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Direct Observations on the Recrystallisation Process of Lead

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The processes of nucleation and boundary migration of cold-worked lead held at room temperature were studied by a new process which permits continuous observation, by optical micrography, of the instantaneous positions of grain-boundaries. The results permit some conclusions as to the relative importance of the different nucleation mechanisms.

1. Introduction

The two basic processes of recrystallisation are the nucleation and the growth of new grains. The latter is a result of migration of the grain-boundaries separating the recrystallised and unrecrystallised material. The rate of this migration has a decisive effect upon the whole course of recrystallisation and so the study of grain-boundary mobility is one of the most important fields of recrystallisation researches.

The study of recrystallisation is usually made by deforming a number of specimens to various degrees, annealing for different lengths of time at different temperatures and then quenching. The extent of recrystallisation is then determined by a statistical metallographic method. This approach is suitable for getting results from which one can determine the time-dependence of the recrystallisation process with an accuracy which is normally sufficient in practice. However, such investigations cannot give much information about the *microkinetics* of recrystallisation. The grain-boundary mobility, the actual time-dependence of the speed of moving grain-boundaries, the effect of different obstacles (inclusions, segregations, grain- and twin-boundaries) can be exactly determined only by the direct, continuous observation of the recrystallisation process.

The direct observation of recrystallisation can in principle be achieved by high temperature vacuum microscopy, by emission electron microscopy and, in the case of optically anisotropic metals, by polarised light microscopy. A new method has recently been worked out suitable for the direct, continuous observation of low melting point metals at and near room temperature. We have investigated the recrystallisation of lead with this new method. The main results of these investigations are described in this article.

2. Experimental Method

The detailed description of the experimental method has been given elsewhere [1]. It is essentially as follows: a Zeiss-ELYPOVIST electrolytic polishing apparatus equipped with a microscope was supplied with a simple tensile apparatus (fig. 1). With the help of the drive

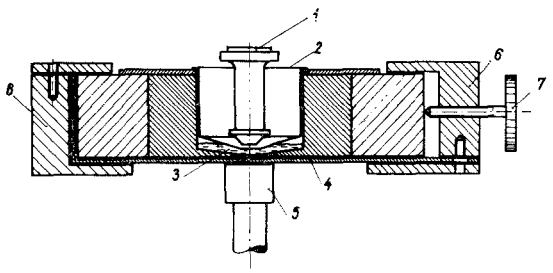
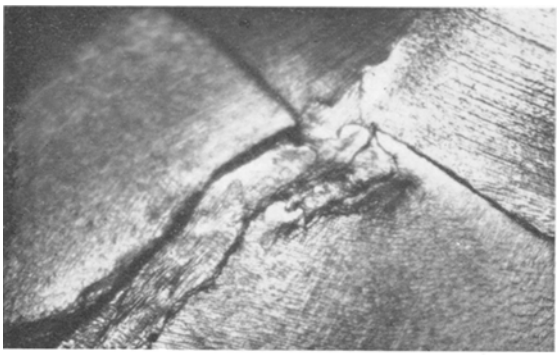


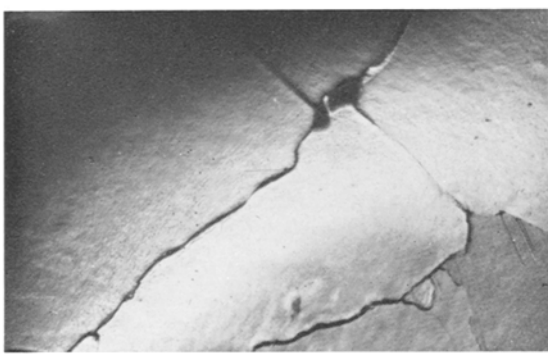
Figure 1 Electrolytic polisher ELYPOVIST with tensile apparatus (schematic): 1 objective; 2 shield (cathode); 3 electrolyte; 4 specimen (anode); 5 specimen holder; 6 tensile apparatus; 7 drive screw.

screw the sheet specimen was deformed to the desired degree. The fixed specimen is first polished, slightly etched, a chosen field photographed and the specimen then deformed. As a

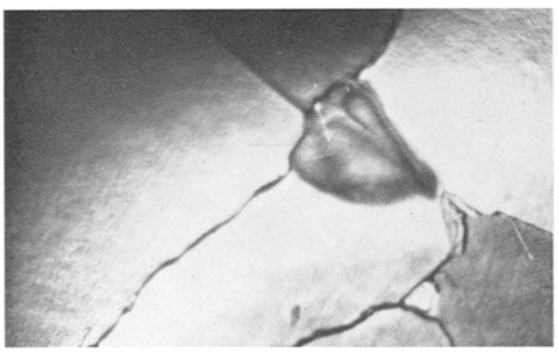
result of the deformation, slip lines appear which can be photographed (fig. 2a). The specimen is then repolished and continuously observed through the microscope while the electrolytic polisher remains in action. The position of the moving grain-boundaries becomes visible by the slight etching effect of the continuously circulating electrolyte. At expedient periods, photographs can be made; with the necessary accessories, the whole process can be filmed.



(a)



(b)



(c)

Figure 2 Nucleation in a specimen deformed by 60% (× 83): a directly after deformation; b 3 min after deformation; c 5 min after deformation.

The photographs obtained in the way described above are suitable for the more exact determination of the time-dependence of recrystallisation processes. The linear growth rate of the crystals can be directly measured from the photos; with the help of a planimeter the extent of recrystallisation can be measured from picture to picture and thus one can determine the function $X = f(t)$, where X = fraction of specimen recrystallised in the time t, for the field investigated.

3. Experimental Material

The material used in our investigations was lead of 99.9% purity in the form of sheet 1.5 mm thick. The specimens cut from this sheet were annealed for 10 min at 80° C before fixing on the tensile apparatus. The composition of the electrolyte was as follows: 200 ml perchloric acid, 700 ml alcohol, 100 ml glycerine.

4. Results of the Investigations

Most of the specimens were deformed 30 to 60%. We made observations and measurements of the fundamental processes of recrystallisation, i.e. nucleation and grain-boundary migration. Some of the more interesting results are summarised as follows.

4.1. Observations on Nucleation

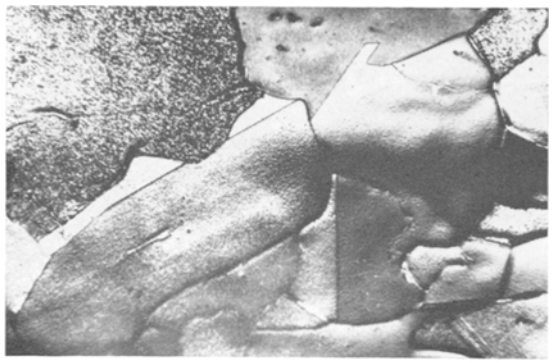
Nucleation occurred in most cases at the boundary of two or more crystals. For instance in fig. 2 nucleation in a specimen deformed by 30% is shown. In this case nucleation has taken place at the junction of four crystals.

During our observation of more than forty specimens, we only once observed the appearance of a new crystal in the interior of a grain. In that case nucleation probably took place inside the specimen under the surface at a grain-boundary and the new grain grew out to the surface.

A further possible mode of nucleation is Bailey's "microbulge" mechanism [2] which can be considered to begin as a form of stress-induced grain-boundary migration. This mode of nucleation is exemplified in fig. 3. The new grain, as one can see, developed by curving out of the old grain-boundary.

4.2. Observations on Grain-boundary Migration

The new crystals grow by the migration of their boundaries. The rate of this migration is affected



(a)

(b)

(c)

Figure 3 Bailey's nucleation mechanism in a specimen deformed by 30% (× 66): a 143 min after deformation; b 144 min after deformation; c 148 min after deformation.

by several factors (e.g. the driving force, the orientation, soluble and insoluble impurities intercepting the moving boundary, etc). The rate of grain-boundary migration affects the whole recrystallisation process decisively and the rate differences in different directions determine the shape of the growing crystals.

To illustrate our observations in fig. 4, the different phases of recrystallisation in a specimen deformed by 60% are shown. This whole process is represented schematically in fig. 5. The thick lines show the grain-boundaries before recrystallisation: the medium-sized broken lines are the result of stress-induced grain-boundary migration, and the thin lines indicate intermediate positions of the grain-boundaries. The arrows show the main directions of growth which altered several times in the course of the recrystallisation. The numbers indicate the chronological order.

The main processes of the recrystallisation illustrated in figs. 4 and 5 can be described as follows:

(*a*) Some movement of grain-boundaries can be observed at certain places after deformation and before nucleation; this can be considered as stress-induced grain-boundary migration.

(*b*) Nucleation takes place at both sides of a grain-boundary. The nucleation on the left side of the boundary is of an interesting type (see fig. 4): at first some needle-like nculei appear; their development is very rapid, then they begin to grow *and merge into one grain.*

(*c*) The growth of this grain is also interesting: in the early stages the shape of the grains is "crystal-like", i.e. bordered by straight lines; later their lines become curved. As the direction of the main growth changes, the shape of the grain alters.

In some directions in fig. 5 we measured the linear sizes of the crystal and represented them as functions of the time in fig. 6. One can see in the diagram that the velocity of growth sometimes changed jumpwise. In some cases these changes were caused by obstacles (e.g. grain- or twin-boundaries); in most cases, however, we did not observe any visible cause. As a result of these sudden changes, steps appeared on the $X = f(t)$ curve determined with the help of a planimeter. The cause of these sudden changes – according to Cahn's theory [3] and Gordon and Vandermeer's article [4] – can be dissolved impurity atoms accumulating at the grain-boundaries. Further investigations of this phenomenon are still in progress.

We also made several observations on the development of twins. Twins appeared partly in already recrystallised grains. When this happened their formation was a very rapid process. In

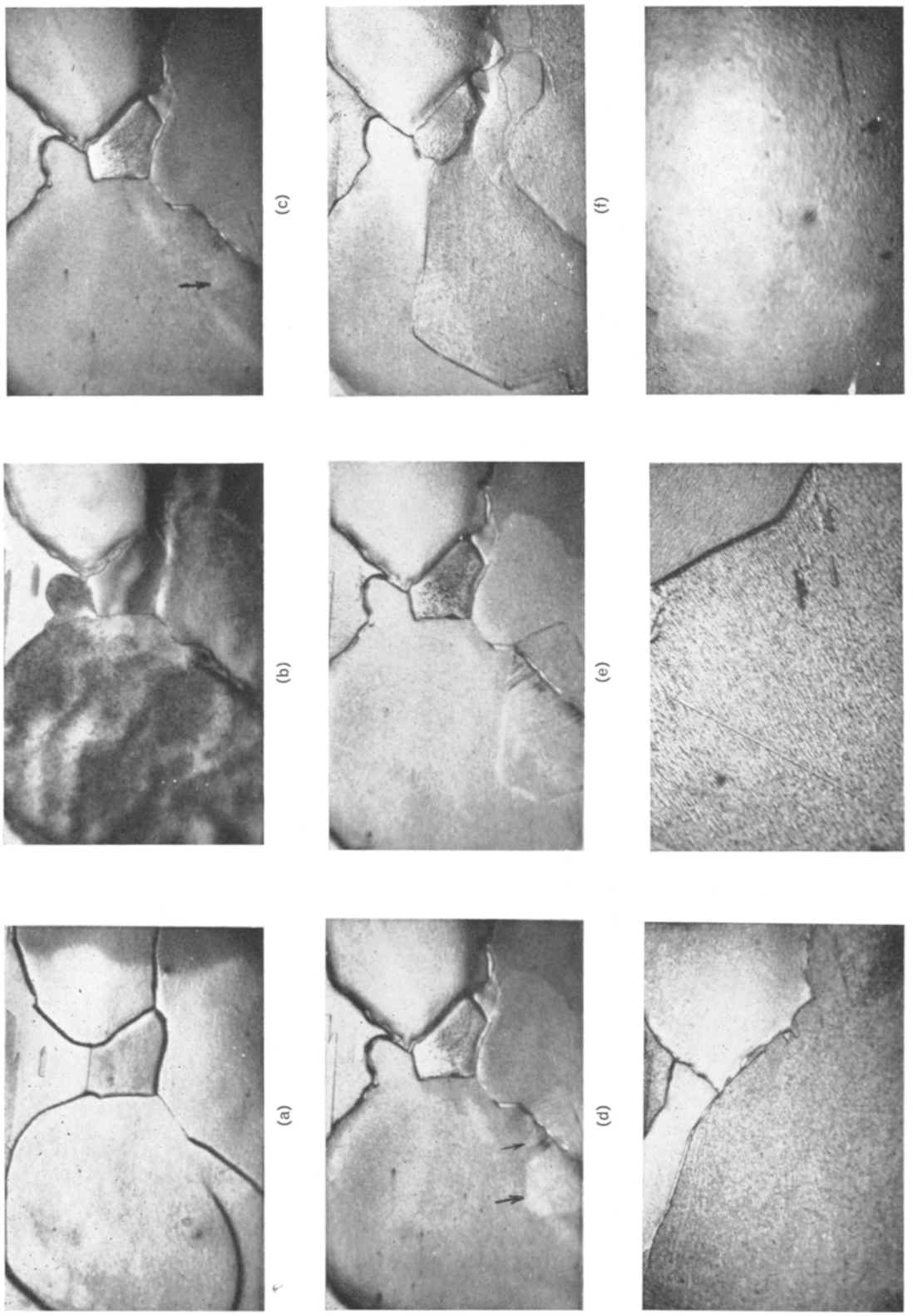


Figure 4 Different phases of the recrystallisation in a specimen deformed by 30% (× 26): a directly before deformation; b 2 min after deformation; c 7 min after deformation; d 8 min after deformation; e 10 min after deformation; f 17 min after deformation; g 20 min after deformation; h 77 min after deformation; i 152 min after deformation.

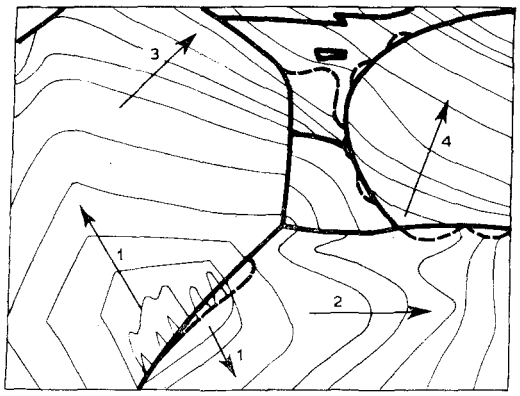


Figure 5 Course of recrystallisation of the specimen of fig. 4 (schematic).

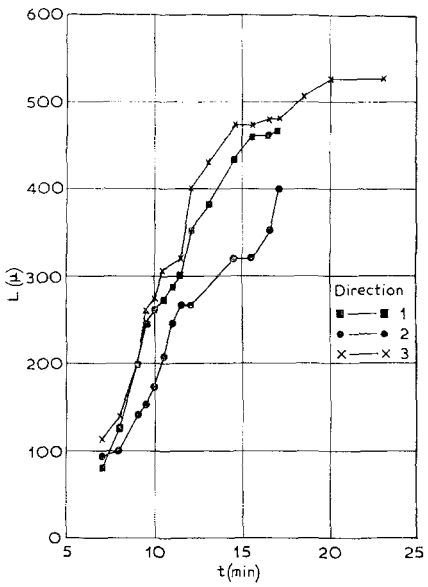


Figure 6 The change of the linear dimensions of the growing crystal in the specimen of fig. 4.

other cases the twin grew together with its "mother" crystal. The growth rate of the crystal was either higher or lower than that of the "mother" crystal. It can be seen in fig. 7 that the twin grows more rapidly than the "mother" crystal, but there is a smooth transition at their mutual boundary.

5. Conclusions

With the help of our new method for the direct, continuous observation of the recrystallisation of low melting point metals it has been confirmed that nucleation takes place first of all at grain-boundaries. Bailey's nucleation mechanism has also been observed, occurring, however, much less frequently and generally later than that at grain-boundaries.

It was observed that the grain-boundary rate sometimes changed suddenly without any visible cause. This is in agreement with Cahn's theory on the dragging effect of dissolved impurities. The direction of most rapid growth also changed. These phenomena caused steps on the $X = f(t)$ diagram taken for the field investigated.

Two modes of twin development were observed. The first is very rapid, when the twins develop in crystals already recrystallised; the second involves twins growing together with their "mother" crystals.

The results described in this article show that with the help of our method of investigation more precise description of the microkinetics of the recrystallisation of low melting point metals is possible. The phenomena observed are of basic importance and their character is the same in metals which recrystallise at higher temperatures. Thus lead can be used as a model metal in recrystallisation studies.

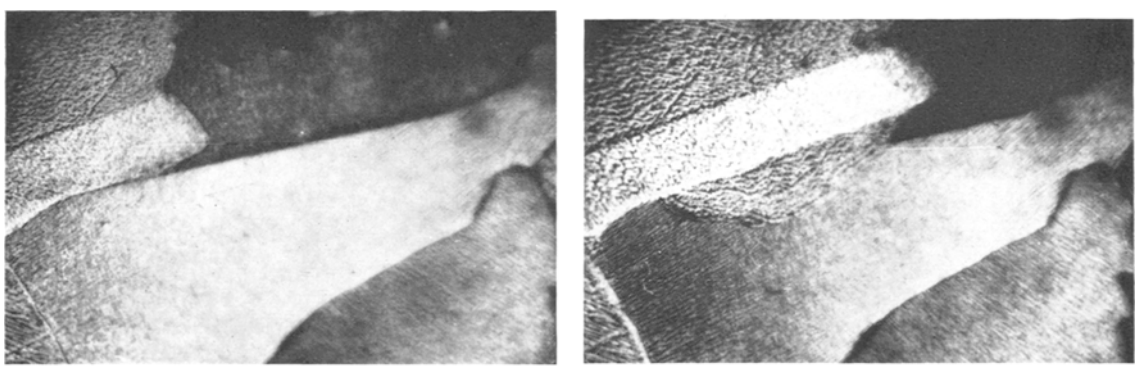


Figure 7 Growth of a twin during recrystallisation (× 227).

Acknowledgement

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