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AN ASSESSMENT OF THE USE OF DIMENSIONAL CHANGES TO DETERMINE GRAIN STRAIN DURING CREEP

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1. INTRODUCTION

Although a considerable volume of work has been published on grain boundary sliding, one major issue has remained unresolved. With only a few exceptions, all of the data appearing in the literature were obtained from measurements taken at the surface, usually in the form of the offsets occurring at grain boundaries, and the question therefore arises as to whether such measurements are representative of the sliding occurring in the bulk of the material. This is particularly important since many of the previous investigations were based on measurements of the offsets occurring perpendicular to the surface, and the extra degree of freedom then available suggests that this parameter of sliding may well be unrepresentative.

In an attempt to resolve this problem, Rachinger[1] suggested taking internal measurements of the grain shape before and after deformation, and attributing any increase in length to the contribution (ε_s) from slip processes within the grains. If the total strain (ε_t) is defined by

$$\varepsilon_t = \varepsilon_s + \varepsilon_{gb} \qquad (1)$$

where ε_{gb} is the contribution to the total strain from grain boundary sliding, it follows that a measure of ε_s yields ε_{gb} by difference. Rachinger applied this technique to aluminum, and found that γ, the percentage contribution to the total strain due to sliding ($= \varepsilon_{gb}/\varepsilon_t$ %), decreased from 87 ± 13% in the interior to 38 ± 12% at the surface. Since measurements of the offsets at the surface also yielded low values for γ ($\sim$ 20%, although these measurements are subject to criticism[2]), it

was concluded that the surface deformation was predominantly by slip, and thus not typical of the interior. To overcome objections by McLean,[3] who suggested that the action of surface tension forces may retain the grains in an equiaxed configuration by migration, Rachinger[4] later obtained additional results which, it was suggested, supported his original thesis. Unfortunately, the evidence from these later results is not conclusive.

Recently, a number of papers have been published quoting results obtained internally using the Rachinger technique, but these have invariably yielded very high values for γ (e.g. up to 78% in Al,[5] 73-88% in a Pb-Tl alloy,[6] 80-100% in Mg alloys,[7] up to 82% in stainless steel,[8] 70-75% in Fe[9]). Furthermore, in those cases where γ was also determined at the surface from measurements of the boundary offsets, it was found that, as with the initial results of Rachinger, the surface values of γ were very much lower.[6,10] It should be noted, however, that an incorrect procedure was used to calculate γ at the surface, as pointed out elsewhere,[2] although the discrepancy involved is not sufficient to account for the large variation observed between γ at the surface and internally.

The purpose of the present investigation was therefore twofold:

(i) to develop a method for unambiguously determining grain strain, and hence γ, at the surface, without reference to grain shape.

(ii) to compare the results with those obtained both at the surface and internally using the Rachinger technique.

2. THEORY

(i) Estimating the contribution of grain boundary sliding to the total strain.

It is not easy to accurately determine the sliding contribution (γ) in polycrystalline materials; the methods available are discussed in detail elsewhere.[2] When grain boundary sliding occurs, the sliding vector at the surface may be resolved into three mutually perpendicular directions; two components, in the plane of the surface (u and w), are revealed by marker lines, and a third, perpendicular to the surface (v), may be observed using an interference microscope. It is particularly advantageous to determine γ from measurements of v, since this requires no preliminary scribing of the surface, and the use of interferometry allows a very accurate determination.

If $\bar{v}_r$ is the average value of v obtained from measurements at randomly chosen boundaries, then ε_{gb} may be computed from an equation of the form[2]

$$\varepsilon_{gb} = k\, n_r\, \bar{v}_r \qquad (2)$$

where k = geometrical conversion factor

n_r = number of grains per unit length measured before testing on lines randomly oriented with respect to the stress axis.

In practice, a calculation of k is difficult from first principles. It has been shown previously[11] that the sliding component v is dependent on the type of surface used, since a polished or "cut" surface represents an unstable configuration, and the boundaries gradually migrate during creep to positions almost perpendicular to the surface. This additional driving force for migration at the free surface tends to smooth out the small boundary irregularities which are formed internally, and lead to

enhanced values of v. If specimens are tested with "annealed" surfaces, however, the boundary configuration is relatively stable, and the sliding component v is thereby reduced. Since any theoretical calculation of k thus requires a detailed knowledge of the factors affecting boundary stability at the surface, it was deemed advantageous to derive a value of k experimentally.

One method of achieving this is by accurately determining the grain strain ε_s, and thus ε_{gb} by substitution in Eq. (1). If this value of ε_{gb}, together with the measured value of $\bar{v}_r$, is then substituted in Eq. (2), a value for k is obtained directly. Since the grain shape technique may be unreliable, ε_s was determined from a series of closely spaced parallel lines on the specimen surface, perpendicular to the stress axis, and of a known initial spacing slightly less than the average grain diameter. The specimen was then creep tested to some known total strain, and ε_s determined by measuring the line separation in those grains which were intersected by two or more lines. In Fig. 1, for example, the stress axis is horizontal, and ε_s can be measured at points A, B, etc.

(ii) Assessing the validity of the grain shape technique.

If measurements are taken to determine the grain shape after creep, ε_{gb} may be calculated and the values compared with those obtained from Eq. (2). Whilst this gives an indication of the reliability of the grain shape technique, it does not usually give a direct comparison, since γ is invariably determined at low strains from offset measurements whereas the grain shape technique can only be conveniently utilized at fairly high strains (greater than $\sim$ 15%). However, a direct comparison is readily possible if a grid is marked on the specimen surface and measurements taken to compare the change in dimensions of the grid with that of

grains. Since the grid is unaffected by movement of the boundaries, and thus not subject to the criticism that the boundaries may migrate to maintain an equiaxed structure, a comparison of ε_s determined by these two methods will directly indicate the validity of the grain shape technique.

3. EXPERIMENTAL TECHNIQUES

The material used in this investigation was Magnox AL80, a Mg-0.78 wt. % Al alloy supplied by Magnesium Elektron Ltd., Manchester; a spectrographic analysis is given elsewhere.[11] Tensile specimens, about 7 cm in length, were prepared from a 1.27 cm diameter rod, with two parallel longitudinal flat faces each approximately 3 cm in length. Specimens were annealed for 2 hrs at temperatures in the range 430-540°C to give varying grain sizes. Prior to testing, the grain size of each specimen was carefully determined using the linear intercept method; this revealed that the grains were elongated $\sim$ 2-5% in the longitudinal direction. Specimens were tested in Dennison Model T47E creep machines under constant load at temperatures in the range 150-300°C. At temperatures of 200°C and below, tests were conducted in air with the polished flat faces coated with a thin film of silicone oil to prevent oxidation; at higher temperatures, specimens were tested in an argon atmosphere.

To determine $\bar{v}_r$, each test was interrupted at regular increments of strain and the specimen removed from the machine. At the lower strains, when $\bar{v}_r$ was less than about 1 μm, measurements were taken on a Zeiss Linnik interference microscope; higher mean step heights were determined by directly focusing on either side of the boundary with a calibrated microscope. In each case, an average value ($\bar{v}_r$) was determined by taking measurements at 300 randomly selected boundaries. A fuller account of

the experimental procedure has been given previously.[11]

The majority of specimens were tested with "cut" surfaces, by polishing them after the heat treatment; however, some specimens were only polished prior to the heat treatment, and then tested with "annealed" surfaces.

For measurements of ε_s, a grid was photographically printed onto the test surface of a number of specimens before testing, using a technique described elsewhere.[12] After printing, the image consisted of a grid of sharply defined lines, with a spacing of 0.1267 ± 0.0003 mm; the appearance of a typical grid after testing is shown in Fig. 2.

After straining, the separation between grid lines was measured on the screen of a projection microscope, by aligning the edge of a line with a vertical ruling on one side of the screen, and measuring the distance to the edge of the next line. At a magnification of X1000, it was possible to measure each separation to an accuracy of $\pm 5 \times 10^{-5}$ cm. A total of 300 measurements was taken in each case, by carrying out a longitudinal traverse of the specimen along one of the grid lines and measuring the line separation at every instance where two adjacent lines were contained within the same grain.

A preliminary test was carried out to check the reliability of this technique, by straining a specimen for 11 hrs to $\sim$ 5% total strain at 100°C. This temperature was chosen since it offered more ductility than at room temperature, but was sufficiently low that, at this high rate of strain, the grain boundary sliding contribution was known to be negligible.[13] By calculating the average grain strain and relating this to the overall extension, it was found that 100 ± 7% of the deformation was due to strain within the grains, thereby confirming the reliability of this method.

To determine the grain strain ε_s, Rachinger[1] suggested measuring the number of grains per unit length in directions parallel and perpendicular to the stress axis. Recently, Hensler and Gifkins[14] have pointed out that it is better to measure the maximum length (L) and breadth (B) of individual grains, since this eliminates the scatter due to irregularities in grain size. The maximum dimensions of individual grains were recorded in arbitrary units, both at the surface and internally, using a crossed grid in the eyepiece of a microscope. ε_s was then calculated from the equation

$$\varepsilon_s = \left(\frac{b\ L}{\ell\ B}\right)^{2/3} - 1 \qquad (3)$$

where ℓ and b, the original length and breadth before testing, were included to allow for the slight elliptical nature of the grains. Using the procedure of Sellars,[15] the grain strain was determined for each individual grain from the measured value of L/B and the known average value of b/ℓ. ε_s was taken as the average of these values after measurements on a total of 150 randomly chosen grains.

4. EXPERIMENTAL RESULTS

(i) Values of k for use in equation 2.

When measurements of v are taken at regular intervals throughout a creep test, it has been shown that, if a "cut" surface is used, $\bar{v}_r$ initially increases linearly with increasing strain. This linearity ceases towards the end of secondary creep, and $\bar{v}_r$ thereafter increases at a slower rate than the strain. In a previous investigation, it was shown that the end of the linear portion occurred at approximately the same strain as that at which the internal boundaries reached stable positions almost perpendicular to the surface.[11]

A number of specimens, having "cut" surfaces, was prepared with grids printed on the surface, and creep tested to total strains slightly greater than 2%. This strain was cho en for a determination of ε_s since it was not easy to measure the extremely small grain extensions a $\varepsilon_t < 1\%$; furthermore, earlier results on specimens without printed grids had clearly shown that this strain was still on the linear portion of the $\bar{v}_r$ versus strain curves.[11] Preliminary tests using specimens both with and without printed grids revealed no difference in their creep behavior, or in the values of $\bar{v}_r$ at the surface, and it was therefore concluded that the presence of a printed grid had no effect on the surface behavior.

Each specimen was removed from the testing machine at regular intervals during straining, and measurements taken to determine $\bar{v}_r$. Thus, the value of $\bar{v}_r$ at the final strain (ε_t) was determined with a high degree of accuracy, since it was known that $\bar{v}_r$ increased linearly with strain in this region. At $\varepsilon_t \sim 2\%$, 300 separate readings were taken of the spacing between grid lines within individual grains, and these were used to calculate ε_s, and thus γ, from Eq. (1). A value of k was obtained by substitution of ε_{gb} and $\bar{v}_r$ in Eq. (2).

The detailed results are listed in Table I, where the error bars on k represent the 95% confidence limits. Although there is some scatter in the individual values of k, they all lie within the range 1.00 - 1.20. A mean value of k of 1.07 was used to determine γ for specimens tested without printed grids, details of which are published elsewhere.[11] The results in Table I show that γ increases with decreasing stress, or, for constant stress, decreases with increasing grain size, in a manner similar to that reported previously.[11] This trend is of a sufficiently large magnitude that it cannot be accounted for merely by the experimental

scatter in the values of k; however, the scatter is sufficient to mask this trend for the three specimens tested in the range 3500-4000 psi.

Further tests were undertaken to determine the dependence of k on (a) the type of surface, and (b) strain; the results are summarized in Table II. At $\varepsilon_t \sim 1.2\%$ (specimens MX68 and MX79) the value of k on the "annealed" surface was larger than that obtained on the "cut" surface for specimens tested under identical conditions, although the values of γ were approximately the same in each case. A similar trend was also observed at a higher strain. This suggests that, in the case of a "cut" surface, it is only the component of sliding perpendicular to the surface (v) which is affected to any great extent by the additional migration, presumably since sliding in the plane of the surface is still impeded by the presence of triple points.

For all three specimens, having both "cut" and "annealed" surfaces, larger values of k were obtained at the higher strain. This trend is readily explicable, since $\bar{v}_r$ has been shown to increase at a slower rate at the higher strains (i.e. $d^2 \bar{v}_r/d\varepsilon_t{}^2$ is negative after the end of the initial linear region),[11] and higher values of k are therefore required to compensate for this. In fact, the increase in k was sufficient to maintain γ approximately constant. It seems unlikely that this is the case, however, since the boundaries tend to become very corrugated during creep and this would tend to limit the sliding. A more likely explanation is that there is a finite contribution from cavitation (ε_c) at the larger strains, so that Eq. (1) should be modified to read

$$\varepsilon_t = \varepsilon_s + \varepsilon_{gb} + \varepsilon_c \qquad (4)$$

By omitting this additional term, ε_{gb} is probably overestimated.

(ii) γ from grain shape measurements.

Several specimens were taken to high strains (> 15%), under varying experimental conditions, and then sectioned parallel to the stress axis, polished and etched. Measurements of the grain dimensions were taken, and γ determined by use of Eqs. (3) and (1). For some specimens, measurements were also taken on the original surface, or on surfaces that had been repolished during the test. Full details of the results are given in Table III, with the error bars again representing the 95% confidence limits.

To obtain correlation with the results obtained for γ by offset measurements, the values of γ determined at the surface from $\bar{v}_r$ are also shown, both at the final strain and at a strain of 1%. It should be noted that in both cases the values of γ were calculated by taking k = 1.07, since the dependence of k on strain was not fully known. However, it has already been shown that k increases with increasing strain (Table II), and thus γ from $\bar{v}_r$ is underestimated at the final strain. Even taking this underestimation into account, the results show clearly that the grain shape technique invariably yields exceptionally high values for γ.

(iii) Comparison of γ by grain shape and the printed grid technique.

Specimen MX74, with a "cut" surface, was used to compare γ by grain shape and the printed grid technique. The specimen was strained to 16.77% under the experimental conditions shown in Table I, and surface readings taken to determine γ by

(i) The average L/B ratio of the grains.

(ii) The average L/B ratio of the printed grid lines within the grains.

The results are shown in Table IV. The lower value of the average L/B ratio from the grain shape measurements confirms that migration is

tending to "round-off" the grains, and the true change in dimensions is thus given by the ratio obtained from the printed grid. Table IV shows that the grain shape technique has overestimated γ by $\sim$ 90% in this case.

To check whether the use of the grain shape technique at the surface was affected by the type of surface tested, ε_s was also determined from grain shape measurements for specimen MX79 with an "annealed" surface (see Table II). At a total strain of 13.18%, the surface value of γ was determined as 59.0%, in contrast to the value of 43.2% from the printed grid. Thus, the general trend for grain shape measurements to overestimate ε_s, and thereby underestimate γ, is not dependent solely on the type of surface tested.

5. DISCUSSION

The results in Table I show that, at constant strain, there is no systematic variation of k with stress, within the fairly large error bars inherent in these measurements. However, it appears that k depends critically on the type of surface, and also on the total strain (Table II).

The method of determining k is, in effect, one of measuring ε_{gb} in in the plane of the surface, and then relating this to the offsets perpendicular to the surface. A recent investigation using internal oxide markers in this material has shown that there is little difference between internal sliding and that taking place in the plane of the surface, regardless of whether the surface is "cut" or "annealed."[16] A similar trend has also been reported for aluminum using "cut" surfaces only.[17] The similarity between the two types of surface is revealed inthe present investigation by the almost identical values obtained for γ from the grid measurements for specimens MX68 and MX79 at $\varepsilon_t \sim 2\%$ (Table II). Since

the value of $\bar{v}_r$, at constant strain, is lower for an "annealed" surface, it follows that higher values of k will pertain to that type of surface. The average value of k ($\sim$ 1.07) obtained on "cut" surfaces at low strains, where the $\bar{v}_r$ versus strain curve is linear, is somewhat lower than the value of 1.58 calculated theoretically by McLean[18] and used by many subsequent investigators.

The results shown in Table III reveal two interesting trends. Firstly, using the grain shape method, the value of γ in the interior are always slightly larger than those obtained at the surface. This trend is similar, but of a much smaller magnitude, to that originally reported by Rachinger,[1] although Ishida et al.[17] found no systematic difference between the surface and interior using the grain shape technique. It should be noted, however, that a direct comparison with previous work is not easy, since Rachinger[1] used specimens having "annealed" surfaces whereas Ishida et al.[17] used specimens with "cut" surfaces.

A second feature of Table III is that the grain shape method always appears to substantially overestimate the value of γ, when the results are compared with those obtained from measurements of $\bar{v}_r$ at the same total strain. This observation is true even when allowance is made for the increase in k at the higher strains which would lead to larger values of γ than those quoted in the Table. In general, the values obtained for γ on the surface by the grain shape method are $\sim$ 2 - 4 times larger than those from offset measurements, although there is no obvious relationship between the two results. The difference bewteen surface values of γ from $\bar{v}_r$ measurements and interior values from grain shape is even larger, but there appears to be a better correlation in

those cases where the initial sliding contribution is known to be small (e.g. specimens MX21 and MX31).

The results of Table III strongly suggest that the grain shape technique is invalidated by the tendency for migration to "round off" the grains and maintain them in an equiaxed configuration; this suggestion is supported by the results shown in Table IV. Some indirect evidence for spheroidization was obtained since it was observed that corrugated boundaries were formed internally during creep, particularly under test conditions where γ was initially relatively small. The presence of such boundaries indicates an absence of any appreciable migration on a large scale, and in those cases better agreement was obtained between the grain shape and offset measurements. This observation therefore supports the suggestion that sliding and migration are inter-related processes.[19,20]

6. CONCLUSIONS

(i) By using a grid photographically printed onto the specimen surface, an accurate determination was made of the grain strain, ε_s, occurring during creep, and hence the grain boundary sliding contribution, γ.

(ii) To permit the use of measurements of offsets occurring perpendicular to the surface ($\bar{v}_r$) for the calculation of γ, an average value of k = 1.07 was obtained experimentally for use in the equation

$$\varepsilon_{gb} = kn_r \bar{v}_r .$$

(iii) The values of k appeared to be independent of stress and grain size, but dependent on the total strain and the type of surface (i.e. whether "cut" or "annealed").

(iv) Values of γ determined by the grain shape technique were usually $\sim 2 - 4$ times larger than those obtained from offset measurements, although there appeared to be some agreement for those specimens in which the sliding contribution was initially very small (< 20%).

(v) A comparison of ε_s determined by the grain shape technique and by measuring the dimensional changes of a printed grid contained within individual grains confirmed that migration was tending to "round off" the grains.

(vi) It is suggested that the grain shape technique is inaccurate for determining the grain boundary sliding contribution, except possibly in those cases in which the sliding contribution is very small.

Table I. Determination of k using printed grid technique

Specimen Number	No. of grains/cm	Stress p.s.i.	Temperature °C	ε_t	ε_s	γ	$\bar{v}_r$ (μm)	k
MX33	54	3500	200	2.01%	1.04%	48%	1.795	1.00 ± 0.15
MX23	60	3750	200	2.17%	0.90%	58%	1.842	1.14
MX67	45	4000	200	2.40%	1.39%	42%	1.960	1.13
MX68	54	4500	200	2.20%	1.26%	43%	1.725	1.02 ± 0.17
MX69	52	6000	200	2.11%	1.56%	26%	0.908	1.20
MX73	26	4000	200	2.08%	1.45%	30%	2.212	1.03
MX74	29	6000	200	2.11%	1.85%	12%	0.858	1.00

Table II. Dependence of k on strain and type of surface

Specimen Number	Type of Surface	No. of grains/cm	Stress p.s.i.	Temperature °C	ε_t	ε_s	γ	$\bar{v}_r$ (μm)	k
MX68	"Cut"	54	4500	200	2.20%	1.26%	43%	1.725	1.02
MX68	"Cut"	54	4500	200	11.41%	6.62%	42%	5.080	1.75
MX74	"Cut"	29	6000	200	2.11%	1.85%	12%	0.858	1.00
MX74	"Cut"	29	6000	200	16.77%	15.08%	10%	4.032	1.45
MX79	"Annealed"	55	4500	200	2.13%	1.17%	45%	1.342	1.41
MX79	"Annealed"	55	4500	200	13.18%	7.49%	43%	5.180	2.01

Table III. Determination of γ from grain shape measurements

Specimen Number	No. of grains/cm	Stress p.s.i.	Temperature °C	ε_t	γ at surface using $k = 1.07$ with $\bar{v}_r$		γ at ε_t from grain shape	
					At 1%	At ε_t	Surface	Interior
MX21	100	8000	150	25.4%	17%	4%	-	7 ± 4%
MX2	104	4000	200	19.0%	63%	23%	31%	41%
MX25	104	6000	200	21.5%	36%	10%	20%*	29%
MX31	107	8000	200	30.8%	15%	8%	-	7 ± 6%
MX3	84	6000	200	26.9%	30%	13%	45 ± 8%	55 ± 9%
MX8	53	6000	200	19.2%	21%	9%	20 ± 8%	33 ± 7%
MX43	108	2000	300	19.0%	58%	27%	50%*	68%
MX7	55	4000	300	35.9%	29%	8%	-	45 ± 12%

* Surface repolished during test (MX25 at 10.0%; MX43 at 2.5%).

Table IV. Comparison of γ by grain shape and the printed grid (Specimen MX74)

ε_t	Method	$(\overline{L/B})$	γ from Eq. (3)
16.77%	Grain shape	1.215	18.8%
16.77%	Printed grid	1.237	10.1%

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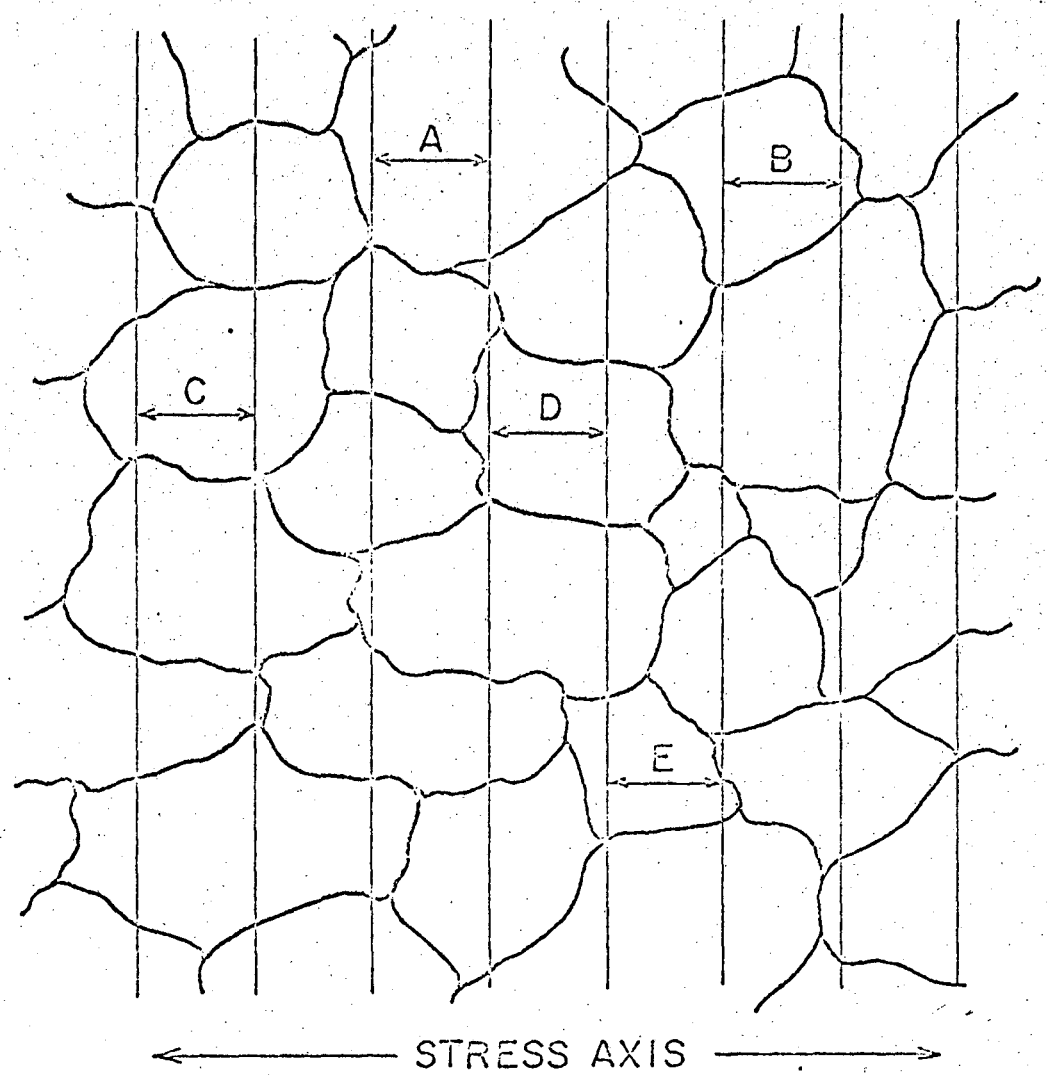

FIG. 1 Schematic representation of a surface prepared for measuring grain strain; ε_s is determined from the separation between the lines at points A, B, etc.

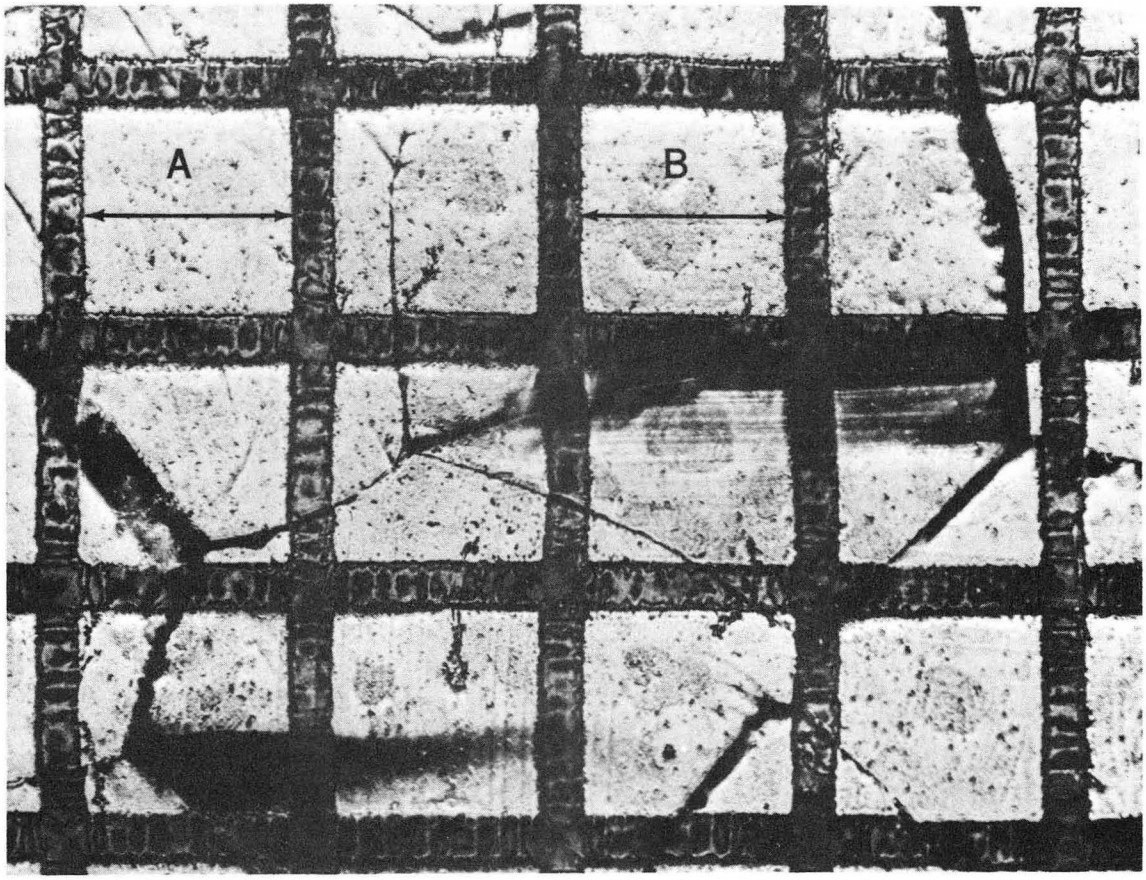

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Fig. 2 The appearance of a printed grid on the specimen surface after straining ~ 2% at 200°C. Stress axis horizontal. ϵ_s is determined from measurements at points A, B, etc. Magnification ~~X220~~ 300.

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