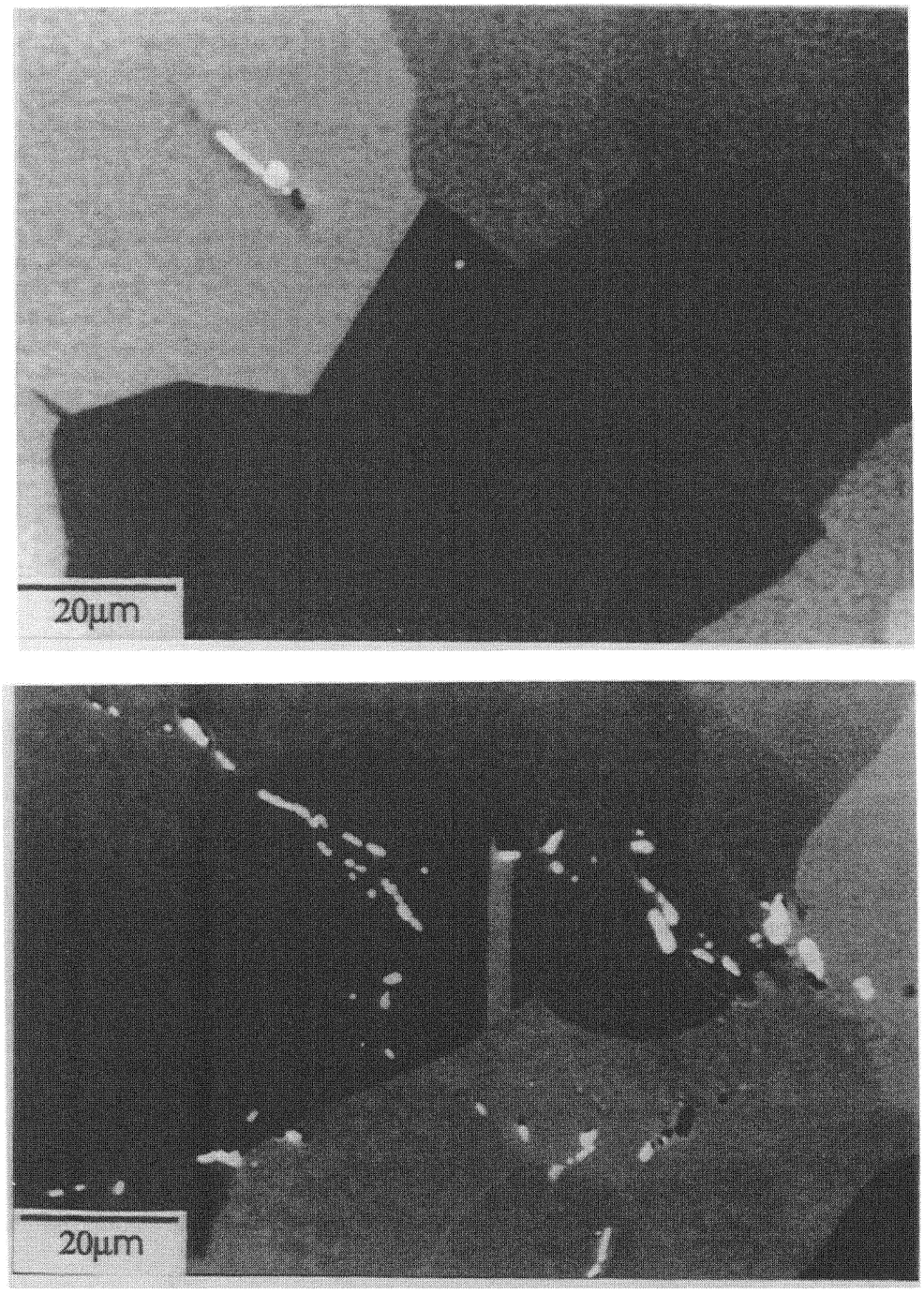


Figure 12 Annealing twins formed in AA1050 during *in situ* annealing. a) Twin at triple point, and b) twin associated with a large second-phase particle.

Aluminium has a high stacking fault energy and the formation of annealing twins is generally thought to be rare. Although it has long been recognised that such twins do form in aluminium (e.g. Aust 1961), little significance was attached to these observations until the recent work of Haasen, Wilbrandt, Berger and colleagues (Berger *et al.*, 1988, Wilbrandt 1992, Haasen 1993). These authors carried out extensive *in situ* annealing of deformed thin foils of various metals in the high voltage transmission electron microscope (HVEM). They found that significant numbers of annealing twins were formed in fcc metals, including aluminium, and suggested that such processes were important in the early stages of recrystallization and were an influential factor in the development of recrystallization textures. Whilst the importance if twinning in the recrystallization of metals of low stacking fault energy such as copper is not unexpected, the proposed role of such twinning in aluminium has been more controversial. The present results show the presence of a free surface promotes twin formation in aluminium. On this basis, the presence of two free surfaces, such as is found in a TEM specimen may be expected to promote twinning to an even greater extent. The occurrence of twinning during *in situ* annealing in the SEM, and presumably also the HVEM, is thus seen to be largely an artefact induced by the technique itself, suggesting that twinning may not play a significant role in the bulk annealing of aluminium. The role of high energy interfaces in the formation of annealing twins is discussed in more detail elsewhere (Humphreys and Ferry).

CONCLUSIONS

1. *In situ* recovery experiments on deformed aluminium have proved inconclusive, probably due to the large effect of the energy of the free surface.
2. The development of microstructures during recrystallization shows great similarity to the bulk, and similar textures and grain sizes are found in AA1050. The *in situ* experiments have confirmed the grain-scale inhomogeneity of recrystallization and have shown that there are significant variations in grain growth rate within a deformed grain.
3. Growth rate experiments on artificially nucleated grains are promising and suggest that the technique may be valuable in measuring boundary mobilities.
4. Grain structures formed during recrystallization of a bulk specimen, and which are then annealed *in situ*, exhibit substantial boundary migration at low temperatures due to the destabilising effect of the free surface on the microstructure.
5. Annealing twins are formed more frequently during *in situ* annealing than in the interior of aluminium, suggesting that the process is promoted by the presence of free surfaces.

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