The effect of grain size on deformation twinning in a textured zinc alloy

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The incidence of deformation twinning in samples of a commercial rolled zinc–0.1% aluminium–0.05 wt % magnesium alloy has been measured at room temperature as a function of strain, grain size and direction of loading relative to the rolling direction. The volume fraction of twinned material has been measured at the surface and in the bulk. It is shown that this volume fraction is a linear function of both strain and grain size in most cases. Bands of heavily twinned grains are found to form inhomogeneously across the gauge length of tensile specimens, by an autocatalytic mechanism. Several examples are given of the interaction of twins and slip bands at grain boundaries which illustrate the formation and the accommodation of twins. The smaller volume fraction of twins found at the surface compared with the bulk reflects a relaxation of Von Mises criterion.

1. Introduction

A variety of deformation modes can operate in zinc at ambient temperatures, depending on the strain rate and grain size. At very small grain sizes ($< 10\,\mu$m) superplastic behaviour may be observed [1, 2] while at larger grain sizes, there are three possible modes of deformation, namely basal slip, $\{0\,0\,0\,1\}\langle 1\,1\,\bar{2}\,0\rangle$, pyramidal slip $\{2\,\bar{1}\,\bar{1}\,2\}\langle\bar{2}\,1\,1\,3\rangle$ and deformation twinning, $\{1\,0\,\bar{1}\,2\}\langle\bar{1}\,0\,1\,1\rangle$ [3]. Basal slip operates at the lowest stress, but contributes only two independent slip systems. Since five independent deformation modes are required for a general strain in an equiaxed polycrystal [4] either pyramidal slip or twinning must operate in addition to basal slip. Pyramidal slip is thermally activated, and is therefore favoured at high temperatures and low strain rates, while twinning is found to occur under the reverse conditions, i.e. in the absence of a favourable slip mode e.g. [5].

Grain size is another factor influencing the incidence of deformation twinning; although few quantitative studies have been made e.g. [6, 7] there is general agreement that a small grain size inhibits twinning [3]. There is also evidence from work on cadmium that pyramidal slip operates less readily at large grain sizes [8].

The influence of grain size on the choice of secondary deformation mode depends on the relative ease of initiation of pyramidal slip and twinning. Work on twinning in a variety of materials provides conflicting evidence for the mechanism of twin nucleation, but it is generally accepted that some form of stress concentration is required. It has been suggested that some slip is a necessary precursor to twinning e.g. [9] and in Fe–3%Si it has been shown that dislocation activity occurs even when no macroscopic strain is recorded prior to twinning [10]. However, in other cases, no dislocation movement was observed before twinning [11]. These observations can be rationalised if it is assumed that localised slip is only required prior to twinning if twin nuclei are not already available [12]. It has been proposed, for example, that such a nucleus could be associated with the strain field of a total slip dislocation [13]. However, any nucleation mechanism is difficult to prove, since twins propagate very rapidly once nucleated.

Macroscopically, in some materials twins are observed from the onset of measurable plastic deformation (e.g. Zr, [5]) while in others a minimum strain due to slip is required before twinning can occur (e.g. Cr–Re and Mo–Re, [14]). Twins are usually nucleated at grain boundaries in polycrystals, often where dislocations have piled up against the other side of the boundary [13, 15].

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The contribution of twinning to the overall strain depends linearly on the volume fraction of twinned material, and on the orientation of the twin with respect to the tensile axis [3]. Hence the fraction of the tensile strain, ϵ_t, due to a single twin is given by:

$$\epsilon_t = v\gamma m_t, \tag{1}$$

where γ is the twinning shear, v is the volume fraction twinned and m_t is the Schmid factor for that twin. This relationship can be extended to many twins in a polycrystal, providing m_t can be estimated. It has been argued [5] that a realistic estimate for m_t is 0.5, while in highly textured material, m_t can be directly related to the Schmid factor for twinning in a single grain of average orientation [15]. The volume fraction of twins has been found to be a linear function of strain in several materials [5, 14].

There were two aims in the present study of deformation twinning in zinc: firstly to quantify the effect of both strain and grain size on the incidence of twinning, and secondly to investigate the differences between deformation at the surface and in the bulk. Von Mises criterion (five independent deformation modes required for a general strain [4]) will be relaxed at the surface; if twinning acts only as a secondary deformation mode, fewer twins should be observed in the surface grains. A dilute zinc alloy in the form of rolled sheet was used for the investigation, hence texture was introduced as a further parameter.

2. Experimental procedure

2.1. Material

The material used in this investigation was a commercial rolled zinc alloy containing 0.1 wt% Al and 0.05 wt% Mg. The as-received microstructure consisted of equiaxed grains with a mean diameter of 4 μm. It has been established [1] that a range of grain sizes may be obtained in this alloy by means of grain growth, and that this treatment does not affect either the grain size distribution (in the bulk) or the strong texture of the rolled sheet.

In the present work, specimens of five different mean grain sizes were used (20, 30, 40, 100 and 160 μm). Grain size measurements were made as described elsewhere [1]. Since twinning measurements were to be made both at the surface and in the bulk, the distribution of grain sizes in the bulk and surface grains were investigated separately. The average grain diameter at the surface was found to be slightly smaller than that in the bulk, but the shape of grain size distribution was unchanged. The grain sizes (mean linear intercepts) are listed in Table I.

TABLE I Average grain sizes of Zn–0.1 wt% Al–0.05 wt% Mg obtained after 10 minutes heat treatment

Temperature of anneal (° C)	Surface/Bulk	Mean linear intercept (μm)
290	Surface	23.2
	Bulk	22.9
315	Surface	30.2
	Bulk	31.6
330	Surface	42.0
	Bulk	43.0
350	Surface	92.6
	Bulk	99.4
370	Surface	159.8

2.2. Assessment of twin volume fraction

Both longitudinal and transverse specimens* were extended in an Instron screw-driven tensometer to known levels of plastic strain up to 5%, at a strain rate of $2.8 \times 10^{-4}\,\text{sec}^{-1}$. The specimens were prepared for twin counting and optical microscopy by chemical polishing in a freshly mixed aqueous chromic acid solution of the following composition [16]: 30 g chromic acid, 4 g anhydrous sodium sulphate, 150 ml water and 8 ml nitric acid. After immersion in the chemical polish, specimens were washed in water, and any remaining chromate deposit removed by dipping the specimens into a dilute aqueous solution of potassium hydroxide and washing again with water. The rate of dissolution of zinc in the chemical polish is such that the amount of material removed can be readily controlled, providing the solution is renewed frequently. Only sufficient material was removed prior to mechanical testing to ensure a surface suitable for optical microscopy.

After mechanical testing, the volume fraction of twins at the surface of each specimen was estimated using a Swift automatic point counter. Providing the twin distribution is random, the volume fraction of twins is equal to the fraction of points falling on twins [14]. The surface layers were then removed by chemical polishing to a

*Longitudinal specimens were punched so that the gauge length was parallel to the rolling direction, while transverse specimens have gauge length and rolling direction perpendicular.

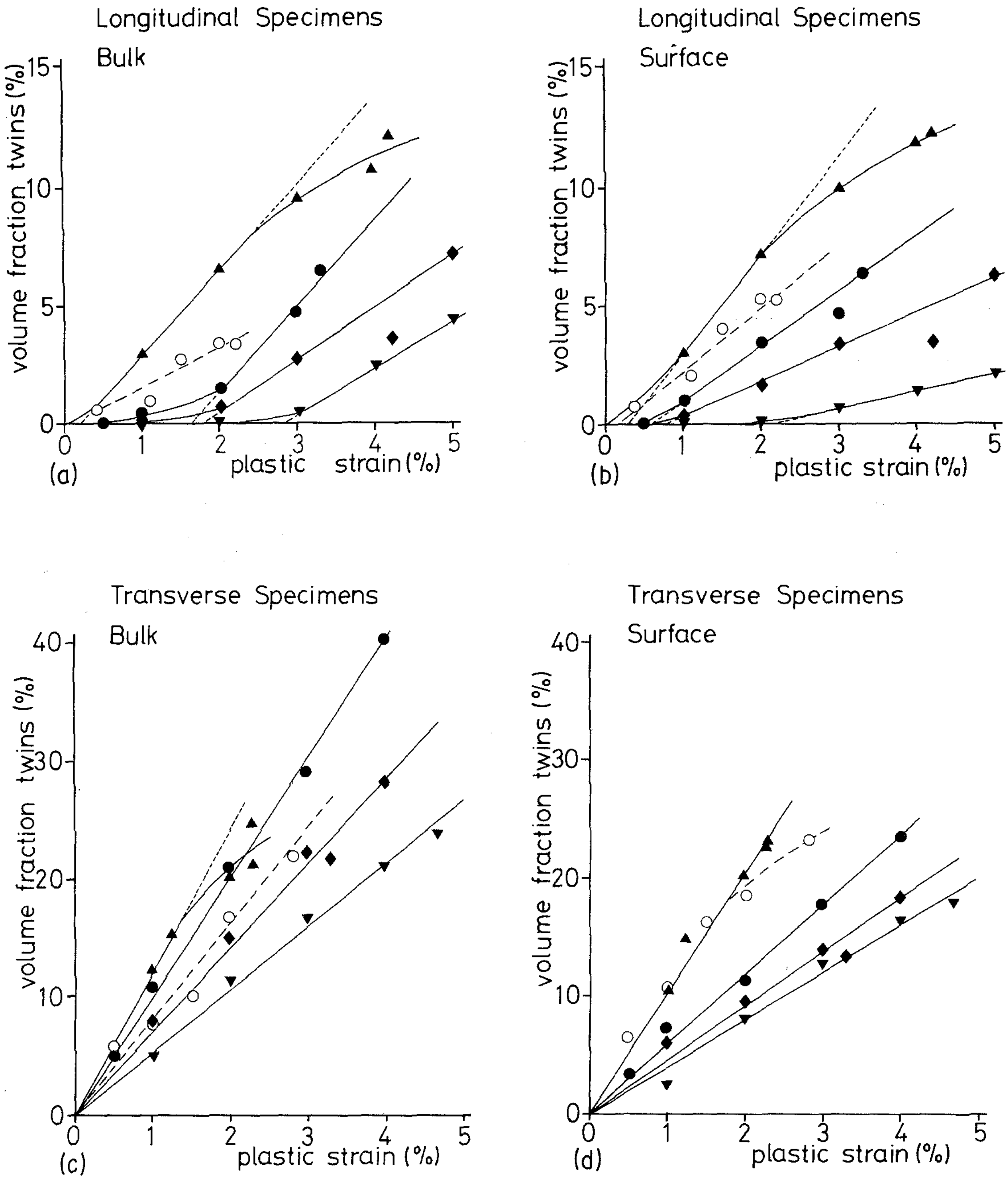

Figure 1 Twin volume fraction plotted as a function of strain: ▼ grain size ~ 20 μm, ◆ grain size ~ 30 μm, ● grain size ~ 40 μm, ▲ grain size ~ 90 μm, ○ grain size ~ 160 μm.

depth of at least 150 μm (on each surface) and the volume fraction of twins in the bulk measured. For specimens of the smaller grain sizes, measurements at this distance from the surface can be considered representative of the bulk; at the larger grain sizes the surface effects probably extend throughout the specimen thickness.

3. Results

3.1. Variation of twin volume fraction with strain

Fig. 1 shows twin volume fraction plotted as a function of strain for all the specimens tested. Longitudinal and transverse specimens behave differently: for transverse specimens the twin

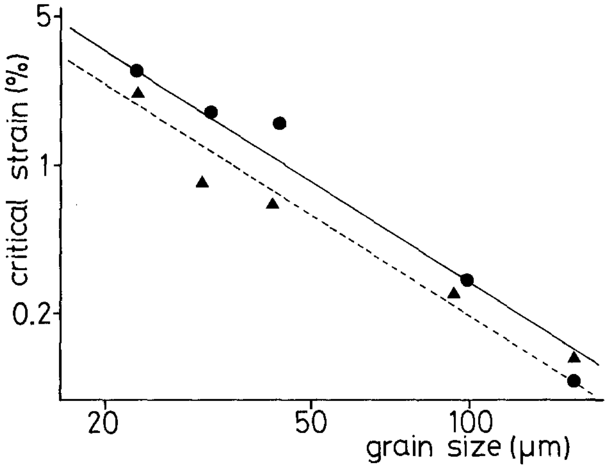

Figure 2 The critical strain for twinning in longitudinal specimens plotted as a function of grain size, ○ bulk and △ surface.

volume fraction is a linear function of strain, while in the longitudinal case this is only true beyond a given level of plastic strain.

With the exception of specimens of the largest grain size (open circles), the volume fraction of twins at a given strain increases with increasing grain size. The anomalous behaviour of the specimens of the largest grain size will be discussed further below. At the larger grain sizes, the twin volume fraction/strain relationship does not always remain linear to high strains.

For longitudinal specimens, the critical strain to initiate twinning is a function of grain size (further evidence for this is presented in [1]). This strain, defined from the linear relationship of twin volume fraction and strain extrapolated to zero twin volume fraction (as shown by the dotted lines in Fig. 1), is plotted against grain size in Fig. 2.

3.2. Variation of twinning contribution with grain size

The volume fraction of twins has been shown to be a linear function of the overall strain, and from the equation is linearly related to the strain produced by twinning. Hence the fraction of the overall strain contributed by twinning (beyond the critical strain) is a constant for each grain size, and is proportional to the gradient of the twin volume fraction/strain plot. The slopes of the linear regions of the plots of Fig. 1 are plotted as a function of grain size in Fig. 3. This figure shows firstly that the contribution to the overall strain from twinning is greater in transverse specimens than in longitudinal ones, as already discussed, and secondly that there is more twinning in the bulk than on the surface.

Transverse specimens and longitudinal specimens beyond the critical strain show exactly parallel behaviour in the bulk. At small grain sizes the relationship between the contribution of twinning to the overall strain and average grain diameter is linear (the log/log plot has a slope of 1.0). However, at large grain sizes the twinning contribution apparently becomes independent of grain size. Using Equation 1 with $m_t = 0.5$, twinning is calculated to contribute nearly 85% of the deformation for transverse specimens with a grain size of 100 μm. This is probably a maximum contribution, since some deformation by slip must be possible in both twinned and untwinned material. Longitudinal specimens behave similarly, with a maximum contribution from twinning beyond the critical strain of about 30%. For grain sizes greater than 100 μm, the contribution to the strain from twinning apparently decreases with grain size. However, specimens of 160 μm grain size probably do not exhibit behaviour truly representative of bulk materials, as discussed elsewhere [1]. Since at this grain size the number of grains contained within the thickness of the rolled sheet is so small (~ 5), the effect of the surfaces on twinning will extend through the whole specimen, and anomalous results will be recorded.

At the surface, the behaviour of longitudinal and transverse specimens is somewhat different. In the longitudinal case, the relationship between grain size and the contribution to the strain from twinning is linear as before, tending to a maximum value similar to that in the bulk. Again, specimens of the largest grain size show anomalous behaviour. Specimens of the smallest grain size have fewer twins than expected, which may indicate that there is a limiting grain size below which twinning

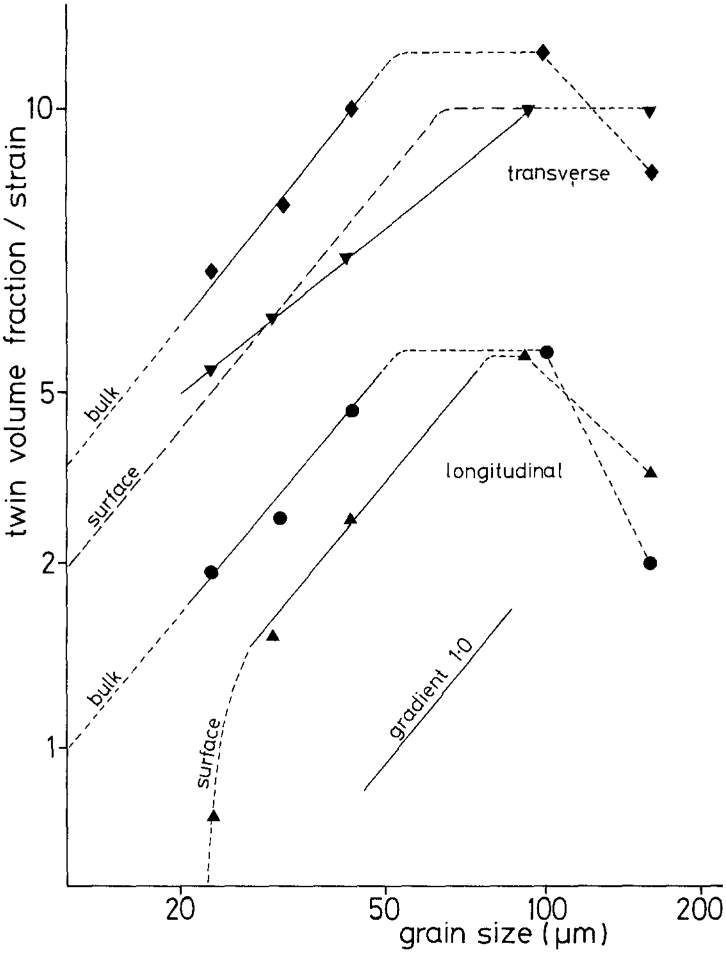

Figure 3 Twin volume fraction/strain (contribution of twinning to the overall strain) plotted as a function of grain size.

is never observed (at this strain rate and temperature). This is supported by the absence of twinning in the as-received material, grain size $\sim 4\,\mu m$, deformed under similar conditions.

The relationship between the contribution to the strain from twinning and grain size does not appear to be linear for transverse specimens at the surface. The best straight line through the experimental points in Fig. 3 (solid line) has a gradient less than unity. However, considering the errors in the experimental data, and the fact that in all other cases a linear relationship was found, it is suggested that the dashed line could be drawn to represent the transverse surface case; the deviations of the experimental points from this line are no greater than for other points in Fig. 3.

3.3. Twin formation and accommodation

It was observed during deformation that twins do not form homogeneously (particularly in specimens of the smaller grain sizes) but are produced in bands which form instantaneously and extend right across and through the specimen, approximately perpendicular to the tensile axis. One such band is shown in Fig. 4 and can be seen to extend across the whole width of the specimen (grain size $\sim 20\,\mu m$). All the twinning is contained within these twin bands; for a set of specimens in which the number of twin bands could be counted after deformation (they become more numerous and less distinct as the grain size increases) the number of twin bands shows excellent correlation with the twin volume fraction for the same specimens, Fig. 5.

The presence of twin bands suggests that twins form by an autocatalytic mechanism; once one twin has been nucleated and has propagated across a grain, the stress concentration at the end of the twin is accommodated by the formation of a twin in the next grain. To investigate this further, some more detailed observations were made of slip and twinning in adjacent grains after deformation.

Slip lines were always observed on the surface of deformed specimens, even in transverse speci-

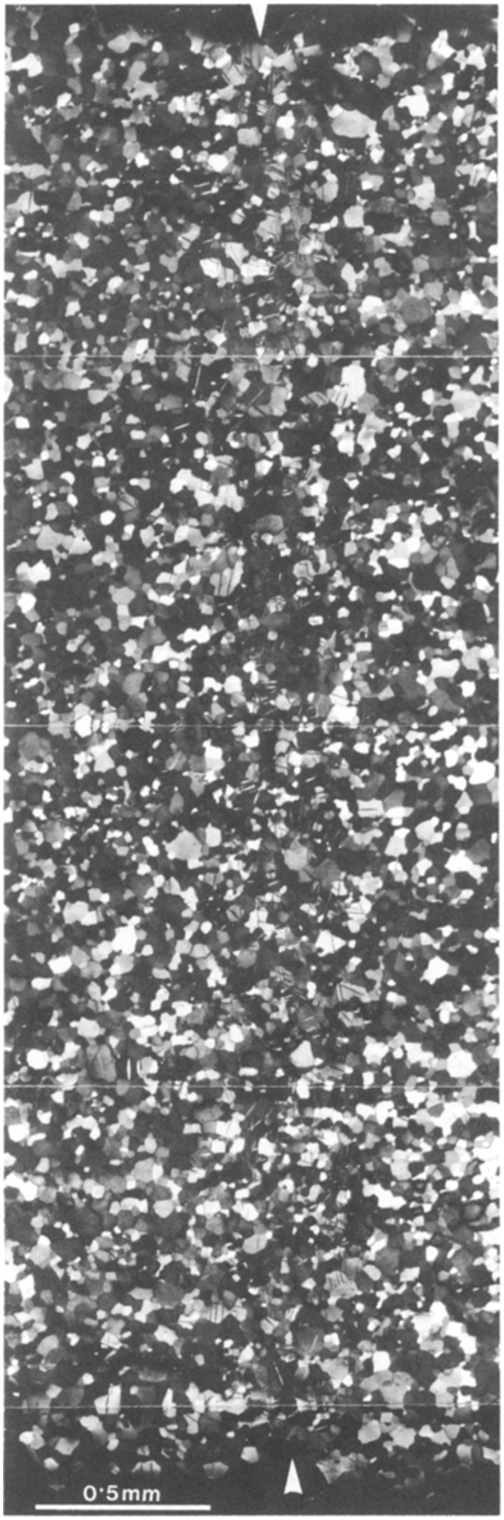

Figure 4 Polarised light micrograph of a twin band (arrowed). Longitudinal specimen, of grain size ~ 40 μm, strained to 3%.

mens of the largest grain size. This observation confirms that deformation cannot proceed entirely by means of twinning. Twins were always found to be in contact with at least one grain boundary, and usually to extend right across the grain. In three dimensions, a twin is shaped like a convex lens, and almost any two-dimensional section through a twin therefore appears lenticular. Since in two dimensions a twin was nearly always observed to be in contact with a grain boundary at both ends of its length it is probable that the edges of the three dimensional twin are defined by the boundaries of the grain in which it forms. The shape change produced by the twinning shear is partly accommodated in the matrix by the lenticular shape of the twin, but a stress concentration must occur at its edges, which can be accommodated either within the grain boundaries or in the surrounding grains. In the relatively rare cases where a twin does not extend completely across a grain, the stress concentration must be accommodated by dislocations with that grain.

The accommodation of twins at grain boundaries was observed to occur by two mechanisms, involving either slip or twinning in the neighbouring grains. Examples are shown in Fig. 6. In Fig. 6a the twins in grain 1 have been accommodated by slip in grain 2. The slip traces at the ends of the twins do not extend across the whole width of grain 2; unless nucleated in the middle of the grain (very unlikely) these slip bands must have been formed to accommodate the twin, and not vice versa. Another twin in grain 1 joins twins in grains 2 and 3, but it is not clear which formed first. Fig. 6b shows a more obvious one-to-one correspondence between twins in neighbouring grains. Twins in grain 1 have nucleated twins in grain 2, two of which are accommodated within grain 2 (although slip at the ends of these twins is not apparent). The other twins in grain 2 are accommodated in grain 3, either by twinning or by slip. In this case it is possible to say that the twins in grain 1 must have formed first, since the twins in grain 2 are most unlikely to have been nucleated in the middle of the grain.

It was observed that twins are not always nucleated by other twins, although this is very frequently the case, hence the formation of twin bands. Slip bands impinging on a grain boundary can provide a stress concentration in the next grain which can be relieved by the formation of a twin. An example of this is shown in Fig. 7;

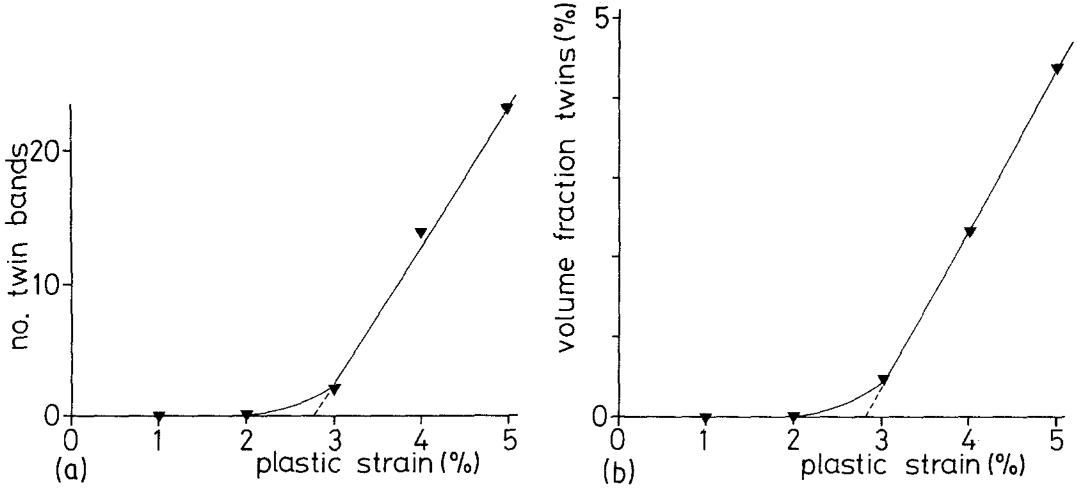

Figure 5 (a) Number of twin bands in longitudinal specimens (bulk) of grain size ~ 20 μm, plotted as a function of strain; (b) The number of twin bands correlates well with the total volume fraction of twins in the same specimens.

there is a clear correspondence between the most marked slip bands in grain 1, and the twins in grain 2. These twins do not extend all the way across the grain, unlike the slip bands, hence the twins are almost certainly nucleated by the slip bands and not vice versa.

Slip is not confined to a single slip system in each grain, although there is usually only one set of slip lines which is observed over the whole grain area. Additional slip systems are observed near the grain boundaries; these systems are required by the need to maintain material continuity. Fig. 8 shows an example of this: in addition to the slip lines which cover the whole

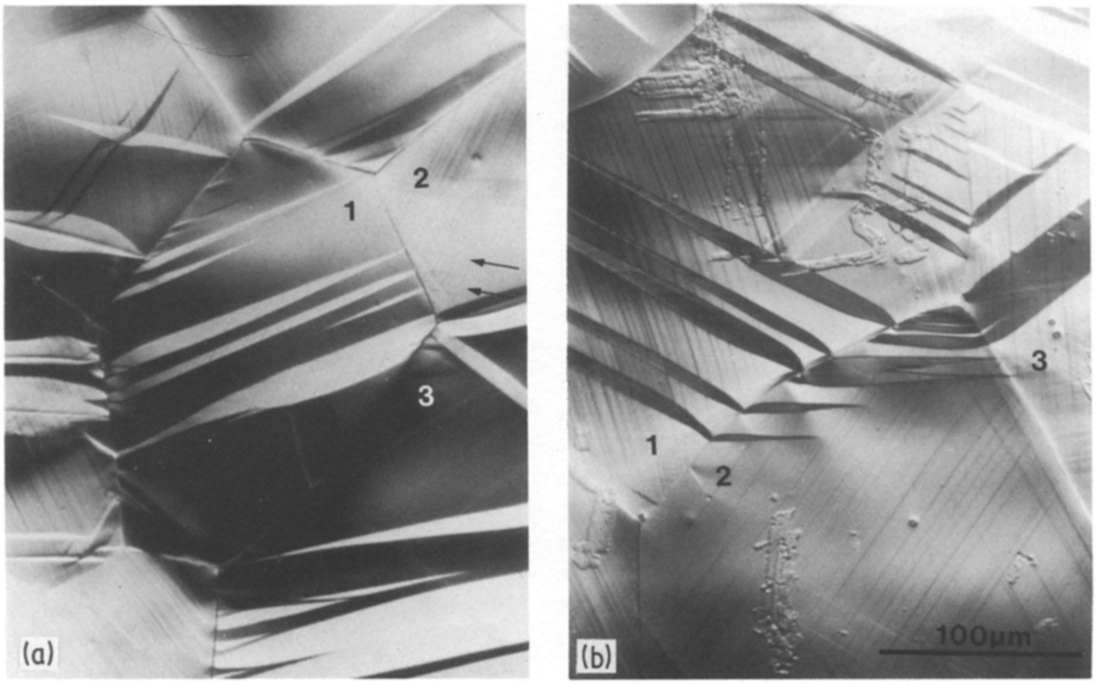

Figure 6 Nomarski interference micrographs of transverse specimens, strained to 2%. Grain size ~ 150 μm. (a) Twins in grain 1 accommodated by slip (arrowed) in grain 2. Four twins meet at the junction of grains 1, 2 and 3. (b) Twins in grain 1 have nucleated twinning in grain 2. Two of these twins are accommodated by slip or twinning in grain 3. Slip has occurred within the twins in grain 1 (see text).

Figure 7 Nomarski interference micrograph of a transverse specimen, grain size ~ 150 μm, strained to 1%. Slip bands in grain 1 have nucleated twins in grain 2.

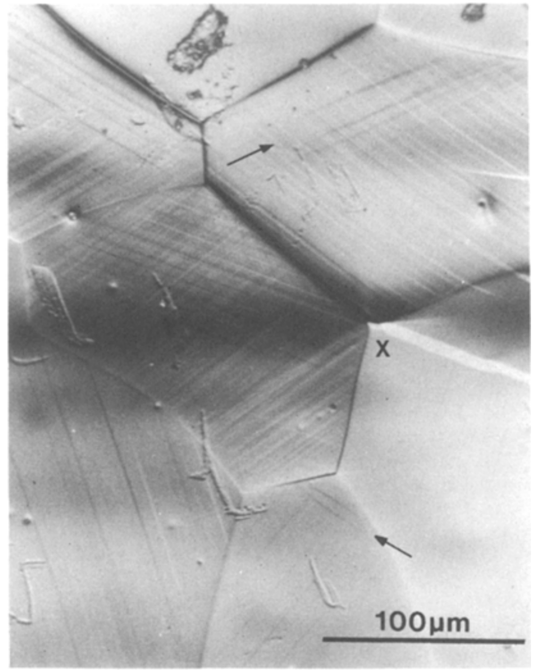

Figure 8 Nomarski interference micrograph of a transverse specimen, grain size ~ 150 μm, strained to 1%. Slip on more than one system occurs in localised regions (e.g. as arrowed). Both a twin and a slip band have been nucleated at the triple point X.

of each grain, additional lines (arrowed) are present in localised regions. Also visible in the micrograph of Fig. 8 is a twin and a slip band apparently nucleated from a triple point (a grain edge in three dimensions) where the need to maintain material continuity is particularly acute.

It was noted earlier that slip lines were always observed, and hence slip must always contribute to the overall deformation strain. Slip not only occurs within untwinned material, but also in the twins themselves. Thus twinning contributes to the overall strain in two ways: firstly from the twinning shear, and secondly by changing the orientation of the matrix to one which may be more favourable for slip. Examples of this can be seen in Fig. 6b. The slip lines in the twins in grain 1 are in a different direction to those in the matrix, indicating that slip had occurred within the twins after they were formed (if the matrix slips before the twin forms, the angle between the slip lines in matrix and twin is very small ($< 8^\circ$, the shear angle) e.g. in grain 3, Fig. 6b).

The observations presented so far were all made on Zn–Al–Mg of large grain size (~ 150 μm). A comparison was made with the deformed alloy of smaller grain size (~ 20 μm) and many similar observations were made. Twins are often almost continuous across many grains within the twin bands, which are separated by regions in which there is practically no twinning. The most striking difference between material of large and small grain size is the distribution of slip lines on the surface. Fig. 9 shows the surface appearance of two longitudinal specimens, both deformed to 2% plastic strain, one of grain size 20 μm and the other of grain size 150 μm (Figs. 9a and b respectively). The slip lines in the specimen of larger grain size are more prominent, and further apart than those in the specimen of smaller grain size (note the different magnifications of Figs. 9a and b). This observation is not surprising if it is assumed that dislocations are nucleated at grain boundaries; in material of smaller grain size there will be more sources of dislocations (particularly grain edges and corners), and hence slip will occur on more planes. The concentration of slip bands as the grain size increases will result in greater stress concentrations at the grain boundaries (in addition to the effect of the increased length of the slip band) and hence twins are more likely to be

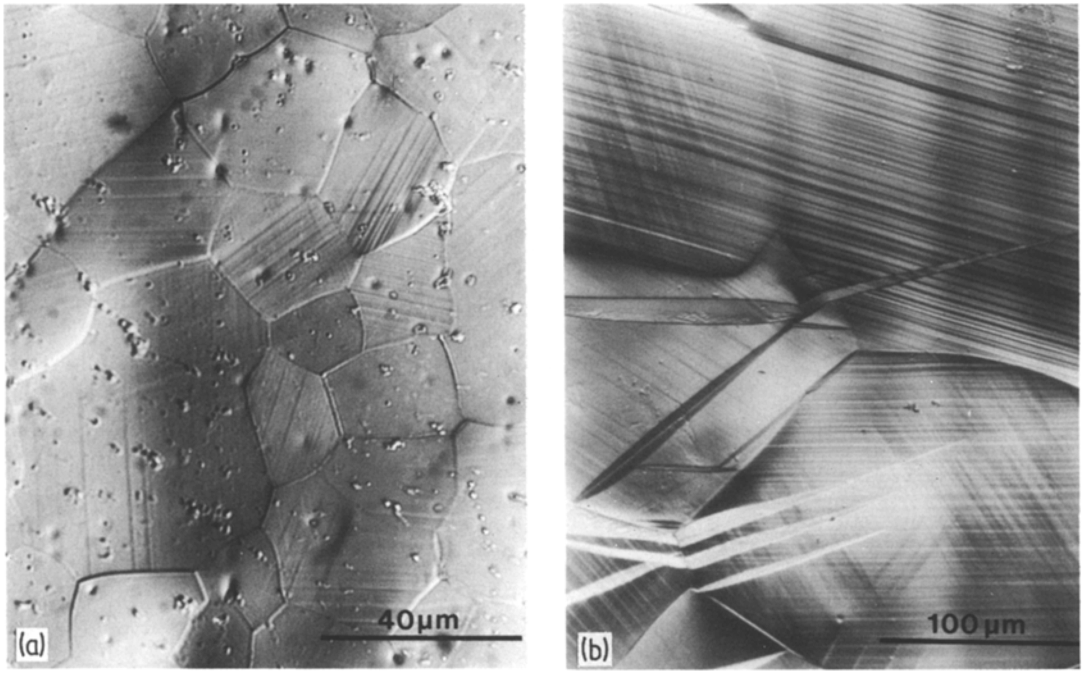

Figure 9 Nomarski interference micrographs of longitudinal specimens strained to 2%. (a) grain size ~ 20 μm and (b) grain size ~ 150 μm. The slip lines in (b) are more prominent and further apart than in (a).

nucleated. These observations at least partially explain the effect of grain size on twinning.

4. Discussion

Longitudinal and transverse specimens were observed to behave differently with respect to twinning. The differences can be rationalised by reference to the maximum Schmid factors for slip and twinning, given the texture of the rolled sheet (Table II). For longitudinal specimens, twinning is relatively unfavourable, while the Schmid factors for slip are high. In transverse specimens, basal slip, which has the lowest critical resolved shear stress, is the least favoured, while the Schmid factor for twinning has almost the maximum possible value. Therefore in transverse specimens twinning and pyramidal slip are expected to be the preferred deformation mechanisms, while in longitudinal specimens basal slip will dominate, especially at low strains. Thus the higher twin density in the transverse specimens and the observation of a critical strain before twinning in longitudinal specimens are predicted by the resolved shear stresses on the slip and twinning systems in rolled zinc sheet.

Differences in behaviour were also found between the surface and the bulk of deformed specimens. In all cases, more of the strain was achieved by twinning in the bulk. If the twinning systems are considered to operate because of the need to maintain material continuity at the grain boundaries, at the surface where this criterion is relaxed less twinning will be required. Thus the presence of surfaces has been shown to be an important factor in determining the mode of deformation. The behaviour of the specimens with the largest grain size can be explained in terms of the low thickness/grain size ratio. The deviation from the expected behaviour in these specimens is greater in the bulk than on the sur-

TABLE II Maximum Schmid factors for slip and twinning in rolled zinc

Slip system	Longitudinal specimens	Transverse specimens
Basal slip		
$(0001)\langle 11\bar{2}0\rangle$	0.470	0.0
Pyramidal slip		
$\{2\bar{1}\bar{1}2\}\langle\bar{2}113\rangle$	0.401	0.313
Twinning		
$\{10\bar{1}2\}\langle\bar{1}011\rangle$	0.164	0.499

Texture: $\{0001\}\ \alpha$ ND-RD ($\alpha = 35°$); $\{10\bar{1}0\} \parallel$ TD (see [1]).

face (Fig. 3), which indicates that the effect of the surfaces extends through the whole specimen.

The observation of a maximum contribution to the total strain from twinning is easily rationalised. The increase with grain size of the strain contribution from twinning cannot continue indefinitely, since it would eventually reach 100%. The greatest contribution from twinning, 85%, is surprisingly high in view of the work on other materials (e.g. Zr, [5]) but reflects both the strong texture which makes basal slip so unfavourable and the high critical resolved shear stress for pyramidal slip, which is the only other alternative deformation mode. However, pyramidal slip is often observed in transverse specimens (where more than one set of slip lines is observed on the surface, at least one must result from the operation of a pyramidal slip system, see Fig. 8).

Not only is there a maximum contribution to the strain by twinning, but a tendency towards a maximum volume fraction of twins is observed in specimens of large grain size (Fig. 1). If, once sufficient material is twinned, the contribution to the strain from slip within twins becomes significant, for a given strain increment fewer new twins will be nucleated. This effect may not only be confined to large grain sizes, but could occur in all specimens at larger strains than those used in this investigation. However, specimens of smaller grain size (transverse) can have a twin volume fraction much greater than the maximum indicated in specimens of large grain size (Fig. 1) suggesting that this effect depends on the size of twins as well as their number.

It has been observed that twins do not form homogeneously, but occur in bands. This suggests an autocatalytic mechanism of formation. This conclusion is supported by observations of twinning and slip on the surface of deformed specimens by interference microscopy. Once one twin is nucleated, for example from a stress concentration caused by a slip band impinging on a grain boundary, it propagates to the edges of the grain in which it is nucleated, causing stress concentrations in all the surrounding grains, which can be relieved by further twinning. Thus a whole band of twinned grains is formed parallel to the original twin (approximately perpendicular to the tensile axis) which extends from the original twin to the specimen edges.

The effect of grain size on deformation twinning has not been quantitatively explained, but it appears to be connected with the formation of fewer slip bands in material of larger grain size. The resulting concentrations of stress will nucleate twins, which in turn generate further twinning. If twinning is regarded as a deformation mode which only operates in the absence of other deformation mechanisms, the predominance of twinning at large grain sizes can be explained by the relative unimportance of pyramidal slip e.g. [8]. The lack of pyramidal slip could also be accounted for by a reduction in the number of available dislocation sources with increasing grain size, as discussed above.

5. Conclusions

1. The contribution of twinning to the overall tensile strain in rolled Zn–Al–Mg at ambient temperatures is a function of grain size and direction of loading, with respect to the texture.

(a) The twin volume fraction varies linearly with strain in most cases.

(b) The critical strain for twinning observed in longitudinal specimens decreases with increasing grain size.

(c) The contribution of twinning to the overall deformation increases with increasing grain size, following a linear law, but becoming independent of grain size at large grain diameters. The linear law remains to be fully explained.

(d) There is a critical grain size below which no twinning is observed.

2. Twinning is not homogeneous, but occurs in deformation twin bands, which suggests an autocatalytic mechanism of formation. The initial twins in a band are probably nucleated at a stress concentration at the end of a slip band.

3. Twinning occurs to a lesser extent near the surfaces of a specimen, which reflects the relaxation of Von Mises criterion near the surfaces.

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