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1. INTRODUCTION

When a constant load or stress is applied to a metal over a prolonged period of time, particularly at high temperatures, plastic flow occurs and the metal very slowly deforms. This time-dependent deformation, which occurs by a thermally activated process, is known as "creep". In this chapter, we will see how quantitative metallography may be applied to creep-tested specimens to yield information on the importance of different deformation mechanisms; such an approach has a wide application in materials science.

During creep tests, measurements are usually taken of the total strain at regular increments of time, and creep curves plotted in the manner shown in Fig. 1. In general, there is an instantaneous strain, ε_o, and then three distinct stages of creep. Primary creep (Stage I), in which the creep rate is decreasing with time, represents the period in which a stable substructure is developing within the grains of the material. This substructure usually remains unchanged during secondary, or steady state, creep (Stage II), and the creep rate then remains essentially constant with increasing strain. Tertiary creep (Stage III) is a period in which the creep rate rapidly increases, ultimately leading to failure of the material. Under conditions of constant load, rather than constant stress, the secondary stage may pass rapidly into an extended tertiary region, since the stress is then continually increasing throughout the test.

In Fig. 1, curve a is typical of that obtained at low temperatures; an increase of temperature or stress will give curves b and c. For pure polycrystalline material, tested at constant stress, the total strain marking the onset of the secondary or tertiary stages is usually insensitive to temperature. However, there is a strong stress dependence such that the onset of each stage occurs at a higher strain for an increase in stress.

Since creep is a thermally activated process, the creep rate $\dot{\varepsilon}$ may be represented by an Arrhenius-type equation of the form

$$\dot{\varepsilon} = Ae^{-Q/RT}$$

where $A \overset{\sim}{} $ constant*

Q = activation energy for creep

R = gas constant

T = temperature in degrees Kelvin.

Experiments on a large number of materials have shown that the activation energy for creep is approximately equal to that for self-diffusion at temperatures in the range 0.5-0.9 T_m, where T_m is the melting point of the material in degrees Kelvin.

The predominant processes of deformation involved in the creep of metals at high temperatures are:

(i) Dislocation glide, giving rise to crystalline slip.

(ii) Dislocation cross-slip and climb, giving rise to recovery and sub-grain formation.

(iii) Grain boundary sliding.

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* A is constant for any given stress but the approximation arises due to a slight dependence on temperature.

(iv) Grain boundary migration.

(v) Vacancy diffusion.

Of these processes, (i), (ii) and (v) are transgranular, so that they contribute to the extension within the individual grains. Process (iv) refers to the bodily movement of the boundary in a direction perpendicular to the boundary plane, and this makes no significant contribution to the total strain. Process (iii) refers to the displacement of adjacent grains relative to each other by movement along their mutual boundaries, and this contributes to the overall strain but not to the strain within individual grains.

The mechanisms by which a metal deforms under creep conditions are dependent upon the applied stress, the temperature, and the grain size. The temperature dependence is particularly important since the grain boundaries act as a source of strength in the material at low temperatures, by obstructing the movement of dislocations from one grain to the next. As the temperature is raised, however, the strength of the boundary region decreases rapidly, until a critical temperatures is reached at which it is equal to the strength of the grains. At temperatures greater than this, an important part of the deformation is due to grain boundary sliding, and the fracture changes from predominantly transgranular to predominantly intergranular. In practice, it is found that this critical temperature is of the order of 0.4-0.5 T_m for specimens tested under slow rates of strain.

2. DETERMINING THE IMPORTANCE OF GRAIN BOUNDARY SLIDING

Grain boundary sliding is an extremely important process in the deformation of materials at high temperatures, since a high incidence

of sliding may lead to the formation of small voids or cavities in the boundaries, and the linking of these will ultimately give rise to intergranular failure. However, it is not easy to accurately determine the contribution made by grain boundary sliding to the overall strain.

If the transgranular processes, such as crystalline slip, contribute an extension ε_s to the overall strain, and grain boundary sliding contributes an extensive ε_{gb}, then the total extension ε_t is simply the sum of these two parts, such that

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

In practice it is usually more convenient to express the contribution made by grain strain or grain boundary sliding as a percentage of the overall extension, viz. $\varepsilon_s/\varepsilon_t \% = \beta$, and $\varepsilon_{gb}/\varepsilon_t \% = \gamma$.

Measurements of grain boundary sliding are best undertaken at the surface of a metal, and the question therefore arises as to whether such measurements are typical of sliding in the interior. Recently, experiments have been reported using specimens with internal markers, and these indicate that there is little difference between sliding at the surface and in the interior; in this chapter we will therefore restrict ourselves entirely to surface observations.

When two surface grains, X and Y, slide over each other in a polycrystalline specimen, offsets are produced at the boundary, in the manner shown schematically in Fig. 2. If $\overrightarrow{AC}$ represents the sliding vector, then u is the component of sliding resolved along the stress axis, w is the component in the plane of the surface perpendicular to the stress axis, and v is the component measured perpendicular both to

the stress axis and to the specimen surface. If we also define θ as the angle between the stress axis and the trace of the grain boundary in the plane of the surface, and ψ as the internal angle between the boundary and the surface measured on a longitudinal section, then it follows that

$$u = \frac{v}{\tan \psi} + \frac{w}{\tan \theta} \tag{2}$$

Equation (2) is difficult to use in practice, since the internal angle ψ can only be measured by sectioning the specimen. A more obvious way of determining the grain boundary sliding contribution is by scribing a line of known length on the specimen surface, parallel to the stress axis, and measuring the u offset at every point along the line. It then follows that the strain due to sliding is given by

$$\varepsilon_{gb} = \frac{1}{\ell} \sum_{o}^{\ell} u \tag{3}$$

$$= n_{\ell} \bar{u}_{\ell} \tag{4}$$

where n_{ℓ} is the number of grains per unit length, parallel to the stress axis, in the unstrained specimen, and $\bar{u}_{\ell}$ is the average u offset determined from longitudinal markers.

Unfortunately, it is often difficult to measure the longitudinal (u) offsets at positions along a longitudinal marker, since boundary migration or other topographical changes will tend to obscure the points at which the line meets the boundary. This is clearly shown in Fig. 3, from which it is apparent that a better procedure is to take readings of u along a transverse line. In this case, however, it is also

necessary to measure the angle θ in the plane of the surface between the boundary and the stress axis at every point of intersection of the marker with a boundary. ε_{gb} is then calculated from the equation

$$\varepsilon_{gb} = n_t \overline{(u \cdot \tan \theta)}_t \qquad (5)$$

where the subscript t denotes measurements taken along a transverse traverse.

The angle θ is included in Eq. (5) to compensate for the fact that the average value of u determined from a transverse traverse ($\bar{u}_t$) is not the same as the average value determined from a longitudinal traverse ($\bar{u}_\ell$). This difference arises since the average angle of intersection between a line drawn longitudinally and the grain boundaries so intersected is not 45°, because of the greater tendency of the line to intersect boundaries which are perpendicular to it. In fact, it can be shown theoretically, and is easily demonstrated experimentally (see section 6), that the average angle is $\bar{\theta} = 57.3°$. For a transverse traverse it therefore follows that the average angle between the intersected boundaries and the stress axis is $90° - 57.3° = 32.7°$. By multiplying the u offset with the tangent of the angle θ at each point along a transverse traverse, and taking the mean resultant value, as indicated in Eq. (6), this difference between the two traverses is eliminated.

An alternative method of obtaining γ is by determining the average displacement, $\bar{v}$, perpendicular to the surface, between adjacent grains. This procedure is particularly attractive, since individual displacements may be measured with a high degree of accuracy, and the method requires no preliminary scribing of the specimen surface. A difficulty arises,

however, since, in order to calculate γ, it is necessary to convert from an average offset perpendicular to the surface to a strain along the stress axis.

In practice, γ is best calculated from the equation

$$\gamma\ \% = 100\ \frac{k\ n_r\ \bar{v}_r}{\varepsilon_t} \qquad (6)$$

where k is a geometrical conversion factor, n_r is the number of grains per unit length measured before testing on lines randomly oriented with respect to the stress axis, and $\bar{v}_r$ is the average displacement perpendicular to the surface from randomly chosen boundaries. A method of determining k experimentally is described in section 5.

3. PREPARATION OF THE MATERIAL

Magnesium is a suitable metal for grain boundary sliding observations, particularly since the limited number of available slip systems suggests that the sliding contribution may be high. This material has a close-packed hexagonal structure, with a c/a ratio of 1.623 at room temperature. The melting temperature is 650°C.

Specimens are prepared from either rod or sheet; typical dimensions for specimens produced from 0.5" diameter rod are given in Fig. 4, showing two parallel test faces approximately 0.25" apart. Annealing is carried out to give the required grain size, but, prior to this, it is advisable to give the test faces of the specimen an initial rough polish on emery paper to remove the machining grooves. It is not possible to stipulate an exact annealing temperature to give a particular grain size, since this will depend on the extrusion conditions of the material. In general,

however, specimens annealed for 2 hours at temperatures in the range 450-580°C should provide a convenient grain size of the order of 25-50 grains/cm. The annealing should be carried out in an argon atmosphere, or in a close-fitting magnesium tube containing a few granules of titanium to absorb any oxygen.

After annealing, the specimen is removed from the furnace, allowed to cool, and polished on successively finer grades of SiC paper and diamond paste. Finally, the specimen is electro-polished for 60 minutes in a solution of 350 ml. of ortho-phosphoric acid and 625 ml. of ethyl alcohol, using a current density of about 0.5 A/dm^2. To obtain the correct current, it is often convenient to "stop-off" all but one test face of the specimen with a suitable lacquer. It is essential to wash the specimen in running water immediately after removal from the solution, since the free acid formed on hydrolysis will tend to attack the surface. After a few seconds in water, the specimen is rinsed quickly in methyl alcohol and dried.

In order to measure the grain size, the boundaries are revealed by etching in a suitable reagent. Typical reagents for magnesium are a 10% acqueous solution of acetic acid (etching time = 20-30 seconds) or 2% nital (etching time = 10 seconds). The grain size is determined by measuring the number of boundary intercepts per unit length, prior to straining, along a number of traverses both parallel, perpendicular, and randomly oriented with respect to the stress axis (to give n_ℓ, n_t and n_r respectively). Measurements should be taken covering the whole of the test surface, and a comparison of n_ℓ and n_t will show whether the grains are entirely equi-axed. Finally, the specimen is repolished on

fine diamond paste, and the electropolishing repeated as before but without the final etch.

4. MEASUREMENT OF GRAIN BOUNDARY SLIDING

Prior to testing, fine lines are scribed on the test face of the specimen perpendicular to the stress axis, using either a very fine needle or a diamond stylus attached to the moving stage of a travelling microscope.

Creep tests are carried out under conditions of constant load or constant stress at a temperature of 200°C (0.51 T_m). This temperature is chosen since it is sufficiently high for grain boundary sliding to occur, but not so high that problems arise with oxidation of the surface. Surface oxidation is easily prevented at 200°C by coating the test face in silicone oil; at higher temperatures it is usually necessary to perform the tests in an argon atmosphere.

A gauge length is carefully scribed on the specimen before testing, by placing two lines, perpendicular to the stress axis, at either end of the test face. The separation between these lines is measured with a travelling microscope, and the cross-sectional dimensions of the specimen are determined using a micrometer. The test face is then coated in silicone oil, and the specimen introduced into the furnace at the test temperature. After the temperature has been regained, it is held steady for at least an hour before applying the load, to permit thermal equilibrium throughout the specimen.

A suitable load is required to give an extension of the order of 3-4% in about 1 week. If the extension is much greater than this, the contribution from grain boundary sliding will probably be small. On the

other hand, at slower strain rates the sliding contribution will be higher, but the offsets produced may be hardly discernible because of the small total extension. A suitable load to achieve this result will probably be of the order of 1000-2000 lb/in^2 (1 lb/in^2 = 7x10^{-2} kg/cm^2) for a specimen of 25-50 grains/cm.

If the specimen is removed from the furnace at regular increments of time, such as each morning over a period of one week, the new gauge length may be measured and the total strain calculated. Alternatively, many creep machines are fitted with dial gauges, which directly indicate the total extension. Using this data, a creep curve is then plotted of the type shown in Fig. 1.

After creep testing, the specimen is removed from the furnace, washed in methyl alcohol to remove the silicone oil, and the new gauge length carefully measured to determine the overall extension ε_t. Before making the detailed measurements described in this section, the specimen surface is critically examined under a microscope, and a record made of any prominent deformation features. For example, folds may be observed emerging from some triple points, due to the stress built up by grain boundary sliding. A typical fold is shown in Fig. 5.

4.1. Offsets Perpendicular to the Surface

On examining the specimen under the microscope, it will be observed that many of the surface grains have been displaced relative to each other, and discrete steps occur at the grain boundaries between two adjacent grains. This is clearly shown in Fig. 6, in which a specimen was tested having two polished faces mutually perpendicular to each other. By the use of a microscope having a calibrated fine focussing

knob, the height of individual steps can be determined by recording the separate readings when the microscope is focussed at oxidation pits on either side of the boundary. Because of the inherent difficulties of this technique, it is usually only possible to measure step heights to an accuracy of ± 0.5μm.

If the individual steps are small, less than about 1μm in height, they are more easily measured by the use of interferometry. This is best carried out by using an interference microscope, since this allows the fringes to be freely rotated into any direction, but, if this is not available, equally acceptable results can also be obtained using an interference attachment with an ordinary microscope.

The principle of the interference microscope is illustrated schematically in Fig. 7. A beam of light from a white or monochromatic source is divided by a beam-splitting prism, so that half of it passes to the specimen whose surface contour is required, and the other half is reflected from a plane mirror, of chosen reflectivity, to provide a reference wavefront. The two reflected beams are then recombined to form interference fringes, which are observed in the microscope eye-piece. The separation between the fringes is equal to $\lambda/2$, where λ is the wavelength of the light source. Thallium is often used as the monochromatic light source, and the separation between the lines is then 0.27μm.

To determine the step height, the fringes are rotated with a system of optical wedges so that they lie perpendicular to the grain boundary under observation. When using an interference objective with an ordinary microscope, however, it is necessary to rotate the

specimen instead. If there is a step at the boundary, this will result in a sharp displacement of the fringes from one side of the boundary to the other, and an estimate is then made of the step height to an accuracy of ∿0.2 of a fringe (∿0.06μm). It may sometimes be observed that the boundary has migrated, or moved laterally, so that a number of boundary traces are then visible. In those cases it is still possible to measure the step height with the same degree of accuracy by simply extrapolating the fringes from one side of the boundary to the other.

All measurements should be taken using monochromatic light of a known wavelength. However, this provides fringes of the same intensity on either side of the boundary, and it is therefore not possible to distinguish steps that are greater than one fringe displacement. To check this, it is necessary to switch to a white light source, which then gives an interference pattern having a dark central fringe and fringes of decreasing intensity on either side. Using white light, an estimate is made of the total number of fringes displaced, if greater than one, and the final reading obtained using monochromatic light in the usual way. Typical offsets obtained with a white light source are shown in Fig. 8. This figure also reveals slip lines in a number of the grains, and interferometry may be used to determine the maximum slip height.

It may sometimes happen that one or two very large steps, greater than about 2μm, occur on the specimen surface. These are difficult to measure since, on checking the fringe displacement with the white light source, it is often found that the dark central fringe on one side of the boundary is displaced into the neighboring grain. This

problem is overcome by calibrating the focussing screw of the interference microscope in microns, by noting the depth of focus between the top and bottom of an impression of known depth. The large steps are then measured accurately using white light, by focussing on one side of the boundary and noting the position of the central fringe, and then re-focussing on the other side so that the central fringe is in the same position. The step height is recorded directly by noting the distance rotated by the focussing screw.

Measurements are taken to determine the mean step height, $\overline{v}_r$. This is done by carrying out a longitudinal traverse of the specimen, using a magnification such that the average grain diameter is less than half of the field of view, and recording the step height at any randomly selected position on every grain boundary passing through the field of view. On completion of one traverse, the field is changed to a new position and the process repeated. A large number of readings are required to determine the mean step height at a statistically acceptable level.

To determine the error bars, representing the 95% confidence limits, the Standard Deviation, σ, is calculated from the equation

$$\sigma^2 = \frac{\sum \overline{v}_r^2 - \{\sum \overline{v}_r\}^2/N}{N-1} \qquad (7)$$

where N is the total number of readings taken.

The Standard Error of $\overline{v}_r$ is obtained from

$$\text{S.E.}(\overline{v}_r) = \frac{\sigma}{\sqrt{N}} \qquad (8)$$

To calculate the 95% confidence limits, the Standard Error of $\bar{v}_r$ is multiplied by 1.96. In general, it will be found that about 300 individual readings of v_r are required to give an accuracy of ±10% at the 95% confidence level.

The value of $\bar{v}_r$ obtained in this way, together with the measured value of k (see section 5), may then be inserted into Eq. (6) to yield γ.

4.2. Offsets in the Plane of the Surface

To measure the offsets in marker lines scribed on the surface, it is convenient to use a projection microscope and take readings directly from the screen. A number of transverse lines are traversed, and readings taken of both u_t and θ_t at each point where a grain boundary is intersected by a marker. An estimate of ε_{gb}, and hence γ, is then obtained from Eq. (5).

A large number of readings is again required to obtain a statistically acceptable result; for example, after taking 300 readings of u and θ, the error bars will probably be of the order of ±15% at the 95% confidence level. Typical offsets in a transverse marker are shown in Fig. 9, which also shows evidence of extensive gain boundary migration.

If marker lines are also scribed on the surface parallel to the stress axis, measurements may be taken to determine $\bar{w}_\ell$, the average w offset determined from longitudinal markers. Unfortunately, it is not possible to obtain an accurate determination of ε_{gb} solely from measurements of w, since this component of sliding is independent of the v component perpendicular to the surface. In practice, it is found that the sliding component is enhanced perpendicular to the surface, since movement is then able to take place without restriction, whereas in the

plane of the surface the movement is restricted by the adjacent grains. This is taken into account when taking measurements of u, since this contains both a v and a w component (Eq. (2)).

The difference between the w and the v components can be easily verified by determining $\bar{w}_\ell$ from a longitudinal traverse, and then re-traversing the same line and taking measurements of v at the same boundaries to give $\bar{v}_\ell$. The results will reveal that, although v and w both represent components of sliding measured perpendicular to the stress axis, $\bar{v}_\ell$ is larger than $\bar{w}_\ell$.

5. MEASUREMENT OF GRAIN STRAIN

The best method of determining the contribution made by grain boundary sliding to the overall extension is by the indirect approach of measuring the percentage extension β due to transgranular processes. To do this, it is necessary to obtain a measure of the average grain strain ε_s, and then subtract this from the total extension ε_t to give ε_{gb} by difference.

ε_s can be determined by putting a series of closely spaced parallel lines on to the specimen surface, perpendicular to the stress axis, and of a spacing slightly less than the average grain diameter. If the spacing of the lines is known accurately before testing, the specimens may be creep tested to some known total strain, and ε_s determined by measuring the line separation in those grains which are intersected by two or more lines. In Fig. 10, for example, the extension due to transgranular processes may be measured at points A, B, C, D, E, etc.

It is quite possible to carry out this work using scribed lines on the surface, although this method should only be utilized if facilities

are available for scribing such lines with an accurately known separation. The high degree of accuracy required for this work can be easily seen by considering a specimen having a grain size of about 50 grains/cm. At a total strain of the order of 2% it is necessary, in order to resolve any increase in line separation, to be able to measure to an accuracy of $\pm 1 \times 10^{-4}$ cm. A better, and less tedious, method is to print a grid of lines on to the surface by a photographic means. This technique, which we will now describe, is also applicable to other metals such as aluminum.

A graticule* is required for this purpose, either in the form of a glass photographic plate with a negative image of the grid printed on one side, or with the grid produced by the direct evaporation of metal on glass. A negative image is required to give a positive contact print on the test surface, and the plate should be of a convenient size so that it fits on to the test face of the specimen. A network of fine lines of the order of 80 per cm is convenient for use with specimens having a grain size of 25-50 grains/cm.

The method consists essentially of direct contact printing, using photographic materials obtainable from the Kodak Company. A preliminary requirement is that the photographic plate is absolutely clean, since any dirt or dust is reproduced in the image. It is therefore advantageous to thoroughly clean the plate in a trichloroethylene vapor

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* Obtainable from numerous optical manufacturers in the United States; refer to "The Optical Industry and Systems Directory" for current listing. In Great Britain, obtainable from Graticules Ltd., 18/20 Garrick Street, London W.C. 2 (products available in the United States through the Ealing Corporation, Cambridge, Massachusetts).

degreaser before each use. A photographic emulsion, Kodak Photo Resist (KPR), is then flowed onto the test surface, so that the whole surface is wetted, and the specimen is clamped horizontally, test surface uppermost, at the center of a whirler, and rotated for 10 minutes at room temperature at a speed of about 100 r.p.m. The KPR is dried by whirling for a further 10 minutes in forced warm air at 60°C. This produces a very thin and even coating of KPR, although, if a whirler is not available, satisfactory results can also be obtained by dip coating.

The master plate is then held rigidly in place on the test surface, and the surface exposed for a fixed time to a single source of ultraviolet light. The optimum exposure should be determined by a preliminary test, but, as a rough guide, an exposure time of about 10 minutes is required for a specimen situated 3 ft. from a 400 watts source. After exposure, the specimen is immersed in KPR Developer at room temperature for two minutes, using intermittent agitation to remove any unexposed emulsion. Finally, the grid is accentuated by immersing for 30 seconds in KPR Dye, and the specimen washed thoroughly in methyl alcohol and dried. To ensure adhesion of the film to the surface, it is advisable to bake the specimen for 30 minutes at 200°C, giving a final image of dark blue lines.

The printing of the grid must be undertaken before testing, and the specimen is then strained to about 3 or 4% in the manner described in section 4. The typical appearance of a printed grid after straining at 200°C is shown in Fig. 11; the offsets at the grain boundaries due to sliding are clearly visible.

Two sets of measurements are then taken. Firstly, the longitudinal offsets, u_t, are measured at points along transverse grid lines and the angle θ_t between the boundary and the stress axis is also recorded at each point of intersection. ε_{gb} is then calculated from Eq. (5) and, since ε_t is known, this directly yields a value for γ. Secondly, measurements are taken, parallel to the stress axis, of the separation between adjacent grid lines when both lines are contained within the same grain. As with the longitudinal offsets, this is done on the screen of a projection microscope, and, at a magnification of about 1000, it should be possible to measure each separation to an accuracy of $\pm 5 \times 10^{-5}$ cm. The method of measurement employed is to carry out a longitudinal traverse of the specimen along one of the grid lines, and to measure the separation between lines perpendicular to the stress axis at every instance where two adjacent lines are contained within the same grain. This is an extremely tedious process, and again a large number of readings are required if the error bars are to be kept to an acceptable level. It should be possible, however, to make at least 100 measurements. These results are then utilized to determine the average grain strain, ε_s, and hence the value of β. The percentage ratios of γ and β should approximate to 100%, within the very large error bars inherent in these measurements; this method therefore serves to directly indicate the reliability of determining γ solely from measurements of u.

Finally, the results are also used to determine a value of k for use with measurements of $\bar{v}_r$ (Eq. (6)). Readings are taken to determine $\bar{v}_r$, in the manner indicated previously, and this value is then substituted into Eq. (6), together with the value of γ determined indirectly from

the grid measurements of ϵ_s. The value of k obtained in this way, usually of the order of 1.0-1.5 at a total strain of 2-4%, may be used with Eq. (6) in subsequent tests to determine γ directly from measurements of $\bar{v}_r$.

6. FURTHER EXPERIMENTS

The experiments already described have referred specifically to metallic substances, and to magnesium in particular. However, grain boundary sliding may also be observed in polycrystalline ceramics, such as NaCl.

The method of producing a single crystal of NaCl has already been described in Chapter 1. To produce polycrystalline material, the single crystal is cut so that it fits tightly into the container of an extrusion press, and extruded at a temperature of about 350°C. Care must be taken to machine the single crystal so that it exactly fits the die chamber, otherwise the polycrystalline sample will probably contain some slight porosity at the grain boundaries. A typical reduction ratio during extrusion is 16:1, and a typical dimension for the polycrystalline material is of the order of 0.25" diameter. An even grain size is attained by annealing the material for 2 hours at a temperature of 400-450°C. Suitable specimens, of about 0.35" length, are cut to size using a wet string, and polished by rolling backwards and forwards in a tray of water. To observe grain boundary sliding, the specimen is tested in compression at ~400°C, using a load such that measurable offsets occur after about a week. Following the test, measurements may be taken to determine $\bar{v}_r$, or, if transverse lines have been scribed on the surface prior to testing, γ can be determined from measurements of u_t and θ_t.

It was suggested in section 2 that an as-cut, or polished, surface should be used for the tests on magnesium, rather than the surface that exists immediately after the heat treatment. The reason for this is that the internal boundaries beneath an as-cut surface make random intersections with the plane of the surface, whereas during the annealing process the boundaries preferentially migrate to positions of minimum energy configuration almost perpendicular to the surface. A simple test can be performed to show the difference between an as-cut and an annealed surface configuration, by annealing two identical specimens at the same temperature for the same length of time. Following the anneal, one specimen is polished by removal of three or four grain diameters, but the other specimen is retained in the annealed condition. Both specimens are then sectioned transversely, mounted in cold-setting resin, and the cross-sectional faces polished and etched. Measurements are taken of the internal angles between the boundaries and the surface, either by projection, or with a goniometer microscope eyepiece calibrated in degrees. The results will show that, whereas the average angle of intersection of the boundaries with an as-cut surface is about 57°, the average angle with an annealed surface is nearer to 90°, and probably of the order of 75-80°. The most realistic way of presenting this data is by drawing histograms to show the number of boundaries recorded in each 5° increment of angle. Usually, no boundaries are recorded on the annealed surface intersecting the surface at angles less than 30°.

It should be noted that the observed distribution of angles along a given section does not, in fact, represent the true distribution of angles with the surface. This can be seen by reference to Fig. 12, where

A, D and E are in the plane of the surface, and ABCD represents a grain boundary inclined at a true angle ϕ. If BEF is the random plane of sectioning, then the measured or observed angle β is less than ϕ. It is clear from the figure that a boundary making a true angle of ϕ with the surface may appear on sectioning at any angle β where $\phi \geq \beta \geq 0°$.

7. CONCLUSIONS

In this chapter, we have studied the behavior of materials when deformed slowly at high temperatures. An attempt has been made to distinguish between the deformation due to transgranular processes and that due to sliding at the grain boundaries. A fairly accurate estimate of this latter contribution is often very desirable, since sliding may play a very important role in the nucleation of grain boundary cavities and thus in the ultimate failure of the material.

Experimental evidence is available to show that, in metals subjected to creep at temperatures greater than about $0.4\ T_m$, the sliding contribution increases with a decrease in stress and/or grain strain. Under favorable conditions, results show that sliding may account for over 70% of the total deformation during the early stages of creep. A reliable method of determining γ in such cases is thus of prime importance.

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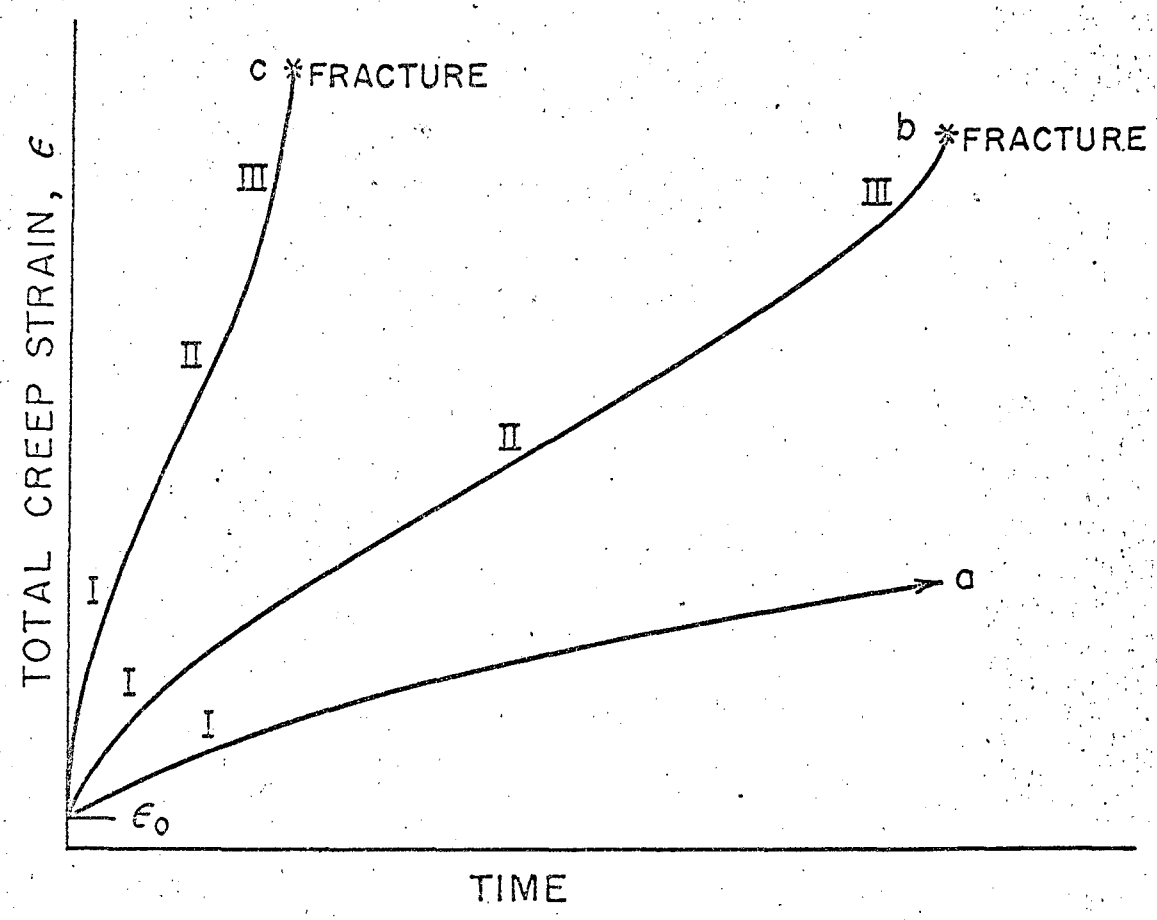

FIG. 1 Typical creep curves obtained at different temperatures and stresses; the three stages of creep are indicated.

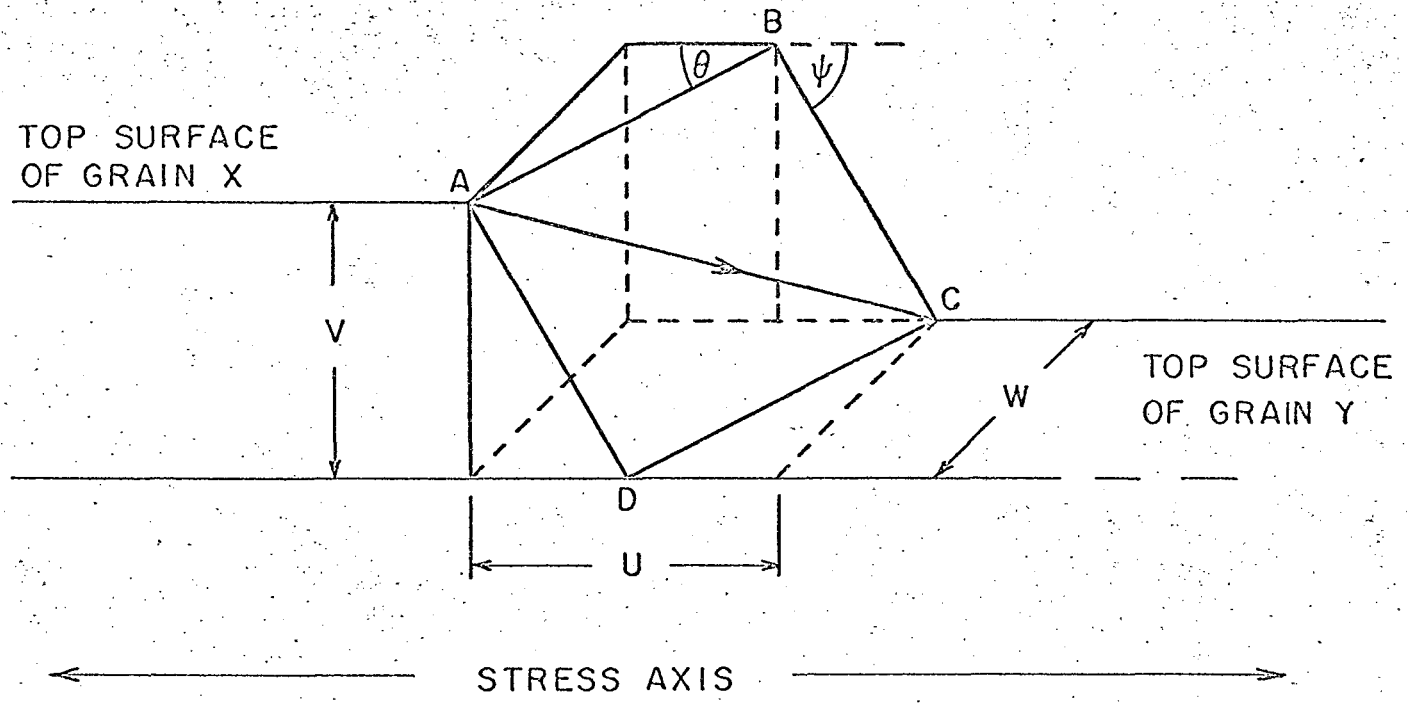

FIG. 2 Schematic representation of grain boundary sliding between grains X and Y. ABCD is the grain boundary offset and AC is the sliding vector.

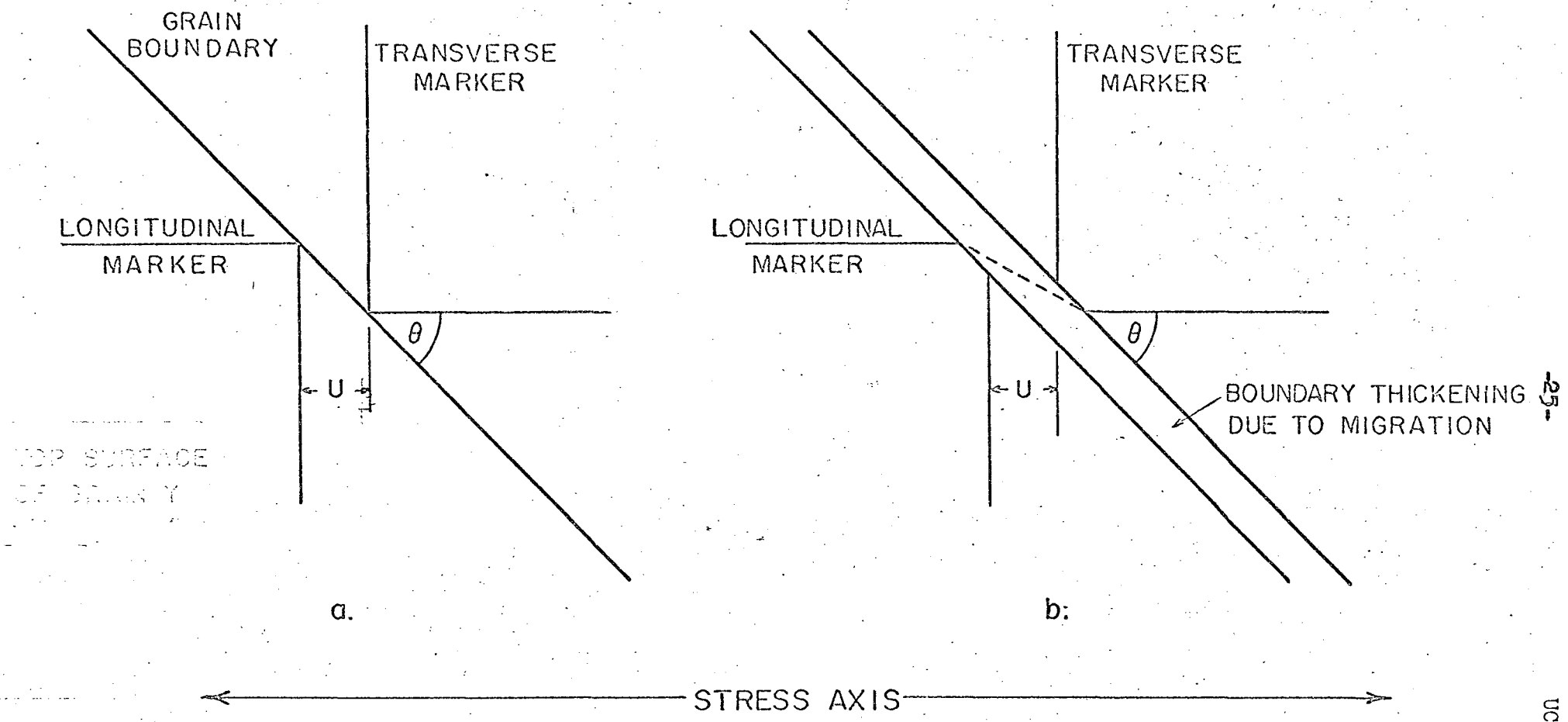

FIG. 3 Schematic diagram showing (a) the ease of measuring u from longitudinal or transverse markers in the absence of migration, and (b) the difficulty of measuring u from a longitudinal marker if migration occurs.

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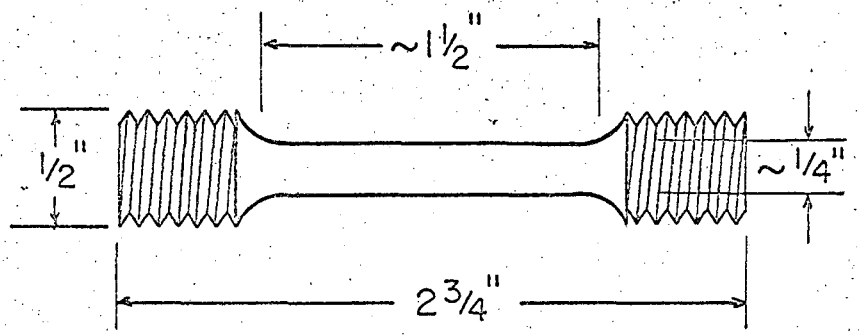
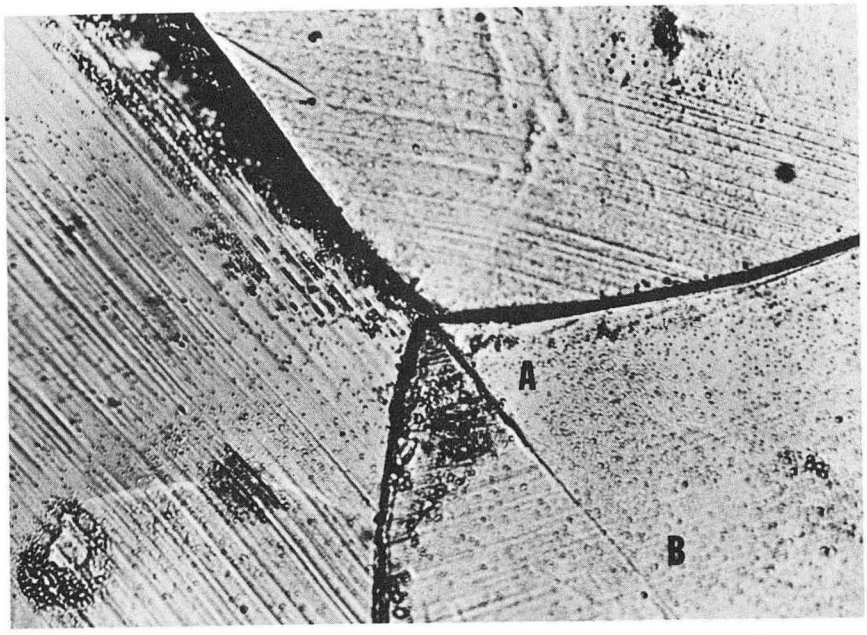
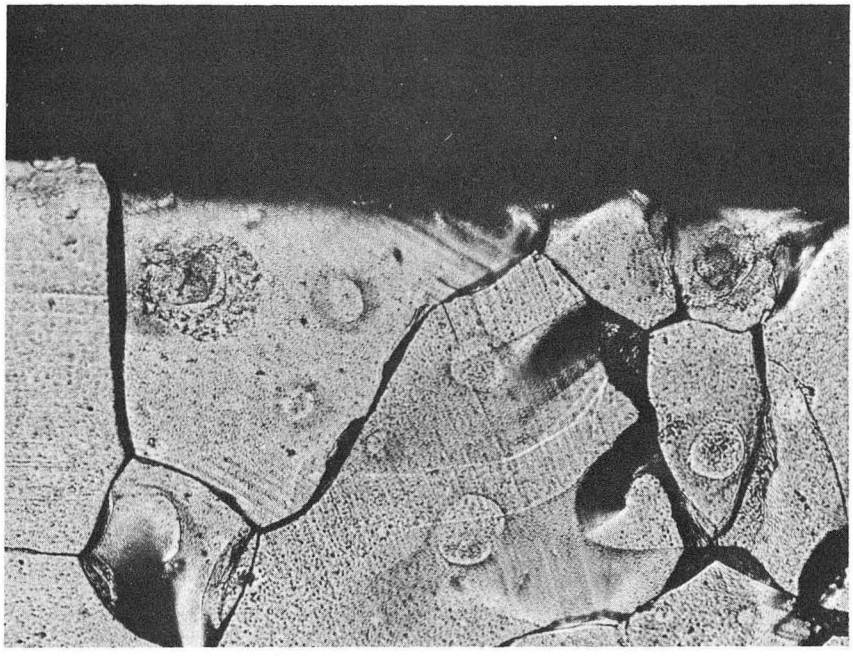


FIG. 4 Typical dimensions for a specimen produced from 0.5" diameter rod. Surface observations are made on two parallel test faces, approximately 0.25" apart.



XBB 678-4474

Fig. 5 Triple point fold AB in a magnesium alloy strained 5.2% at 200°C (×640)



XBB 673-1381

Fig. 6 Surface steps, due to grain boundary sliding, revealed by having two mutually perpendicular surfaces. Magnesium alloy strained 4.2% at 250°C; stress axis horizontal (×190).

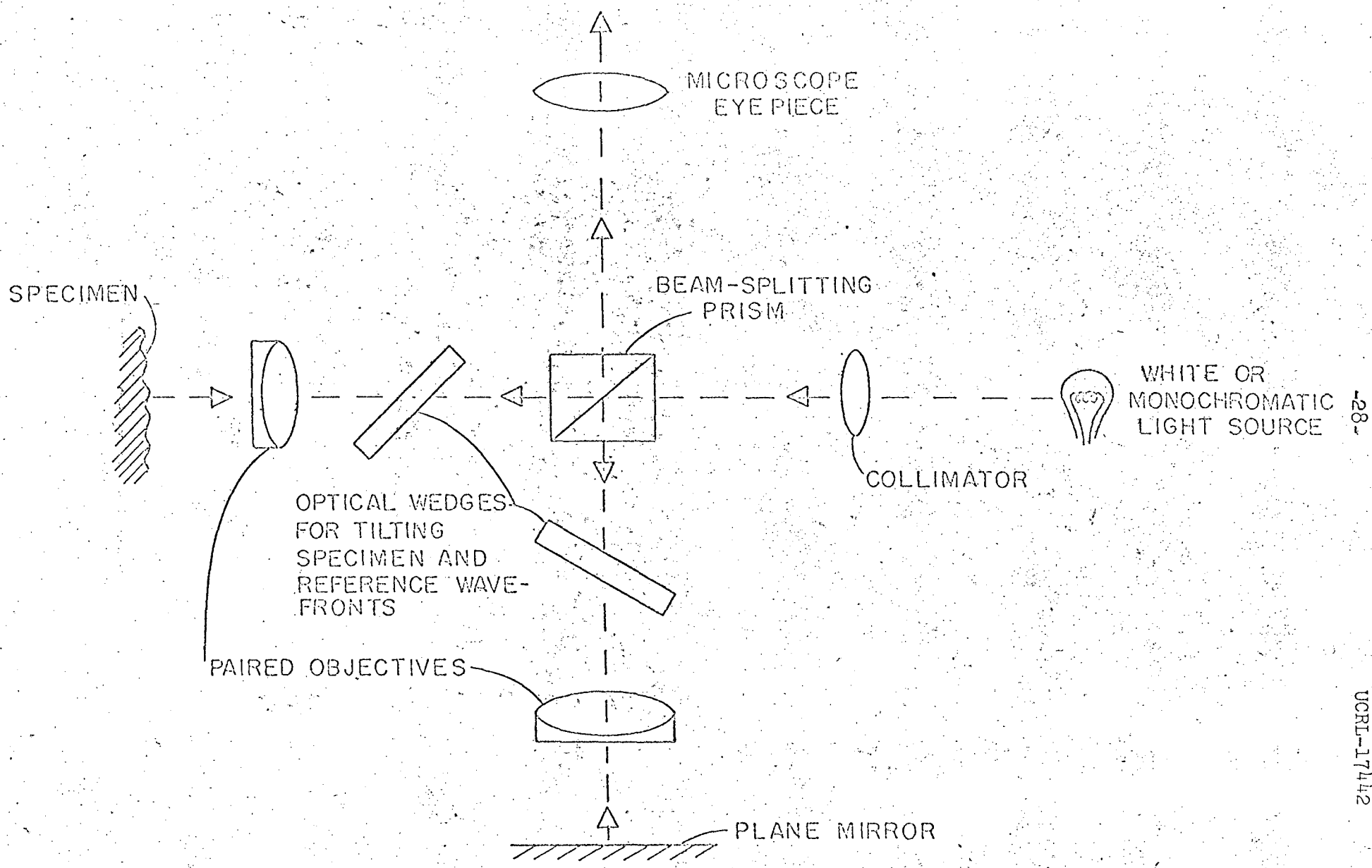
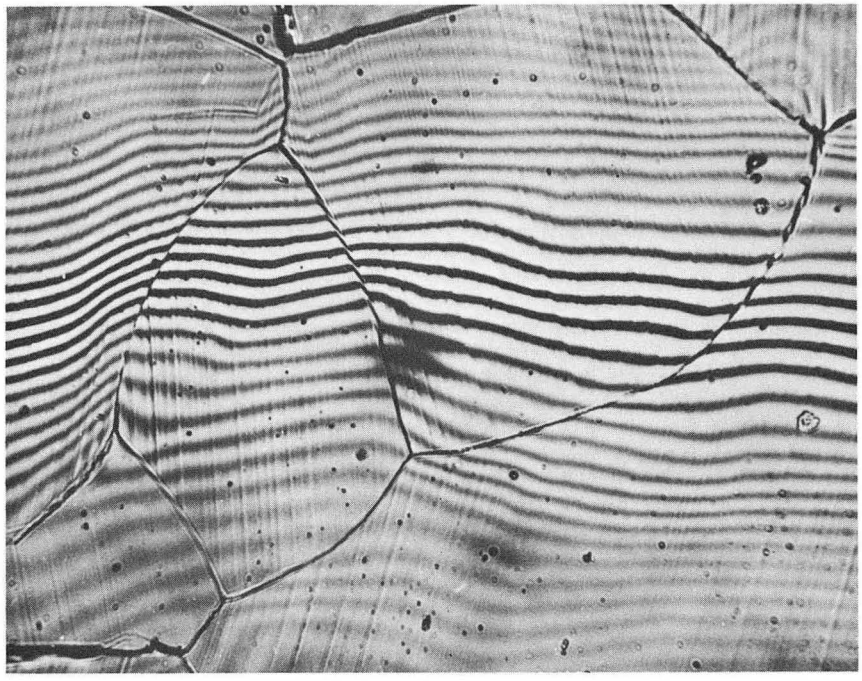
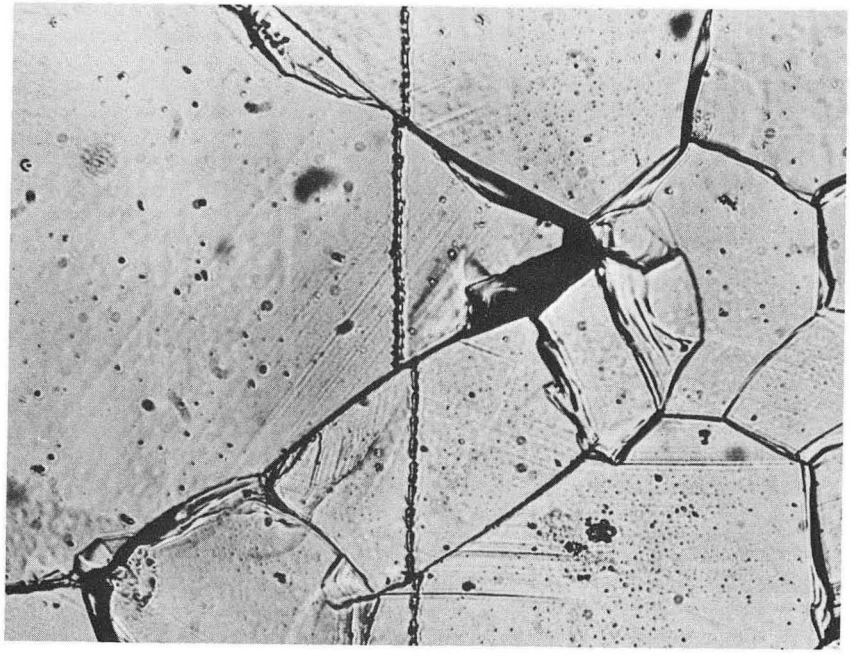


FIG. 7 Schematic outline of an interference microscope



XBB 673-1378

Fig. 8 Grain boundary offsets revealed by interference fringes. Magnesium alloy strained 1.5% at 200°C. (×430)



XBB 673-1377

Fig. 9 Offsets revealed by a transverse marker line on a magnesium alloy strained 2.5% at 200°C. Stress axis horizontal (×410).

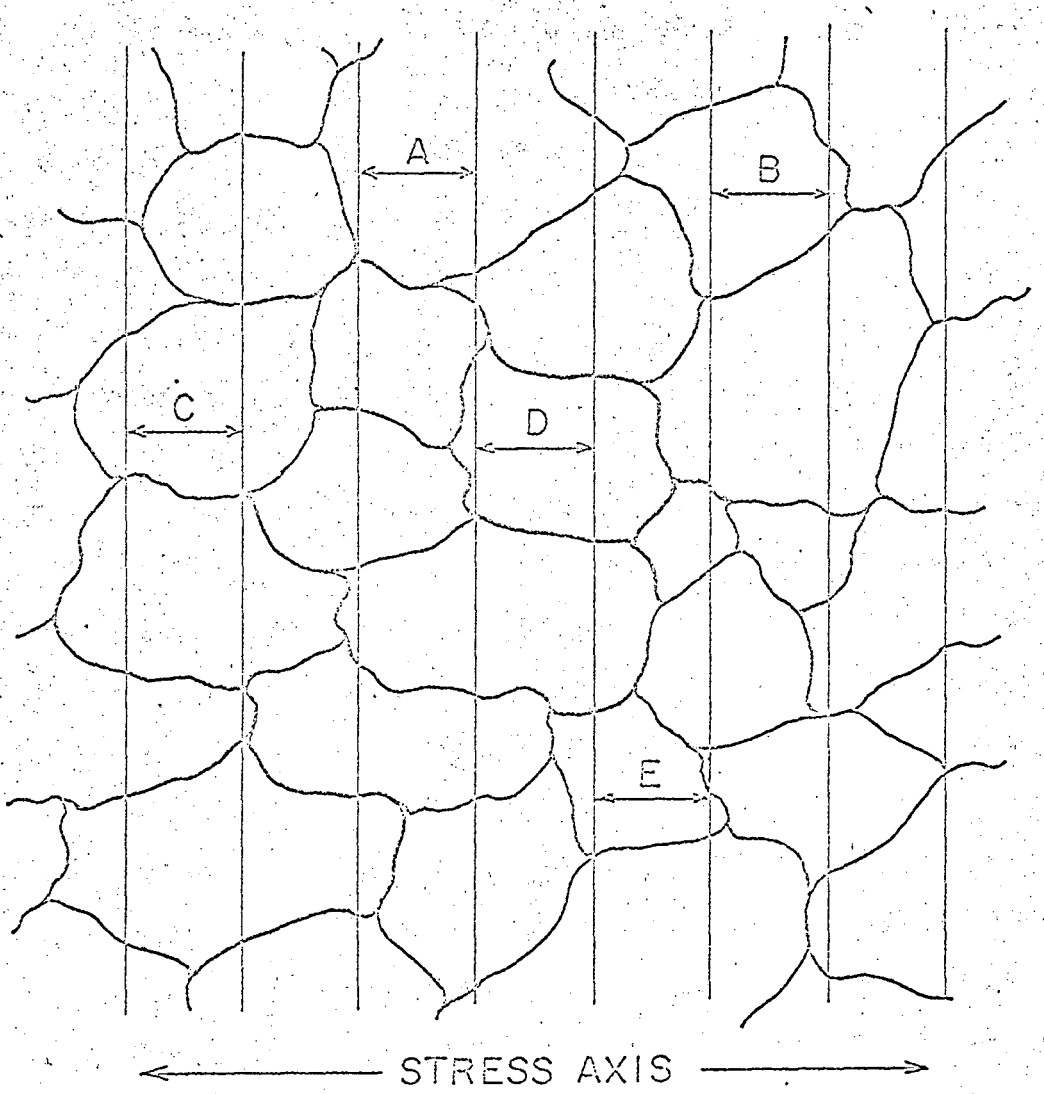
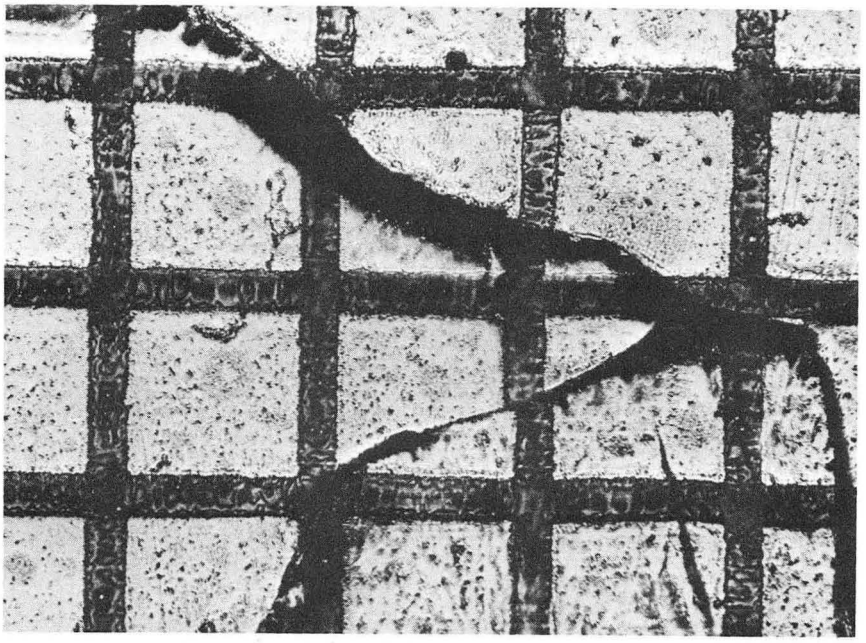
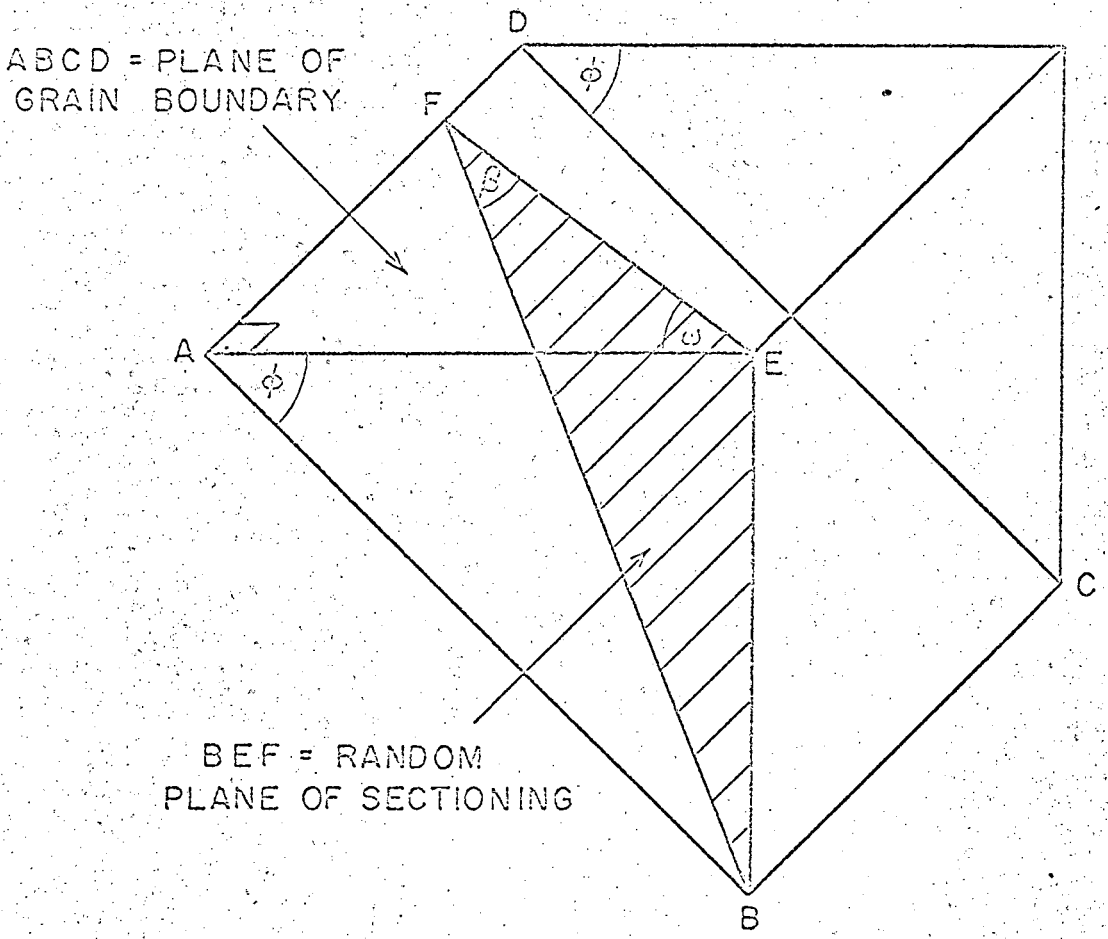


FIG. 10 Schematic representation of a surface prepared for measuring grain strain; ε_s is determined from the separation between the lines at points A, B, C, D, E, etc.



XBB 673-1379

Fig. 11 Offsets revealed by a grid printed on the surface of a magnesium alloy; specimen strained 1.1% at 200°C. Stress axis horizontal (×200).



$$\tan \beta = \tan \phi \cos \omega$$

FIG. 12 Relationship between the true (ϕ) and observed (β) angles at a surface