SOME FACTORS AFFECTING THE CLEAVAGE OF METALS

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A STUDY OF SOME FACTORS AFFECTING THE CLEAVAGE OF METALS

by

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PART IV: SOME EXPERIMENTS ON THE CLEAVAGE FRACTURE OF PURE POLYCRYSTALLINE ZINC IN SIMPLE TENSION

SYNOPSIS

Experiments have been made with the object of elucidating the mechanism of the cleavage of metals.

Since cleavage was known to occur in certain body centred cubic metals a series of notched impact bend and tensile tests have been carried out on β-brass and two Al β-brasses down to $-196^{\circ}C$ to determine whether a similar cleavage occurred. The alloys showed no indication of cleavage failure.

A study has been made of some of the cleavage properties of pure polycrystalline zinc in simple tension and the effects of grain size, temperature, strain rate and plastic deformation on the true fracture stress have been investigated. The results obtained have been used to examine the various theories proposed to explain cleavage phenomena and it is concluded that dislocation theory can be developed to account for the build up of large internal stresses from which cracks leading to cleavage can be formed.

A similar investigation has been carried out on pure polycrystalline magnesium. The results are markedly different from those for zinc. The theory requires to be developed in much greater detail to account for the diversity of phenomena between the two metals.

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GENERAL INTRODUCTION

It has long been known that metals can fracture in a variety of ways. Not only do different metals fracture by apparently different mechanisms, but a given metal can often fracture in more than one way, depending on the conditions of service or test. However, types of fracture can generally be divided into two classes, brittle and ductile, though intermediate cases can occur. It can be said that brittle fracture occurs suddenly and after little, or no, plastic deformation. Ductile fracture occurs relatively slowly and after a considerable amount of plastic deformation.

Pure metals usually fail in a ductile fashion at high temperatures and some, in particular the face-centred cubic class, retain their ductility to very low temperatures. On the other hand, for several metals, brittle fracture occurs, particularly at low temperatures. It is either intergranular, that is, it occurs along the grain boundaries, or transcrystalline, occurring along a definite crystallographic cleavage plane.

The purpose and scope of the present work was to investigate some of the factors influencing the occurrence of cleavage fracture and to attempt to elucidate the mechanism.

The desirability of understanding the mechanism of cleavage arises for two reasons. First, a point of immediate and great practical importance, is to obtain a means of preventing the cleavage failures of metals and particularly of steels which have on several occasions been used in service with disastrous results. Secondly, since cleavage has been found in extremely pure metals, it might be expected that cleavage studies would yield valuable results concerning the properties and more particularly the structural imperfections present in metals.

Several recommendations which have recently been given should eliminate, or at least considerably reduce, the failure of steels under service conditions, particularly if adequate attention is paid to good design and the avoidance of sharp notches and other stress concentrators. Recent research has also provided much data on the effects of alloying elements.

However, it is still necessary to search for more fundamental results and as far as possible to attempt to formulate a theoretical basis so that some guidance may be obtained for future work. A review of the present state of theoretical knowledge has been given in Chapter II.

A point of some significance is that cleavage has been found to occur in α-iron of high purity. Cleavage has also been observed in molybdenum and in tungsten, which have the same body-centred cubic structure and similar plastic properties to α-iron. The cleavage plane (100) is the same in all three metals and this would indicate a property of the lattice or of the form of deformation, or both.

In line with this, in the present work an attempt was made to induce cleavage in β-brass and in two Al β-brasses of high purity. Though β-brass is an alloy it is of superlattice form and it possesses similar structure and plastic properties to α-iron, tungsten and molybdenum. The experimental work and results are described in Chapter III.

Several other metals have been reported to show cleavage. The cleavage planes have been identified in bismuth, zinc, magnesium, antimony and tellurium, and a list has been given by Schmid and Boas[1]. Nevertheless, the fundamental problem still remains one of accounting for the low cleavage fracture stresses obtained for all metals which show cleavage and which are of the order of 100 to 1,000 times weaker than the cleavage stress values given by the classical cohesion theory for crystals, which has been recently reviewed by Orowan[2]. In addition, little is known of the effects of alloying elements or of geometrical size and shape (except for steels). Likewise little is known of the effects of grain size, temperature, strain and strain rate etc. on the properties of cleavage.

In the present series of investigations a systematic study was made of the effects of several variables on the cleavage properties of polycrystalline zinc of high purity. A number of new and interesting results have been obtained. These are presented and discussed in Chapter IV.

Work was also carried out on the cleavage of pure polycrystalline magnesium since this metal was known to have a similar (hexagonal close packed) structure to zinc. The study of the cleavage fracture showed results markedly different from those obtained for zinc. The results for magnesium are given in Chapter V.

Finally, Chapter VI contains some collected results which have been obtained from the present work.

I - PREVIOUS STUDIES OF CLEAVAGE

(1) HISTORICAL INTRODUCTION

The fact that solids can often be broken easily along definite crystallographic planes has long been known. This phenomenon is known as cleavage. The first observations were on minerals and the fractures obtained were frequently used as a means of identifying and classifying crystals. The absence of quantitative measurements of "cleavability" were no doubt largely due to the complex stress systems which arose in the usual methods of cleaving.

With microscopic and goniometric studies, however, observations were made on the properties of freshly cleaved surfaces. In all cases they were found to be along planes of low index and high atomic density. The surfaces were smooth, flat and of high reflectivity though often not without deformation markings. In addition it was observed that little energy was needed to cleave a crystal.

The first systematic investigations into the influence of crystal orientation and the direction of applied stress was made by Sohncke[3] in 1869 on sodium chloride. For all orientations he examined, the cube plane (100) appeared as the fracture plane and the results indicated a "critical normal stress" law for cleavage. That is, the applied stress $\sigma = \frac{N}{\sin^2 \chi}$ where N was the constant critical normal stress and χ the angle between the cleavage plane and stress direction. However, this law was not accepted as a general criterion in later work[4]. More recent experiments have shown the law to hold for certain cases, though it is certainly not applicable to all cases of cleavage. This remains a point of considerable interest.

The early experiments reported by Smekal[5] showed conclusively that constancy of the elastic energy required for fracturing was not valid as a criterion of cleavage.

The tensile strength values for the (100) plane of rock salt were obtained between 200 and 600 gm/mm^2 and the elastic extension perpendicular to the cleavage plane prior to fracture was of the order of 10^{-4} cms. It was shown by Ewald and Polanyi[6] that, in the case of "perfectly brittle" fractures, the modulus of elasticity remained very nearly constant right up to the point of fracture. This indicated that there was no gradual weakening of the cohesive forces of the crystal prior to fracture. Using very small crystals of less than 1 m.m. diameter, Jenckel[7] observed significant increases in tensile strength with decreasing crystal size. This was interpreted by assuming that smaller crystals were likely to have fewer of the defects which might be responsible for cleavage. If additions of other substances were made, Schonfeld[8] found that the tensile strength was increased in much the same way as the yield point. The work gave some indication that a connection might exist between the yield stress and the cleavage stress.

Investigations have been reported on the temperature dependence of cleavage. Joffé[9] and others found that the tensile strength of rock salt crystals was approximately constant over a wide range of temperature. However, at high temperatures, or when the stress was applied sufficiently slowly for a marked plastic deformation to occur prior to fracture, then tensile strengthening was found to occur. From photographic studies the time to fracture has been estimated to be of the order of 10^{-3} to 10^{-4} secs.[10]

Microscopic examinations of metals which had shown brittle fractures revealed in many cases that the individual crystals of the metal had fractured in the manner which was well established for the cleavage of other materials. It was apparent that cleavage could occur in certain polycrystalline metals and spread throughout by the cleavage mechanism. This type of fracture was found to be the cause of a number of failures in steel structures. Hence the problems of the occurrence and mechanism of cleavage fracture became of great practical importance in addition to their intrinsic scientific interest. Thus there arose a necessity to devise tests which would show the susceptibility of metals to cleavage and also indicate whether they could be safely used in service.

(2) TYPES OF FRACTURE IN METALS AND TESTS TO DETERMINE THE SUSCEPTIBILITY TO CLEAVAGE.

It was found that steels showing a very high ductility in normal tensile tests were often liable to show brittle fracture in service. The problem then arose of designing a suitable test whereby a measure of the brittleness could be determined. It was observed that impact loading and low temperatures were conditions under which brittleness was likely to be revealed. Severe winters were known to have caused numerous brittle fractures of railway stock and rails etc. andthe most spectacular epidemic of brittle fractures was found in welded ships during the 1939-1945 war.

The easiest means of estimating brittleness in steels was found to be by a bending test on notched bars - hence the term "notch-brittleness". A rather more refined test was devised by S.B. Russell[11] in 1897, in which a notched bar specimen was broken by the impact of a pendulum hammer, the energy loss of which measured the work of fracture. This form of test has been further developed and most machines in present use are of the "Charpy" or "Izod" type.

A very general theory of "notch brittleness" has been developed and this is discussed adequately in a review by Orowan[2]. From hypotheses due to Ludwik[12], brittle fracture was considered to occur if the "brittle strength B" was less than the yield stress Y. For an increase in temperature, Y was thought to decrease at a faster rate than the brittle strength B, so that at some temperature, Y would be less than B and then the metal would be ductile and give a fibrous fracture. The temperature, representing the change in the type of fracture in a given test, has been termed the "Transition Temperature".

This theory was not only relevant to the cases of transition between cleavage and ductile fracture. It was also applicable to the transition between any other forms of brittle fracture and ductile fracture. A list of fracture types has been given by Orowan[2]. The theory, then, was quite general in assuming a brittle strength value B without specifying the cause of brittleness. Because of this vagueness in the theory, it can tell nothing of the cleavage mechanism, but it will be presented here in so far as it helps in the understanding of tests to determine the susceptibility to brittleness.

Ludwik first drew attention to what is at present regarded as the main direct cause of notch brittle behaviour, namely the triaxial state of stress arising on plastic deformation of specimens containing notches. This state of stress causes an effective increase in the yield stress. An analysis[13] has shown that the effective yield stress cannot be increased to a value greater than $3Y$, where Y is the "unnotched yield stress".

Without explaining the details involved, the hypotheses described are useful in so far as they give a picture of the notch effect and it can be said that:-

If $B < Y$ the material is brittle.

If $Y < B < 3Y$ the material is "notch brittle".

If $B > 3Y$ the material is fully ductile even when notched.

These conditions are presented graphically in Fig.1, taken from Orowan's review. The graph, however, can only be regarded as presenting an outline of the phenomena and it will be seen later that the problems of flow and fracture are much more closely connected than would appear from Fig.1. Nevertheless, this representation indicates that a "transition temperature" should exist below which the metal is brittle and breaks with little energy absorption and above which it is ductile and requires much energy in breaking with a ductile fracture. A typical graph of the energy required for fracturing against the temperature is shown in Fig. 2. The above hypotheses also indicate that the transition temperature should be dependent on the type of test employed for a given metal, and this is one of the most important features found experimentally. They tell us nothing of the mechanism of the fractures nor do they give any clues to the effects of many variables.

The Charpy and Izod types of test carried out over a range of temperature on both sides of the transition temperature give a good indication of the brittle tendency. If the fracture is by cleavage, bright, flat facets are seen over the fracture surface.

Above the transition temperature the fracture surface is dull, rough, and does not reveal any crystallographic characteristics.

Whilst the Charpy, Izod and similar tests are of great practical importance, the complex stresses set up during the test make it impossible to obtain precise quantitative measurements in terms of the fundamental properties of the material. The only alternative, however, in the case of investigations on steels is to carry out tensile tests at extremely low temperatures which involves costly and difficult technique. The first such investigation was carried out by Hadfield(14). More recently Eldin and Collins have reported an investigation on a steel down to $12^{\circ}K$(15). The steel was found to be completely brittle over the range 12 to $61.5^{\circ}K$ and fractured without any reduction in area over that range. They found that the brittle strength increased with decreasing temperature. Such procedure, though, is unnecessary in determining empirically the effects of metallurgical structure on steels.

(3) METALLURGICAL FACTORS AFFECTING THE CLEAVAGE OF STEELS.

An immense amount of recent research has been devoted to the problems of cleavage in steels and many useful summaries have been given(16)(17)(18)(19)(20). Several valuable studies have been made concerning the effects of quantities of added elements together with the best heat treatments for toughness.

Investigations on mild steel by Barr and Tipper(21) indicated that steels with higher manganese contents had lower transition temperatures. Further work by Barr and Honeyman(22) confirmed the beneficial effects of manganese and the practical recommendation was made that for structural steels for shipbuilding, the ratio of manganese to carbon should not be less than three.

In numerous researches austenitic steels have been found to retain their toughness down to the lowest temperatures of test. It is to be noted that these belong to the face-centred cubic class of metals, which have, up to the present, invariably shown good impact properties.

Studies of the effects of oxygen, silicon and phosphorus in steels at the National Physical Laboratory[23] have shown that these elements raise the transition temperature and give fractures which are part intergranular and part cleavage.

A number of researches have shown that grain size has an influence on the fracture properties of steels. Larger grain sizes have generally been found to raise the transition temperature. Alloying elements such as vanadium which reduce the grain size lead to better impact properties and give lower transition temperatures[24].

The effect of 13 alloying elements on the brittleness of pearlitic steels has been investigated by Rinebolt and Harris[25]. The transition temperature was lowered by manganese and nickel and raised by carbon, copper, molybdenum, phosphorus and silicon. The effects of the other elements were complex. With increase in carbon content the transition temperature was less sharp, the change from brittle to ductile fracture taking place over a range of temperature.

Hopkin and Pickman[26] have studied the effect of hydrogen on steels. If the hydrogen was not given time to diffuse during testing its effect on plain carbon steels was low, but hydrogen reduced the brittle strength of low alloy hardened Cr-Mo., Mn-Mo. and Ni-Cr-Mo. steels. An extensive review[27] has been published on the effects of hydrogen in steels. Several cases are cited where defects can be eliminated by keeping the hydrogen content to a minimum but in many cases there is no proof that hydrogen is the only cause.

The results obtained, concerning the effects of metallurgical structure on cleavage, have enabled several recommendations to be made which should eliminate, or at least considerably reduce, the failure of steels under service conditions particularly if serious attention is paid to good design. However, in spite of their great practical importance, the results have not received any theoretical interpretation. The phenomena would appear to be of great complexity and for an attempt to understand the fundamental aspects of the mechanism it would seem preferable to work with very pure materials.

(4) CLEAVAGE IN NON-FERROUS METALS

Closely analagous with the cleavage fractures in pure α-iron are the cleavages which have been observed in tungsten[28] and in molybdenum[29][30]. These metals all have body centred cubic structures and Andrade has shown them to have similar plastic properties[31] if the effects of temperature are taken into account. In all three metals the (100) plane has been identified as the cleavage plane. This gives some indication that crystallographically related metals might be expected to show similar cleavage properties. The relatively little work done on the fracture properties of tungsten and molybdenum has no doubt been due to the relative unavailability of the pure material in bulk form.

β-brass, which is again of the same crystalline form and has similar plastic properties to α-iron, tungsten and molybdenum, might be expected to show similar cleavage. There is also the advantage that this material can be obtained very readily in a pure form. In the present series of experimental investigations a study was made of the fracture of β-brass and of two Al β-brasses.

The non-ferrous metals which have previously been used most frequently for studies of cleavage have been bismuth and zinc. The structure of these metals (rhombohedral and hexagonal close-packed respectively) being considerably different from that of α-iron, they could not be expected to provide a very close correlation with it, nor to have immediate bearing on the practical aspects of cleavage failure and its remedy. Nevertheless the study of the fractures of bismuth and zinc has contributed considerably in the accumulation of fundamental experimental data on cleavage.

In bismuth and zinc work with single crystals has proved possible and many such studies have been reported. The "Critical Normal Stress Law" has been considered to be valid for both metals[32][33] but recent work by some of the author's colleagues has shown that it is not valid for zinc[34]. Early work on zinc was also considered to show a "second best cleavage plane" in the prismatic $(10\bar{1}0)$ plane as well as on the more usual basal (0001) cleavage plane, but now it appears that what was regarded as prismatic cleavage is actually cleavage in the basal planes in twins[35] which have almost the same orientation as the prismatic plane in the matrix.

Some results have been reported for the effect of temperature on fracture stress. For bismuth there was found to be no detectable change between $-80^{\circ}C$ and $20^{\circ}C$. For zinc, Fahrenhorst and

Schmid[36] found no change in fracture stress between $-253^{\circ}C$ and $-80^{\circ}C$ though the scatter of results was large. However, it appeared probable that deformation increased the fracture stress at $-80^{\circ}C$, had no effect at $-196^{\circ}C$ and decreased the fracture stress at $-253^{\circ}C$. From several experiments on zinc it was concluded that a limiting shear stress could not be a criterion of failure. Schmid[37] found that small amounts of cadmium increased the fracture stress of zinc.

A survey of the literature indicated a rather surprising lack of fundamental data on the cleavage of pure polycrystalline zinc. The present work contains an account of experiments to determine some of the cleavage properties of this material.

In addition to the metals already mentioned, according to Schmid and Boas[1] cleavage planes have been observed in magnesium, arsenic, antimony and tellurium, though no details have been given for the first three. For tellurium Schmid and Wasserman[38] found that the critical normal stress law was valid over a range of orientations, but outside the range there were marked deviations. In their experiments, single crystals of tellurium fractured on the $(10\bar{1}0)$ prismatic planes, and no overall deformation tookplace prior to fracture.

To explain the phenomena associated with cleavage several theories have been proposed. These will be discussed in the next Chapter.

II - A BRIEF REVIEW OF THEORIES PROPOSED TO EXPLAIN CLEAVAGE

(1) THE FUNDAMENTAL PROBLEM. THE THEORETICAL TENSILE STRENGTH OF A FLAWLESS SOLID.

An estimate of the theoretical tensile strength of a solid can be obtained from considerations of the surface energy, as has been shown by Polanyi[39] and Orowan[40]. The energy required for fracture should be equal to the energy needed to separate two adjacent atomic planes.

If a solid is extended uniformly, the excess of the attractive forces over the repulsive forces acting across unit area perpendicular to the tension, is indicated in Fig. 3; the resultant force σ should vary with the distance x between neighbouring atomic planes as shown. The extension should be stable until the maximum of the curve is reached, then fracture should occur.

If α is the surface energy
E Young's Modulus
a the interatomic distance
σ_m the fracture stress

then the elastic energy between neighbouring planes in the stressed specimen must provide the energy for their separation and thus create two new surfaces at fracture. The elastic energy in the specimen is $\frac{\sigma_m^2}{2E}$ per unit volume, and the energy per unit area of two neighbouring planes is $\frac{a\sigma_m^2}{2E}$. Now the value of σ_m is reached when the energy between the two planes is $\sim \frac{\alpha}{2}$, and since two new surfaces are created,

$$\frac{a\sigma_m^2}{2E} \text{ is of the order of } 2\left(\frac{\alpha}{2}\right)$$

Hence the order of magnitude of the fracture stress σ_m to be expected is given by:

$$\sigma_m = \sqrt{\frac{2\alpha E}{a}}$$

For many metals it is generally found that:

$$E \sim 10^{11} \text{ to } 10^{12} \text{ dynes/cm}^2$$

$$a \sim 3 \times 10^{-8} \text{ cms.}$$

$$\alpha \sim 10^3 \text{ ergs/cm}^2$$

The value of α can be found approximately from the extrapolation of surface tension measurements made on the liquid metal. However, recent theory and experiments have provided methods for determining α for metals in the solid state, though up to the present measurements have only been made on copper. Uddin, Schaler and Wulff found that $\alpha = 1370$ ergs/cm^2 for copper just below its melting point[41]. Between 800 and 900°C, Bailey and Watkins[42] obtained the value of 1800 ergs/cm^2. These results are in reasonable agreement with the theory given by Huang and Wyllie[43] which predicts a value of about 1820 ergs/cm^2 for solid copper. For the liquid metal $\alpha = 1100$ ergs/cm^2 at 1100°C. The results indicate that for other metals, for which α has not yet been measured for the solid state, the extrapolations from surface tension measurements on the liquid are unlikely to be much in error.

Using the above equation to calculate σ_m, it is found to be of the order of 3×10^{11} dynes/cm^2. This value is of the order of 2,000 tons/sq.in.

The value theoretically obtained for σ_m is found to be 100 to 1,000 times greater than the highest cleavage stresses which have been experimentally measured. This represents the fundamental problem of cleavage fracture. Further it is found that cleavage occurs at very small extensions. The theory presented would require fracture to occur after about 30% elastic deformation, whereas plastic flow occurs at low extensions, usually much less than 1%.

Two quite different interpretations have been suggested to account for the great discrepancy between the experimental values of cleavage stress and the values obtained from cohesion theory. One approach has been to use the general methods of thermodynamics, with certain added assumptions, for stress calculations. The other approach, which has been much more completely developed, has been to assume that flaws, or other forms of inhomogeniety giving rise to stress concentrations, are present which provide the necessary high internal stress so that σ_m is in fact reached locally.

These two different lines of approach and the theories resulting from them will now be discussed.

(2) THERMODYNAMIC THEORIES OF TENSILE STRENGTH

From thermodynamic considerations it has been suggested that a relation exists between the energy required for fracture and the energy required for melting. Fürth[44] has proposed a theory based on the theory of melting developed by Born[45], by regarding the process of fracture as a kind of melting imposed by external forces. The form of melting is visualised to take place at relatively low stresses and this is the reason why the theoretical cleavage stress σ_m is not attained. By this theory the tensile strength is found to be of the order of magnitude which is found by experiment and the theory does not need to assume any details of the fracture mechanism.

To simplify the treatment of the problem as far as possible Furth considered an ideal homogeneous and isotropic material, completely elastic and with no plasticity. If an applied stress is increased gradually then it is assumed that a stage is reached where cracks or holes are formed inside the body and cause fracture immediately.[46]

If σ is the tensile strength

Q the energy of melting unit mass

ρ the density of the material

then the approximate condition for fracture is $\sigma \sim Q\rho$ When a more detailed thermodynamical treatment is applied which considers more completely the energy changes in the system, then the fracture criterion is found to be given by the equation

$$\sigma = Q\rho \frac{1-2\nu}{3-5\nu}$$

where ν is Poisson's ratio for the material.

Furth discusses the experimental conditions under which the formula could best be tested and concludes that the nearest approaches to the idealised material envisaged in the theory are pure unworked polycrystalline metals tested at low temperatures. However, the results he quotes for comparison with the theory have unfortunately been taken from ultimate tensile stress measurements for metals showing high ductility and not from true fracture stress measurements, which are the quantities which would be required for a valid test of the theory. In the relatively few cases in which they can be and have been measured, values of the true fracture stress of pure polycrystalline metals have been shown to be strongly dependent on factors such as size and geometrical shape of the specimens tested and on grain size etc. The formula thus includes too little detail to be tested directly by experiment.

A closely analogous thermodynamic theory of fracture which agrees in essential details with the Furth theory has been developed by Saibel[46]. He has attempted to cover the cases where some flow precedes fracture by including some of the properties of the stress-strain curves. Nevertheless, the formulae derived do not take into account the chief factors influencing true fracture stress and so it is difficult for valid comparison with experiment to be made. It appears that the formulae can have little, if any, practical utility.

These theories, whilst possibly suggesting reasons for low fracture stresses, do not contribute further to the understanding of fracture. The chief criticism against the theories is that they do not take into account the observations from a great many experiments that fracture is essentially a structure sensitive phenomenon. Seitz and Read[47] have further advanced the arguments against thermodynamic theories in the form in which they exist up to the present time in that they assume a uniform strain energy density which is not likely to be present throughout the material and that they do not indicate how elastic energy can be converted into heat.

It would appear that the thermodynamic theories must include much more detail if they are to be of any use in understanding fracture. Some of the present results will be discussed later in the light of these theories.

(3) THE GRIFFITH THEORY OF FRACTURE

In marked contrast to the more recent thermodynamic theories, a very successful theory was proposed by Griffith[48] in 1920 to explain the low fracture stresses observed in glasses. He assumed the presence of small cracks or other flaws around which strong stress concentrations arose when the material was stressed. According to this theory, the theoretical cohesive strength σ_m is the true microscopic fracture stress which must be actually reached in small volumes of the specimen whilst the mean overall applied stress remains very low. The theory of Griffith considered the effects of elliptical cracks in a homogeneous, isotropic material, elastic up to the breaking point.

From a calculation by Inglis[49] the stress concentration σ_i at the tip of an elliptical crack of length $2c$ is given by

$$\sigma_i = 2\sigma\sqrt{\frac{c}{\rho}}$$

where σ is the applied stress
and ρ the radius of curvature at the crack tip.

Now it is assumed that the crack will expand spontaneously under the applied stress σ if the stress concentration σ_i at the crack tip is greater than the theoretical cohesive strength value. That is, the crack expands if $\sigma_i \geqslant \sigma_m$

$$\text{or } \sigma_i \geqslant \sqrt{\frac{2\alpha E}{a}} \quad \left(\text{since } \sigma_m = \sqrt{\frac{2\alpha E}{a}} \text{ from page 15}\right)$$

Now for the sharpest possible crack we can assume the radius at its tip ρ is of the order of the interatomic distance a.

Thus $$\sigma_i = 2\sigma\sqrt{\frac{c}{\rho}} = 2\sigma\sqrt{\frac{c}{a}}$$

Eliminating σ_i we find the criterion for fracture. The material fractures if the overall applied stress σ is given by

$$\sigma \geqslant \sqrt{\frac{\alpha E}{2c}}$$

This formula is found to be quantitatively applicable to glass, but it is difficult to see how it can have direct reference to pure metals without some modification. A recent analysis by Elliott[50] has shown that unless cracks are held open by precipitates etc. they are unstable and are unlikely to exist in metals of a length of more than 10 to 20 interatomic distances since they would close by thermal fluctuations and plastic flow even at low temperatures. A further argument against the theory is that to explain the very low cleavage stresses of single crystals a crack length greater than the crystal, and therefore impossibly large, would be required.

Orowan has suggested[2] that in applying the theory to metals a term should be added to the surface energy α to take into account the plastic work P which is done near the fracture surface and by the fracture process. He estimated the value of P to be greater than the value of α by a factor which could be as great as 1,000. The fracture criterion then becomes:-

$$\sigma \geqslant \sqrt{\frac{(\alpha + P)E}{2c}}$$

Two arguments can be advanced against this modification. The crack lengths required for fracture would be far too great, even in polycrystalline specimens. Also the essential assumptions of spontaneous crack extension by the release of elastic energy when the value of σ_m is exceeded locally cannot be modified without changing the whole basis of the Griffith theory.

However, the Griffith Theory has been of great service in indicating why flaws or other forms of internal stress raisers in a material could be regarded as the most likely cause of brittle fracture. A further discussion will be given where this theory has relevance to results obtained in the present work.

(4) IMPURITY LAYER THEORIES OF CLEAVAGE

Amongst many attempts to present a more realistic form of internal stress raiser than the unlikely concept of a pre-existing crack as visualised in the Griffith Theory, a form of equilibrium segregation of impurity atoms along the cleavage plane has been suggested. Zener has pointed out[51] that interstitial atoms present in a body centred cubic lattice may tend to segregate on the (100) plane because the total strain energy may thereby be reduced. This would appear to present a reason for the (100) planes being found to be the cleavage planes.

Further considerations would seem to show that similar segregations are possible in hexagonal close packed metals along the basal (0001) planes if the $^c/_a$ ratio of the axes is greater than that for hexagonal structures of perfect close packing. McClean[52] has suggested that this form of segregation on specific planes would be unlikely to occur in face-centred cubic metals.

The above suggestions are interesting in so far as they attempt to give some indication of the experimentally observed fact that face centred cubic metals do not show cleavage embrittlement even at the lowest temperatures.

It seems possible that, where it exists, forms of equilibrium segregation may facilitate cleavage. Nevertheless, some experimental results have been obtained of cleavage occurring where it is extremely unlikely that any impurity layer segregations can exist. The investigations of Matthewson and Phillips[35] showed that what was formerly regarded as cleavage on prismatic planes in zinc was actually cleavage in the basal plane in twins which have almost the same orientation as the prismatic plane in the matrix. Experiments on single crystals tested in tension in liquid air show that, for crystals over a certain range of orientations, twinning occurs and this is followed by cleavage in the basal planes of the twins. Theoretical considerations indicate that it would be impossible for impurity atoms to migrate to the basal planes in the twins in the time prior to fracture.

(5) THE APPLICATION OF DISLOCATION THEORY TO CLEAVAGE

A number of recent theories have indicated that slip bands might be capable of producing the necessary internal stress concentration to cause a crack which would lead to cleavage fracture. Zener has given some qualitative ideas[18] of how a slip band might initiate fracture and several suggestions along similar lines have been reported. When a stress is applied to a polycrystalline metal then the dislocations forming the slip band travel along the slip planes until the leading dislocation meets the grain boundary, or perhaps a hard precipitate in an impure metal. This forms an effective barrier through which the dislocations cannot pass and a piling up of dislocations occurs at the barrier. The concentration of dislocations at the end of the slip line increases under the applied stress and a small crack is formed as shown in Fig. 4(a) when the two leading dislocations coalesce. If more dislocations coalesce then the size of the crack increases as in Fig. 4(b) and the mechanism of crack growth is clearly apparent. However, the opening up of such a crack through the stopping of a slip band does not necessarily lead to immediate fracture. The crack will extend throughout the region of high stress concentration at the end of the slip band, but it will then stop unless its length is above the critical value required for self-propagation under its own stress concentration. Elliott[50] has given an analysis to show when the crack will become self-propagating. If the crack does not grow to the required length then it will be closed by the general yielding and thermal fluctuations of the metal.

The ideas have been developed quantitatively and for fracture to occur in a metal a crack of the order of the Griffith Crack size must first be produced. Calculations have been made of the stress concentration caused when the dislocations pile up against a barrier and the calculations have shown that if n dislocations of the same sign are pushed against barriers in their slip plane, then the stress concentration at the tip of the array is n times the applied stress. It thus appears that if a sufficiently large number of dislocations could be piled up against a barrier then the stress concentration would be of the order of magnitude of the theoretical cohesive strength σ_m of the material.

Evidence that high internal stresses can be obtained by a pile up of dislocations has been provided by Nye[53]. He made a photoelastic study of stresses in deformed silver chloride crystals and showed that the stresses could be considered to be due to rows of edge dislocations of the same sign which piled up and tended to bend the slip plane. An indication of the stresses set up by piled up dislocations in metals has been given by Barrett[54]. He twisted oxide coated zinc and iron wires plastically and showed that they would spontaneously twist further when the oxide coats were removed and the dislocations were allowed to run out.

The origin of dislocations in a crystal no longer remains a problem since the mechanism postulated by Frank and Read[55] gives a clear indication of the process of creation of dislocations. However, it is still necessary to determine whether a sufficiently large number of dislocations can be piled up to cause the stress magnification required for fracture. Fig. 5 shows an array of dislocations in a slip plane piled up between the source and barrier. A simple analysis has been given[56] which determines approximately the number of dislocations n piled up in a length L of slip plane between the source and barrier under an applied shear stress σ_s. Assuming the source becomes active under a very small stress, then under the applied stress σ the source produces dislocations until the back stress from them cancels the applied stress. If μ is the shear modulus, the source stops working when the elastic strain over the length $2L$ (if the source is in the centre of the grain) has been reduced from $\left(\frac{\sigma_s}{\mu}\right)$ to zero. For this to be accomplished a slip displacement of $2L\left(\frac{\sigma_s}{\mu}\right)$ must occur at the source. If each dislocation produces a displacement b then the number of dislocations n which are created at the source is given by

$$n b = 2L\left(\frac{\sigma_s}{\mu}\right)$$

$$\therefore n = \frac{2L\sigma_s}{\mu b}$$

The formula gives approximately the number of dislocations which can be piled up when general yielding of the grains does not occur. The relevance of the formula to the present work will be discussed later.

It appears as a consequence of dislocation theory that here is a mechanism which can be regarded as providing a realistic conception of the cause of internal stress concentrations from which cracks can grow and ultimately lead to fracture. There are significant differences between these theories and those which consider pre-existing Griffith cracks and these will be dealt with further and in more detail with reference to the results obtained in the present work.

(6) DELAYED YIELD THEORIES OF CLEAVAGE

Since internal stresses in metals can often be relieved by plastic flow, a point of some significance is to understand how, and in what circumstances, the large internal stresses required for cleavage can be built up. In hexagonal metals, which have only one slip plane, the orientation difference between neighbouring grains may prevent a slip band in one grain from initiating yielding in another grain. Hence it is possible that internal stresses can be built up.

In cubic metals, with a few slip planes and several slip directions, yielding in one grain would be expected to cause yielding in neighbouring grains. However, the experiments of Kramer and Maddin[57] have indicated a mechanism whereby body centred cubic metals can withstand high stresses without yielding if the stresses are only applied for a relatively short time. Using single crystals of aluminium, α-brass and β-brass in turn, they measured approximate values of the yield stress at various temperatures and under different times of stressing. For the β-brass at the lowest temperature, $-196^{\circ}C$, and for a short time of stressing, they found the yield stress was markedly increased. To obtain the usual value of the yield stress as measured under static loading the stress had to be applied for about 10^{-2} secs. The delay time necessary for yielding decreased with increasing temperature. Thus at higher temperatures the stress concentrations can be expected to be relieved in a shorter time. This mechanism has been proposed as an explanation of the phenomenon of the transition temperature, since at higher temperatures it would be impossible to build up the internal stress to the magnitude required for cleavage. It appears that the "delayed yielding" is associated with the mechanism of the sharp yield point attributable to very small quantities of carbon and nitrogen in solution. It can be concluded that some of the Frank-Read sources are locked by impurity atoms and thus they do not come into operation to relieve the high internal stress before fracture can occur. This would appear as a further reason for anticipating cleavage in β-brass in common with other body centred cubic metals.

It has been found that aluminium and α-brass and other face centred cubic metals had either no delay time for yield or a time which was too small to be measured experimentally even at very low temperatures. On the above theories an explanation can thus be given of the fact that face centred cubic metals have not been found to show cleavage fracture.

The "delayed yield" theories must have relevance to the cleavage mechanism but, up to the present time, they have not been developed to provide quantitative data.

III - THE FRACTURE CHARACTERISTICS OF β-BRASS AND TWO AL β-BRASSES OF HIGH PURITY IN IMPACT AND TENSILE TESTS.

(1) INTRODUCTION

An investigation of the impact bend and tensile properties of β-brass and 1% and 2½% Al β-brasses was undertaken primarily to determine whether cleavage fractures occurred and, if so, to attempt to elucidate the mechanism of this type of fracture in body centred cubic metals.

β-brass is an alloy of superlattice form occurring over a relatively narrow range of composition based on equal atomic percentages of copper and zinc. It is of body centred cubic structure and a comparison of its plastic properties(31) has shown them to be closely similar to those of pure metals of high melting point which crystallise in the body centred cubic form.

It has been mentioned previously that experiments have shown α-iron, tungsten and molybdenum to give well defined cleavage fractures on (100) planes and these metals are structurally and plastically similar to β-brass. A survey of the literature, however, indicated that there were no reports of investigations with the specific object of determining whether a similar cleavage could occur. Impact tests have been reported by Teed(16) on ($\alpha + \beta$) brasses without indicating any tendency to brittleness even at low temperatures. Nevertheless it might appear that the soft α matrix in the brasses investigated would not permit the conditions of cleavage to be developed in the regions of the β-phase.

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The appreciable "delay time" for yield in β-brasses at low temperatures reported by Kramer and Maddin[57] would indicate that here is another property conducive to the occurrence of cleavage. Some unpublished reports are known which describe observations on forms of cracking in the β-phase of the commercial brasses from ships' propellors etc. However, it is possible that this type of cracking could have occurred through "season cracking" mechanisms which are of entirely chemical origin and not related to cleavage cracks produced completely by applied stresses.

The work to be described was carried out by standard notched bar impact tests and tensile tests down to -196°C and the fractures which occurred were studied. Two Al β-brasses were also investigated since it was known that the addition of aluminium had a marked influence in increasing the yield stress and from the theory of Ludwik[12] this should increase the susceptibility to brittleness. It was also known that aluminium caused intercrystalline brittleness and it might have been possible for cracks to occur at grain boundaries and be propagated by a cleavage mechanism.

(2) PREPARATION OF THE ALLOYS

Since impurities were known to cause various forms of brittleness it was necessary to use material in as pure a form as possible consistent with its use in relatively large quantities. The analyses of the copper and zinc used were:-

	Ag.	Fe.	Pb.	Bi.
% Impurities in copper	0.002	0.028	0.0015	0.003

	Cu.	Fe.	Pb.	Cd.
% Impurities in zinc	0.0003	0.001	0.0025	0.0008

The aluminium which was used in the Al β-brasses in amounts up to $2\frac{1}{2}\%$ was of $99.999^{+}\%$ purity.

It was necessary to produce the alloys free from gas porosity, shrinkage or other casting defects since these can cause spurious results in tests to determine the fracture properties. For each cast a sand mould was made from a "keel bar" pattern. The castings were thus obtained in the form of keel bars and the base of the "keels" were used to provide the material from which the specimens for test were made. The basal area of the keels was 6 x $1\frac{1}{2}$ sq. ins. and the area of the top of the feeder heads $6\frac{1}{2}$ x 4 sq. ins. The total depth of the keel bars was 5 ins.

It was most important to ensure that the material used was entirely of the single β-phase. Any regions of the soft α-phase present would be likely to hinder the propagation of fracture during test. If, on the other hand, any γ-phase was present then the alloys were shown to break under very low stresses and even extremely small traces of the γ-phase caused marked intergranular embrittlement. Some castings not entirely of the β-phase were rejected. Since the β-phase is narrowed to a very small range of compositions on the addition of aluminium, particular care was necessary in the preparation of the Al-β-brass alloys.

Sections of the castings were examined metallographically after polishing on papers and finally with Brasso and White Spirit on a cloth. Ferric chloride was found to be a suitable etch.

A very satisfactory electropolish and etch, recommended by Perryman[58] was also used:

Solution: 2 parts orthophosphoric acid (S.G. 1.75)
1 part distilled water

Current Density: 0.03 amps/cm^2

Voltage: 1.8 to 2.0 volts

Temperature: About 20°C.

Time required: 15 mins.

Cathode: Copper (electrodes horizontal).

An excellent grain contrast was produced by reducing the voltage to about 0.8 for 30 secs.

(3) NOTCHED-BAR IMPACT TESTING

The notched bar impact tests were carried out on standard Charpy specimens of dimensions 10 x 10 x 55 m.m.^{3}; with Izod notch of depth 2 m.m., with the notch angle 45° and root radius 0.25 m.m. The machine used for test was an Amsler Universal Impact Testing Machine of work capacity 120 ft lbs and a calculated striking velocity of 16 ft/sec.

The tests were carried out at room temperature and at -77°C and -196°C. To obtain the temperature of -77°C the specimens were placed in a bath of acetone and solid CO_2, they were then removed and fractured within 3 secs. The temperature of -196°C was obtained by cooling the specimens in liquid nitrogen. They were again tested within 3 secs. of their removal. By copper-constantan thermocouple it was shown that the temperature rise 3 secs. after the removal from liquid nitrogen was not likely to be greater than 3 degrees.

In each test the value for the energy absorption by the pendulum hammer was measured. This was the energy used in deforming and fracturing the specimens. A close examination of the fracture surfaces of the specimens was also carried out.

In the tests on the pure β-brass the appearance of the fracture surface did not change over the range of temperature. The value for the energy absorption only showed a slight decrease from room temperature to -196°C; the average values being 70 and 55 ft. lbs. respectively. This small decrease was no doubt entirely due to a small decrease in the deformation prior to fracture at the lower temperature. These fractures indicated that, even at -196°C, they were above the transition temperature in notch impact tests if one is assumed to exist for β-brass. It might have appeared that, since the grain size of the specimens was large, there were too few cleavage planes suitably orientated opposite the notch. However, by straining and annealing the material before machining, a considerably finer grain size was produced. Still the results remained essentially the same.

Figs. 6 and 7 show a typical β-brass Charpy specimen before and after fracture. It had been electrolytically polished and etched and some indication of the cast grain size is obtained. The fracture surface is as shown in Fig. 8, and, for the most part, it can best be described as "fibrous" except for the patch on the opposite side from the notch and roughly parallel with it. This patch is relatively smooth and shiny and at first sight it might be regarded as a cleavage fracture. However, microscopic examination showed it to be curved and with few surface features and apparently with none having crystallographic significance. Moreover, no grain boundaries were revealed. The surface could be likened to the sides of a "cup and cone" fracture in tension. It could probably thus be regarded as a shear fracture. The relative areas of fibrous and shear fracture appeared to be completely independent of temperature over the range considered. All the specimens tested gave fractures almost identical with Fig. 8.

A series of tests were carried out on Charpy specimens which were machined from material which had previously been cold-worked by 12% compression without subsequent heat treatment. This effectively raised the yield stress of the material and the specimens were tested at the three temperatures as before. Later experiments indicated that the yield stress had been approximately doubled by the prior cold work. It was considered that an increase in yield stress might promote brittleness but very similar results were obtained to those on unworked material.

Tests on specimens from the two Al β-brasses again showed typically fibrous and shear fractures. The average impact energy was 35 ft.lbs. and so was rather less than in the former cases but this was due to rather less specimen deformation before fracture because of the higher yield stress of the material.

It was evident that in the three alloys investigated by notched bar impact tests down to $-196^{\circ}C$, fibrous and shear fractures invariably occurred and there was no indication of cleavage or other forms of brittleness. Moreover, the values of the energy absorptions would indicate that the three alloys tested have remarkably good "impact properties".

(4) TENSILE TESTS

Tensile tests were carried out to determine the approximate yield stresses, plastic and fracture properties of the three alloys at room temperature and at $-196^{\circ}C$. The tests were carried out on a Hounsfield Tensometer and stress-strain curves were plotted to fracture. For tests at $-196^{\circ}C$ the specimen was surrounded by liquid nitrogen in a container similar to the one described by Woodfine(59). The progressive increase of the yield stress with decreasing temperature and with increasing aluminium content is shown in Table 1.

TABLE 1

Alloy		β-brass	1% Al β-brass	2.5% Al β-brass
Yield stress	Room temp.	9	12	21
(tons/sq.in)	$-196^{\circ}C$	13	21	30

All the alloys plastically deformed more than 20% before fracture even at $-196^{\circ}C$. At all temperatures, however, the fractures occurred suddenly. All the alloys failed with a shear type of fracture which was seen to be initiated by intergranular cracks which opened up on the surface on plastic deformation. The true fracture stress did not change significantly with temperature and its value was about 40 tons/sq.in.

An examination of the fracture surfaces showed that the general direction of fracture was at approximately 45° to the direction of applied stress; that is, they were along the planes of maximum resolved shear stress and could be regarded as shear fracture surfaces. The surfaces were shiny, though not microscopically flat or smooth. They did not reveal any crystallographic characteristics and they were similar to the shear fracture patch found in the Charpy specimens. This type of fracture was also observed when a block of β-brass was fractured in compression, further confirming that the planes of fracture were the planes of maximum shear stress. In the tensile tests the elongation at fracture was greater at room temperature than at $-196^{\circ}C$. This would indicate why the impact energy in Charpy tests was found to be rather less at lower temperatures.

Tests were also carried out on specimens with sharp notches. The increase of the yield stress by a factor of 2 to 3 compared with the unnotched specimens was noted. This was in accordance with the theory of the notch effect presented by Orowan and others(13). However, in spite of the high increase in yield stress in these tests, cleavage was not observed in any of the three alloys. The fracture surfaces in each case were entirely of the fibrous type. They were rough, but approximately in the plane of the notch.

(5) CONCLUSIONS

Cleavage fractures could not be produced in any of the three alloys tested in notched bar impact or tensile tests down to $-196^{\circ}C$. Thus the investigation threw no light on the cleavage mechanism in body centred cubic metals.

All three alloys showed considerable toughness under all conditions of test. The "impact properties" of the alloys have been shown to be remarkably good. The alloys fractured by fibrous and shear mechanisms requiring considerable energy absorption. Below $-196^{\circ}C$ it is possible that cleavage fractures may occur but this would require further investigation.

IV - SOME EXPERIMENTS ON THE CLEAVAGE FRACTURE OF PURE POLYCRYSTALLINE ZINC IN SIMPLE TENSION

(1) INTRODUCTION

Pure zinc has long been known to show a well defined cleavage fracture and it has often been used in experiments for studies of the cleavage mechanism. However, little quantitative work has been reported on the properties of the pure polycrystalline material.

Experiments of Polanyi[60] in 1924 showed a strong dependence of true fracture stress on the grain size in tests carried out in liquid air, though only two grain sizes were investigated. The results were later interpreted by Orowan[61] on the basis of the Griffith Crack Theory to show that if cracks could be assumed to exist of the grain diameter d then the true fracture stress σ would be expected to be proportional to $\frac{1}{\sqrt{d}}$. From present concepts of defects in metals, however, such an explanation would appear much over-simplified and a recent analysis by Elliott[50] has shown that it could hardly explain the phenomenon in pure metals because cracks greater than 10 to 20 interatomic distances in length would be unstable and would be expected to close spontaneously under thermal fluctuations and plastic flow even at low temperatures.

The aims of the present work were to investigate the effects of several variables on the occurrence of cleavage fracture so that the mechanism might be better understood. In the present work particular attention was paid to measurements having direct relevance to the theories proposed to explain cleavage.

Several preliminary experiments were made to determine the most suitable experimental procedure. Since, for most grain sizes considered, the zinc was found to show cleavage in simple tension even up to room temperature, there was no need to introduce arbitrary notches. This was a great advantage in the present work since the precise "notch effect" is still incompletely understood quantitatively and accurate stress calculations can not be made. The fact that true cleavage fracture stresses could be measured directly in simple tension permitted the effects of several variables on this quantity to be determined. This was clearly of the utmost importance in obtaining data which could be used to examine the relevance of the various theories.

The aims of the present work were thus resolved into determining the relation between the true fracture stress σ and the grain size d over a range of grain sizes. The effects of temperature T, strain rate $\dot{\varepsilon}$ and plastic deformation ε on the type of fracture and the value of σ were also investigated. In addition reasons were sought for the sharp transition temperature which was reported by Agnor and Shank(62) from impact tests in polycrystalline zinc.

(2) MATERIAL AND EXPERIMENTAL METHODS

(2.1) Material and Specimen Preparation

The material used was zinc of 99.99+% purity. The "Crown Special" (distilled) and "Tadanac" (electrolytic) varieties were both used, but they gave identical results under test. Spectrographic analyses were kindly carried out by Mr. S.W.K. Morgan, of the Imperial Smelting Corporation, Avonmouth. The impurity contents in the two varieties were:

	Pb. %	Cd. %	Cu. %	Fe. %
Crown Special ...	0.0003	0.001	< 0.0001	0.002
Tadanac	0.0003	0.0015	< 0.0001	0.001

The material was obtained in the extruded form in bars of $\frac{1}{8}$th ins. diameter from Messrs. Chas. Clifford & Sons Ltd. It proved possible to obtain a range of grain sizes by selection of specimens from different parts of the bars. To obtain an extension of the range of grain sizes, material was specially extruded to form a fine grain. The range then obtained from all the extruded bars was from 100 to 500 grains/in.

To obtain larger grain sizes annealing treatments were carried out. The range of grain sizes then available was extended down to 25 grains/in. The annealing treatments were carried out after the specimens had been completely machined. The annealing conditions for suitable grain growth were half an hour at 300°C.

Great care was taken in the machining of the specimens. They were of 1/10th sq.in. cross section, with the dimensions as shown in Fig.10. The surface of the gauge length was finished off finally by removing the last 0.002 ins. by polishing with 000 emery paper cooled with paraffin.

(2.2) Tensometer Methods

To determine the values of the true fracture stresses, stress-strain curves were plotted using a Hounsfield Tensometer. This instrument had a maximum load capacity of 2 tons, thus providing a maximum stress of 20 tons/sq.in. in the specimen sizes used. This was found to be adequate in all the tests. The extension of the specimen caused the bending of a beam by an amount which was proportional to the applied stress. The beam was connected by lever to a piston in a cylinder containing mercury and the stress was measured by the movement of the mercury along a tube of fine bore. Hence the distance of mercury movement was proportional to the stress in the specimen. Graph paper was placed on a drum which rotated by amounts proportional to the specimen extension. Thus the values of the stress given by the position of the mercury could be plotted, corresponding to known strains.

In designing a temperature bath so that the specimens could be tested at predetermined temperatures it was necessary that its position should not interfere with the action of the Tensometer or with the recording of stress. It was considered most suitable to use the Tensometer in a vertical position and the temperature bath was of the form shown in Fig. 9. A leather seal prevented leakage of the liquids used at the specific temperatures and the extension shaft prevented undue cooling of parts of the Tensometer and also assisted in the "axial loading" of the specimens. Thermal insulation was provided by lightly packed cotton wool.

The Tensometer calibration was checked by checking a known value of Young's Modulus for a steel specimen which was elastic up to the full load capacity of the Tensometer.

The range over which the temperature bath was used was from $-196^{\circ}C$ to $+50^{\circ}C$. The temperature of $-196^{\circ}C$ was readily obtained by pouring liquid nitrogen into the bath.

A temperature of $-160^{\circ}C$ was obtained by first filling the bath with liquid nitrogen and then slowly pouring in isopentane alternately with more liquid nitrogen until the bath was almost filled with solidified isopentane. A time was allowed for the liquid nitrogen to evaporate and the test was commenced when the isopentane began to melt. The temperature was checked by copper-constantan thermocouple. The tests were each completed whilst most of the isopentane remained solid. The temperature of the specimen did not vary more than 3 degrees from $-160^{\circ}C$.

In a similar manner a temperature of $-117^{\circ}C$ was obtained, in this case ethyl alcohol at its melting point was used as the coolant. The procedure was exactly the same as in the case of isopentane. In obtaining the temperaturesof $-160^{\circ}C$ and $-117^{\circ}C$ it was inevitable in the procedure that the specimens tested at these temperatures were first cooled to $-196^{\circ}C$ and allowed to warm up to the temperature of test.

Tests were carried out at $-77^{\circ}C$ using solid CO_2 and acetone as the coolant. Solid lumps of CO_2 were placed in the bath and pre-cooled acetone was poured in. To obtain a uniform temperature throughout the bath some stirring was necessary.

For tests at $0^{\circ}C$ the bath was filled with ice and water. At $20^{\circ}C$ water at this temperature was used. For tests at $50^{\circ}C$ a heating coil was at first placed round the bath to maintain this temperature. However, this proved to be unnecessary when it was found that tests could be carried out at $50 \pm 3^{\circ}C$ when hot water was poured into the bath.

Preliminary experiments showed that strain rate had a considerable influence on results at the higher temperatures of test. For the faster strain rates, up to 1% per sec., the Tensometer was hand operated, the handle being turned at various fixed rates. For the slower strain rates down to 1/500th % per sec., where hand operation would be far too tedious and exhausting, an $\frac{1}{8}$th H.P. electric motor with a gear system was used. The Tensometer handle was replaced by a set of cog wheels and these were rotated by chain from one of a series of driving wheels on the motor. The range of strain rates thus obtained, between 1 and 1/500th % per sec., ~~were~~ was adequate to determine the effects of its variation.

(2.3) <u>Electropolishing and Grain Size Counting.</u>

Some preliminary experiments clearly showed the necessity for accurate grain size measurements. A check on the grain size could be relatively easily obtained by grain counts along the cleavage surface. However, it was desirable to choose specimens of a given grain size before they were tested.

First a grain count was carried out on the ends of the extruded rods to indicate the grain size variation along the length. By choosing appropriate sections of the rods specimens of approximately the required grain size could be obtained. When certain tests required specimens of equal grain size (or as near as possible) then the specimens had to be obtained from different rods. For the required accuracy it was necessary to count the grain sizes as near to the gauge length as possible. In practice both ends of each specimen were electropolished and the mean grain size measured.

A very satisfactory and extremely rapid electropolish was used as recommended by Kehl[(63)],

Solution: 200 gms. Cr_2O_3

in 1,000 ml. water

Current density: More than 3 amps/cm^2

Voltage: 15 volts.

Time required: 10 secs.

Cathode: Platinum.

The specimen was placed about 1 cm. from the cathode and best results were obtained when the specimen was slightly agitated. The voltage was obtained from 12 Ni Fe cells connected in series directly across the electrodes. The polish was highly satisfactory in 10 seconds. However, the surface grains were invariably found to be heavily twinned in machining and the polishing was continued until a thin surface layer was removed and twin-free grains were observed.

The zinc was not etched. The electropolished surfaces were viewed under polarised light and excellent grain contrast was observed. On scanning electropolished cross sections of the full extruded rods, the grains adjacent to the circumference were found to be very small and next to them was usually a ring of rather large grains. The grains over central diameters of more than $\frac{3}{8}$ths ins. were found to be of a very uniform grain size. The machined gauge lengths of the specimens consisted entirely of the uniform grains over the central region of the extruded rods.

The grain size counting was carried out by viewing the electropolished ends of the specimens under a microscope using polarised light. The numbers of grains lying along two perpendicular diameters of the field of view were counted. Eight fields of view were taken in all for each specimen so that an accurate mean value of the number of grains per in. was obtained. Measurements indicated that for each specimen the grain size was uniform and the accuracy of the measurements was considered to be within $\pm$ 4% for specimens from the extruded rod. With the annealed specimens, however, the accuracy was considerably less because there were relatively fewer grains across the gauge diameter. For the largest annealed grain sizes tested the error could be $\pm$ 20% in the extreme case. Since, however, by far the greater number of specimens tested were of small grain size directly from the extruded rods, the error from grain size measurements is seen to be small. Moreover, since results indicated the square root of the grain diameter to be a factor of great importance in cleavage properties, the error in this quantity was half that of the grain diameter measurements.

Electropolishing of the gauge length was carried out in certain cases. This presented certain problems notably because of the shape of the specimens and the need for a high current density. These were largely overcome by covering the ends of the specimens with wax and leaving only about half an inch of the gauge length uncovered in the centre. Good polishes were obtained in these cases and the form of deformation of the specimens could be microscopically examined, but these specimens were unsuitable for true fracture stress measurements since they tended to fracture at the junctions of the electropolished length with the unpolished length.

So that some light could be thrown on the connection between twinning and fracture, several specimens were electropolished after fracture to observe the amount of twinning.

(3) EXPERIMENTAL RESULTS

(3.1) The Effect of Grain Size on Cleavage at Low Temperature.

(3.11) The Relation between True Fracture Stress and Grain Size at $-196^{\circ}C$

Specimens were selected over the range of grain sizes available and mounted in the temperature bath on the Tensometer in turn. They were cooled to $-196^{\circ}C$ by pouring in liquid nitrogen and a stress-strain curve was plotted in each case up to the point of fracture. The fracture always occurred suddenly. Preliminary experiments had shown that variation in the strain rate over the limits of the apparatus had no influence on results at $-196^{\circ}C$ and a convenient rate was chosen as 0.053% per sec. The stress-strain curves showed that no overall plastic deformation had taken place at this temperature. This was further checked by diameter measurements before and after test at several points along the gauge length.

Examination of the cleavage facets showed them to be crossed by parallel striations as shown in Fig.11 at x 200. Thus highly reflecting long narrow areas were observed in each grain. The dark striations were steps of varying depths. The long narrow areas were easily demonstrated to be sections of the basal (0001) planes by observing them under a microscope using polarised light. To do this the analysing and polarising nicols of the microscope were crossed and the specimen was then rotated, keeping any particular facet in view. The areas were seen to remain dark on rotation, thus indicating that they were sections of basal planes. Fig. 12 shows the gauge length of the specimen, electropolished after fracture. Some twins are seen to be present in the grains adjacent to the fracture surface marked by S. It appears then that the cleavage facets are crossed by twins and these produce the striated structure. Fig. 12 clearly shows that the gauge length is completely twin free except very near the fracture surface. This would appear to indicate a connection between twinning and fracture. Figs. 11 and 12 were typical of specimens fractured at -196°C.

On correlating the true fracture stress values σ with the mean grain diameters d, the results indicated that when σ was plotted against $\frac{1}{\sqrt{d}}$ then the graph was a straight line through the origin, as shown in Fig.13. This provided confirmation over a range of grain sizes of the relation originally suggested from the work of Polanyi and Masing[60]. The deviation of results from this line was seen to be small. The values of could be measured to $\pm$ 0.2 tons/sq.in. and, as was previously discussed, the calculations of $\frac{1}{\sqrt{d}}$ were not likely to be more than $\pm$ 2% in error for extruded rod specimens, though in the extreme case of the largest grain annealed specimens, the accuracy in $\frac{1}{\sqrt{d}}$ was of the order of $\pm$ 10%. It is thus seen that the accuracy is greatest for specimens of smallest grain size, since the value of d can be more accurately determined and the percentage error in the σ measurements is considerably less. Considering the product $\sigma\sqrt{d}$ (which corresponds to the gradient of the graph), then the combined experimental errors in determining this quantity for each specimen of the extruded rod should not be greater than 5%.

Of the points shown on Fig. 13 the two specimens showing lowest σ values, corresponding to a large grain size d and thus a low value of $\frac{1}{\sqrt{d}}$ were obtained from the annealed material. The remaining seven specimens were chosen from sections of the extruded rod so that the values of σ could be obtained for the complete range of grain sizes available. The result for a specimen cycled three times between room temperature and -196°C before testing was also found to lie on the line.

Since the tests at -196°C gave fractures in tension without overall plastic deformation, though with some localised twinning associated with the process of fracture, it might be anticipated that the experimental values of the true fracture stresses would have considerable significance. The fractures obtained at this temperature could be regarded as the closest approach for metals to the idealised condition of fracture in the elastic range. Some interpretations will be discussed in the next section.

(3.12) Interpretation of Experimental Values obtained for True Fracture Stresses at -196°C

Consideration of the numerical values obtained for the true fracture stresses at -196°C plotted in Fig. 13 shows them to be very much less than the theoretical cohesive strength value which was calculated in Section II(1) to be of the order of 2,000 tons/sq.in. However, Fig.13 shows the increase of true fracture stress with decreasing grain size and the question arises as to whether sufficient grain refinement, assuming this were possible, would ultimately increase the σ value to the theoretically calculated value of σ_m.

If we could assume that the grain size d could be reduced to the order of magnitude of the interatomic distance a, then we should have:

$d = a = 3 \times 10^{-8}$ cms. $= 10^{-8}$ ins.

and therefore $\frac{1}{\sqrt{d}} = 10^{-4}$ ins.$^{-\frac{1}{2}}$

Further assuming that the $\sigma \propto \frac{1}{\sqrt{d}}$ relation exists over the entire range, then the value of σ would be 4,200 tons/sq.in. corresponding to $\frac{1}{\sqrt{d}} = 10^{-4}$ ins.$^{-\frac{1}{2}}$. Taking into consideration the many assumptions and approximations in the theory, it can be said that the true fracture stress of zinc, having a grain size of the order of the interatomic distance, should be of the order of the theoretical cohesive strength σ_m.

The above reasoning would suggest that the value of the theoretical cohesive strength σ_m might be attainable if sufficient grain refinement could be carried out. The problem can perhaps be regarded as being closely similar to that of obtaining a "flawless metal" (that is, one without internal stress raisers) corresponding to nearly "flawless glasses", the nearest approach to which are fibres of newly drawn glass which have been shown to have true fracture stresses approaching the theoretical cohesive strength σ_m. These arguments are seen to be in complete opposition to the fundamental postulates of the present thermodynamic theories of strength which assume that, even in "flawless solids", a form of melting should set in to cause fracture long before the theoretical cohesive strength is attained.

It is interesting to compare the values obtained for the true fracture stresses measured in the present experiments with those predicted by the Griffith Theory. If the crack length 2c is assumed to be of the order of the mean grain diameter d then we obtain immediately the relationship $\sigma \propto \frac{1}{\sqrt{d}}$ from the formula

$$\sigma = \sqrt{\frac{\alpha E}{2c}} = \sqrt{\frac{\alpha E}{d}}$$

It is to be noted first that the formula predicts fracture on the planes perpendicular to the direction for which Young's Modulus has the least value. In zinc the values are:

E at 70° to the c axis = 12,130 Kg./m.m.^{2}

E parallel : : c : = 3,560 Kg./m.m.^{2}

Thus there is a considerable difference between the two directions. This would indicate that the (0001) plane is likely to be the most favourable cleavage plane and this has been borne out by experiment. To compare the σ values calculated from the above formula with those obtained from experiment, we can choose any arbitrary grain size.

Assuming d = 0.01 ins. = 0.025 cms.

α = 10^3 ergs/cm^2

E = 3.560×10^3 Kg/m.m.^{2} = 3.56×10^{11} dynes/cm^2

$\therefore \sigma = \sqrt{\frac{\alpha E}{d}}$ = 1.2 Kg/m.m^2 = 0.8 tons/sq.in.

We can compare this value with that obtained from experiment when d = 0.01 ins. or $\frac{1}{\sqrt{d}}$ = 10 ins.$^{-\frac{1}{2}}$ The experimental value = 4.2 tons/sq.in. is obtained directly from Fig. 13.

The Griffith formula is thus seen to provide numerical values of roughly the required order of magnitude for σ as well as indicating the correct σ vs. $\frac{1}{\sqrt{d}}$ relationship if Griffith cracks can be assumed to exist of a length which can be identified with the mean grain diameter. A further discussion of the relevance of the formula will be given later in the light of further work.

(3.13) The Relation between True Fracture Stress and Grain Size at $-160^{\circ}C$

A point of some importance is the variation of σ with temperature T. The results at $-196^{\circ}C$, however, showed that in considering this variation the effect of different d values must be taken into account. The Griffith Theory, successfully applied at $-196^{\circ}C$, would appear to predict a small decrease in σ with increase in T (provided no overall plastic deformation occurred prior to fracture) because the values of the surface energy α and the Young's Modulus E are both expected to decrease slowly with increase in T.

For the experiments at $-160^{\circ}C$ specimens of a range of grain sizes were again selected from the extruded rods and two further specimens were annealed to obtain larger d values on grain growth. The temperature of $-160^{\circ}C$ was attained as described in the experimental methods and stress-strain curves were plotted to the point of fracture in each case.

A graph was again plotted of σ against $\frac{1}{\sqrt{d}}$ and the results are shown in Fig.14. The four specimens of largest d and therefore of smallest σ fractured without overall deformation. The stress-strain curves and careful measurements of the gauge diameters showed that in the two specimens of smallest d value some slight overall deformation had occurred prior to fracture.

An examination of all the fracture surfaces showed them to be similar to the one illustrated in Fig.11 broken at $-196^{\circ}C$. In the two specimens where some deformation had occurred by slip and twinning prior to fracture along the gauge length, there was no apparent change in the appearance of the cleavage facets.

Fig. 14 shows the line of σ vs. $\frac{1}{\sqrt{d}}$ drawn through zero and the first four points since these corresponded to the specimens which fractured without overall prior deformation. It appeared possible that the σ values of the two specimens of smallest grain size were slightly increased by the deformation which occurred prior to fracture. Later work, which will be discussed in the next section, indicated a small increase in σ after small amounts of cold work. The dotted portion of the σ vs. $\frac{1}{\sqrt{d}}$ line might be considered to represent hypothetical σ values which would be obtained in the absence of prior deformation.

The graph at -160°C (Fig. 14) can be profitably compared with the graph at -196°C (Fig. 13). The relative slopes of the two σ vs. $\frac{1}{\sqrt{d}}$ lines are further given for easier comparison in Fig. 15. It would appear just outside the experimental errors that the line gradient at -160°C was slightly greater than the gradient at -196°C. Thus σ is found to be greater at -160°C than at -196°C for specimens of similar grain size and in the absence of overall deformation pmior to fracture. To confirm this result, however, it was thought desirable to carry out experiments at higher temperatures rather than test further specimens at -160°C. The results at -160°C were in opposition to the predictions of the Griffith Crack Theory. A further discussion will be given later.

The results for the two smallest grain sizes tested at -160°C indicated that some plastic flow had taken place prior to fracture. It was to be anticipated that specimens tested at higher temperatures would undergo increasing flow before fracture. It was necessary to attempt to determine as far as possible the effects of prior deformation on fracture.

(3.2) <u>The Effects of Plastic Deformation prior to Fracture.</u>

A point of great difficulty in investigations of fracture properties has been to attempt to compare the results on specimens which undergo differing amounts of cold work prior to fracture. The problem has been invariably to deduce the effects of the cold work given during the test. Thus a study of the fracture characteristics must involve a knowledge of the flow properties and the mechanisms by which the plastic deformation occurs.

In many previous studies of fracture, attempts have been made to obtain a value for σ by extrapolation to a value which it might be considered to have at zero deformation, though this quantity would seem to be of only hypothetical interest. The attempts have usually taken one of two forms. One method has been to introduce notches whereby the plastic deformation was largely inhibited. The other method has been to prestrain the metal at temperatures where it was ductile and then to test at some lower temperature where relatively little or no further deformation occurred. A discussion of the methods has been given by Gensamer and others[64] in an extensive review.

Both methods are open to several objections. The notch effect is still incompletely understood in an exact quantitative form, though some calculations have been made of the effective stresses at a notch. There is also the further difficulty that it is not possible to be certain that plastic deformation at the notch does not occur. The prestrain method can be criticised since the effects of deformation are well known to be dependent on the temperature at which it is carried out.

In the present work the prestrain method was used. To counter the objection of the effect of variation of temperature, the prior deformation was carried out at three temperatures. Also some metallographic work was carried out to determine the form of the deformation and the effects of its variation on the appearance of the cleavage facets. After the various pre-strains had been given, in no case did further deformation occur during the tests at $-196^{\circ}C$.

(3.21) The Effect of Prestrain on the True Fracture Stress at $-196^{\circ}C$.

First it was considered necessary to determine the effect of prestrain ε_p given at one temperature to specimens of very nearly equal grain size on the true fracture stresses measured at $-196^{\circ}C$. To obtain a sufficient number of specimens of grain size as nearly equal as possible from the material available, they were chosen from lengths of extruded rod having about 115 grains per in.. So that a conveniently large range of pre-strains could be given without the specimens fracturing during prestraining, the prestrain temperature was chosen as $50^{\circ}C$, with the strain rate 0.053% per sec. It was found that even small variations of strain rate had appreciable effects at this temperature.

The prestraining was carried out at $50 \pm 2^{\circ}C$ with hot water in the temperature bath. Stress-strain curves were plotted in each case so that the amount of prestrain could be ascertained. Some interesting observations were noted on the stress-strain curves at this temperature. These were probably attributable to the presence of nitrogen in the zinc. A discussion is given in the Appendix. In measuring the value of the prestrain the units of "natural strain" were used. The natural strain ε_p is defined by the value of $\log \frac{Ao}{A}$ where Ao and A are the initial and final areas of cross section of the specimens respectively. The values of Ao and A were measured at various positions along the gauge length by micrometer. The values of the natural strain can be considered accurate to 0.005.

When the required value of prestrain was reached, as shown from the stress-strain curve, the specimen was unloaded and the hot water was emptied from the bath. Then liquid nitrogen was poured into the bath and the true fracture stress was measured at -196°C.

The results obtained are shown in Fig. 16. They are immediately seen to be of considerable interest. The value of σ measured at -196°C first increases to a maximum at about 0.04 prestrain and the further prestrain causes a steady fall in the value of σ to about 60% of its value without prestrain. The experiments reported in the review by Gensamer[64] on the effect of prestrain on the fracture stress of steels indicate that a continuous increase is usually caused.

The present findings would not appear to have very obvious interpretations. It seems that the effects of deformation on true fracture stress are likely to be complex. A metallographic examination was made of the cleavage facets and this showed considerable changes in their appearance for differing values of prestrain. For specimens which were given a prestrain ε_p of less than 0.07 the cleavage facets were striated by twin markings and had an appearance similar to the one shown in Fig. 11. For specimens given prestrains greater than 0.07 an increasing number of grains were observed without striations due to twinning. Non-crystallographic markings became increasingly apparent. A typical cleavage facet from a specimen having a prestrain more than 0.07 is shown in Fig. 17. The facet was from a specimen having ε_p = 0.174 and fractured at $-196^{\circ}C$. The facet, at x 250 magnification, is seen to be flat and the polarised light test previously described identified it as a basal plane. The generally rough shape of the fracture surface of the specimen as a whole made it impossible to view any particular facet under higher magnifications. Nevertheless Fig.17 clearly shows a great number of "river-like" structures.

To determine further characteristics of the effects of prestrain additional factors were investigated.

(3.22) Further Experiments on the Effects of Prestrain.

It appeared desirable to determine whether the form of the true fracture stress against prestrain curve was similar for specimens of differing grain sizes. As shown in Fig. 18, a graph was drawn of the product $\sigma\sqrt{d}$ of the specimens tested, against ε_p. This was clearly valid for ε_p = 0, because of the results shown in Fig. 13, and it remained to be determined whether specimens of different grain sizes could be fitted on the same curve. Table 2 gives the details of the results obtained for the points plotted on the graph of Fig. 18. The results indicated that specimens over a range of grain sizes could be fitted on the same curve of $\sigma\sqrt{d}$ against ε_p. Moreover the metallographic observations on the cleavage facets of the specimens over the range of grain size indicated that their appearance only depended on the amount of prestrain given prior to fracture. When ε_p was less than about 0.07 all the cleavage facets were crossed by striations due to twinning. For greater prestrains the facets showed fewer striations but increasing "river-like" structures.

TABLE 2

(as illustrated in Fig. 18)

Prestrain Temperature °C	Prestrain $\epsilon_p = \log \frac{A_0}{A}$	σ tons/sq.in. at -196°C.	$\frac{1}{d}$ grains/in.	Product $\sigma\sqrt{d}$ (tons/sq.in.) $\text{ins}^{\frac{1}{2}}$	Time at room temperature between prestrain and test.
50	0.022	5.0	111	0.476	1 day
50	0.047	5.1	114	0.475	1 day
50	0.068	4.5	118	0.413	1 day
50	0.077	4.1	112	0.387	1 day
50	0.090	3.2	109	0.308	3 days + 4 hrs. at 100°C.
50	0.127	3.1	116	0.287	3 days
50	0.174	2.6	118	0.240	1 day
50	0.243	2.5	120	0.227	1 day
50	0.259	2.5	129	0.216	10 secs.
20	0.011	4.9	121	0.446	10 secs.
20	0.032	5.7	140	0.480	2 days
20	0.068	5.1	143	0.425	2 days
20	0.086	5.3	189	0.384	10 secs.
38	0.092	4.1	135	0.352	½ hour
20	0.045	7.3	258	0.454	½ hour
20	0.165	4.4	273	0.266	½ hour
Prestrain in Compression					
20	0.016	8.7	357	0.461	5 days
20	0.102	8.0	430	0.387	5 days

The effect of prestrain in compression was also investigated. Two sections, each about 4 ins. long, were cut from the extruded rods and machined to have smooth flat ends perpendicular to the rod axis. The height to diameter ratio was too great for stability at large prestrains and so these could not be obtained. A prestrain of 0.102 was, however, effectively carried out in compression. The apparatus was a Dennison machine which could be used in compression to provide a load of up to 50 tons. The lengths of the extruded rod were placed in turn in the machine with their ends resting on flat, highly polished steel blocks and a load of 12 tons was adequate for their compression. Two specimens were then machined from the compressed lengths.

As shown in Table 2, the specimen for which $\varepsilon_p = 0.016$ showed a small increase in the $\sigma\sqrt{d}$ value at -196°C. This indicated that compression had similar effects to tension. The specimen for which $\varepsilon_p = 0.102$ showed a small decrease of $\sigma\sqrt{d}$ which again showed the similar effects of prestrain in compression and tension, though the result was somewhat above the curve. Table 2 also gives results for specimens prestrained in tension at 20°C. They lie closely on the curve in Fig. 18.

To determine any effects due to stress recovery, various times were allowed after the prestraining and before the true fracture stress measurements were made. These are shown in Table 2. The effect of the prestrain was independent of the time allowed after it was carried out. In order to obtain an estimate of the rate of recovery, a series of tensile tests were made on a specimen at room temperature over a period of several weeks and also after holding the specimen for 6 hours at 100°C. Measurements of the approximate yield stress in each test indicated that recovery could not be brought about without recrystallisation. This was different from the observations of Haase and Schmid[65] who found that for single crystals complete recovery took place in less than 24 hours at room temperature. The present work emphasises the differences in the mode of deformation of poly and single crystals of zinc.

The results obtained showed that the curve plotted in Fig.18 had general application over the range of grain sizes and was not dependent on the time allowed for resting after prestrain. It appeared desirable, however, to examine the effect of prestrain at a lower temperature.

(3.23) The Effect of Prestrain at -77°C.

Since the effects of deformation are known to be dependent on temperature, it was necessary to examine the effects of prestrain at a temperature lower than 20°C on the σ value at -196°C. A difficulty here was that only small prestrains could be given, otherwise the specimens fractured during prestraining.

Specimens of several grain sizes were prestrained at -77°C. The technique of obtaining this temperature was described in the section on experimental methods. Even the smallest grain size, however, could not be given a prestrain ε_p greater than 0.04 without fracture during prestraining. Nevertheless this range of ε_p proved sufficient to determine the general prestrain effects. Table 3 shows the results obtained for prestraining at -77°C and, taken from this, Fig. 19 shows the graph obtained by again plotting $\sigma\sqrt{d}$ against ε_p .

TABLE 3

(as illustrated in Fig.19)

Prestrain ε_p at $-77^{\circ}C$	σ tons/sq.in. at $-196^{\circ}C$.	$\frac{1}{d}$ grains/in.	Product $\sigma\sqrt{d}$ (tons/sq.in.)$ins^{\frac{1}{2}}$
0.009	5.3	119	0.486
0.017	8.5	300	0.491
0.019	6.0	169	0.462
0.025	10.0	490	0.453
0.028	9.5	462	0.424
0.033	9.6	457	0.449

Over such a small range of prestrains it is difficult to draw an accurate curve, but the graph of Fig. 19 shows a maximum value of $\sigma\sqrt{d}$ occurring for prestrains of about 0.015 given at $-77^{\circ}C$. The shape of the curve is similar to the one of Fig. 18 for prestrain at higher temperatures except that the same variation in $\sigma\sqrt{d}$ is brought about by relatively smaller values of ε_p at $-77^{\circ}C$.

At $-77^{\circ}C$ a given prestrain clearly had a greater effect than the same value of the prestrain given at $20^{\circ}C$. This might possibly be expected from a consideration of the stress-strain curves at $-77^{\circ}C$ and $20^{\circ}C$. These are shown in Fig. 20 which represents the true stress - natural strain relationship for specimens of nearly equal grain size to their points of fracture in tests at various temperatures. An exact measure of the relative amounts of strain hardening was not calculated since gauge diameter measurements were not made continually during test because the temperature bath prevented accessibility to the specimens. However, the curves clearly show that the rate of strain hardening at -77°C is considerably greater than that at 20°C. Thus it might be expected that a smaller pre-strain at -77°C would have similar effects to a larger prestrain at 20°C. This is borne out by the corresponding curves in Figs. 18 and 19.

The tests at -196°C were again carried out immediately after prestraining and also after an interval of a few days and no difference could be detected in the general effects of the pre-strain.

From a consideration of the effects of prestrain at 50°C, 20°C and -77°C, it seemed that a generally similar effect of the prestrain occurred, but with a given prestrain at a lower temperature being equivalent to a rather larger prestrain at a higher temperature.

(3.3) True Fracture Stress Measurements at -117°C and -77°C.

Specimens over a range of grain sizes were selected for test at -117°C by the techniques previously described. Two large grain specimens were obtained by annealing. Stress-strain curves were again plotted to fracture and the results were again correlated in a graph of σ against $\frac{1}{\sqrt{d}}$ in a similar manner to the results previously obtained in tests at -196°C and -160°C. The graph at -117°C is shown in Fig. 21.

Only the two specimens of largest grain size showed no overall deformation prior to fracture during the tests. The ductility of the specimens was seen to increase continuously with decreasing grain size, though accurate measurements were difficult since the maximum deformation prior to fracture for the smallest grain size specimen was only 0.017. The points obtained on the graph in Fig. 21 show a marked deviation from a straight line. It is of some interest to compare Fig. 21 with the results obtained for the effect of prestrain at -77°C on σ at -196°C as shown in Fig. 19. From the comparison it would appear a reasonable assumption that there was a deviation of points from a straight line of $\sigma \propto \frac{1}{\sqrt{d}}$ at -117°C (shown dotted in Fig. 21) because of the deformation which occurred prior to fracture at this temperature. The hypothetical dotted line can be considered to be the line of true fracture stresses which, theoretically, would be obtained for specimens fracturing without overall deformation. The gradient of this line at -117°C is also shown on Fig. 15 for comparison with the line gradients at -196°C and -160°C. It indicates a further increase of σ with temperature.

In a similar manner, true fracture stress measurements were carried out on specimens over a range of grain sizes at -77°C. A σ vs $\frac{1}{\sqrt{d}}$ plot was again made and this is shown in Fig. 22. The actual values of σ obtained are represented by circles. At -77°C only the specimen of largest grain size fractured without overall deformation. The specimens showed increasing ductility prior to fracture, with decreasing grain size. The ductility vs. grain size relationship is given in Table 4. The points obtained for the true fracture stresses in Fig. 22 are seen to lie on the arc of a curve. This would give further support to the indication from Fig. 19 that a small amount of strain causes an increase in σ up to a maximum value. Further strain before fracture then decreases the σ value. This opened up the possibility of obtaining hypothetical σ values corresponding to values at zero deformation.

(3.4) The Extrapolation of True Fracture Stress Values at $-77^{\circ}C$ to Hypothetical Values corresponding to Fracture without Deformation.

In line with many other previous investigations on fracture, in the present work it appeared relevant to compare quantitatively the σ values obtained after the amount of strain which occurred naturally in the tests, with a similar value of prestrain given to a specimen at the same temperature and then tested to fracture at $-196^{\circ}C$ where no further deformation occurred. Since the effects of prestrain at $-77^{\circ}C$ on σ at $-196^{\circ}C$ were known, the fractional change in the σ value caused by changes in ε_p, was applied to the results obtained for σ measurements at $-77^{\circ}C$. The assumption was made that the same value of strain had the same fractional effect on the value of σ. This assumption has been made in many previous studies of fracture and several applications have been listed in the review by Gensamer[64]. McAdam, Geil and Mebs[66] have plotted low temperature cleavage strengths for steels against prestrain given at a higher temperature and used the results to correct the cleavage strength values to zero deformation. The method cannot be considered to be exactly applicable in practice since the necessary prestrain cannot be given at the required temperature without fracture taking place in prestraining. Nevertheless, a close approximation could be obtained in the present case since the required value of prestrain was given to specimens of rather smaller grain size having the required ductility.

The method of calculating the values of σ by extrapolation to zero deformation is given in Table 4 and will be understood from an example. It is seen from Fig. 19 that the value of σ is a maximum for $\varepsilon_p = 0.015$. This value is 15% greater than the value without prior deformation. Now in the tests at $-77^{\circ}C$ shown in Fig. 22, the specimen for which σ = 7.8 tons/sq.in. deformed by an amount 0.016 prior to fracture. It is assumed that this value of σ is 15% greater than the value which would have been measured at $-77^{\circ}C$ if no deformation had taken place. Thus the hypothetical value without deformation would be 6.6 tons/sq.in. In all cases, to extrapolate values of σ at $-77^{\circ}C$ to the hypothetical values corresponding to zero deformation, a percentage change of the true fracture stress is assumed which depends on the value of strain prior to fracture. To distinguish between prestrain given prior to testing at $-196^{\circ}C$ and the strain occurring naturally in a test before fracture, they will be denoted by ε_p and ε_c respectively.

TABLE 4

(Illustrated in Fig. 22)

Measured value of σ at $-77^{\circ}C$ σ tons/sq.in.	Strain before fracture during test ε_c	$\frac{1}{d}$ grains/in.	% change in σ due to ε_p as in Fig.19.	Value of σ extrapolated to zero deformation.
2.7	0	18	0	2.7
4.2	0.006	35	+ 11	3.6
7.8	0.016	116	+ 15	6.6
8.3	0.022	171	+ 12	7.3
8.6	0.032	182	+ 6	8.1
9.9	0.043	257	0	9.9
11.3	0.046	402	− 3	11.6
11.8	0.048	443	− 4	12.3

The Table shows how the ductility increases with decreasing grain size. Now, if the σ values, extrapolated to zero deformation, are plotted against $\frac{1}{\sqrt{d}}$ as shown by crosses on Fig. 22, the result is seen to be a straight line through the origin. The deviations from this straight line are within the experimental errors due to extrapolation. It appears that σ can be considered proportional to $\frac{1}{\sqrt{d}}$ at $-77^{\circ}C$ if correction is made for the plastic deformation occurring prior to fracture. Moreover, when the gradient of the dotted line at $-77^{\circ}C$ is compared with the line gradients previously obtained for lower temperatures, then a uniformly increasing gradient is found to occur with increasing temperatures. This is illustrated in Fig. 15. It is to be noted that the gradients of all the lines at -196, -160, -117 and $-77^{\circ}C$ have some points obtained from specimens which fractured without prior deformation. Thus the observation that σ increases with temperature T is determined without assuming the validity of the hypothetical stress calculations involved in the extrapolation of the σ values to zero deformation. The corrections applied to the σ values for the effect of deformation in the specimens of smaller grain size merely bring them into line with the general results.

(3.5) <u>True Fracture Stress Measurements at $0^{\circ}C$.</u>

Tensile tests were carried out over the range of grain sizes at $0^{\circ}C$. At this temperature the effects of deformation became increasingly important. Even the specimens of largest grain size showed some ductility prior to fracture and increasing ductility with decreasing grain size was very marked. Small variations in strain rate had considerable effect. The strain rate was maintained constant at 0.053% per sec. since this value had been used

When the values obtained for the σ measurements were plotted against $\frac{1}{\sqrt{d}}$, the results were as shown by circles in Fig. 23 and could apparently be represented by a straight line not passing through the origin. This, however, was only if no correction was made for the differing amounts of deformation occurring in the specimens prior to fracture. The elongations of all the specimens tested at 0°C were greater than the value corresponding to that giving the highest σ value as shown in Fig. 18. If the effect of deformation was now taken into account exactly as described in the preceding section and the σ values were extrapolated to the hypothetical values corresponding to zero deformation on the basis of the prestrain curve in Fig. 18, then the results were as indicated by crosses in Fig. 23. The specimens of large grain size had low ductilities and thus the amount of deformation they received in test increased the values of σ. The specimens of smaller grain size showed greater ductility prior to fracture and the amount of deformation occurring in them was sufficient to decrease the value of their true fracture stresses as indicated in Fig. 18.

The results, marked with crosses in Fig. 23, and indicating the hypothetical σ values extrapolated to zero deformation are again seen to lie on a straight line passing through the origin. An indication of the value of the gradient of this line can be obtained from Fig. 15. Again it would appear that a $\sigma \propto \frac{1}{\sqrt{d}}$ relation should hold at 0°C in the absence of deformation prior to fracture.

Interesting observations were noted in the change of appearance of the cleavage facets with differing amounts of deformation. In the three specimens of largest grain size where the elongation to fracture was less than 0.07 the cleavage facets were crossed by twins, and they appeared similar to the one shown in Fig. 11. For the smaller grain size specimens fracturing after greater strains an increasing n mber of facets had an appearance similar to the one shown in Fig. 17. Marked "river-like" structures were developed and there was an absence of striations due to twinning. This provided further evidence that the strain ε_c occurring in the tests to cause fracture had comparable effects to the prestrain ε_p given at the same temperature followed by test at -196°C. Thus the metallographic observations supported the arguments used for the correction of the σ values for the deformation occurring prior to fracture.

(3.6) True Fracture Stress Measurements at 20°C.

In tensile tests at 20°C and with a strain rate of 0.053% per sec. the specimens showed a considerably greater ductility prior to fracture than in the tests at 0°C. The smaller grain sizes tested at 20°C showed ductile fractures and they necked down to almost 100% reduction in area. This represented fracture above the transition temperature. Fig. 25 shows the types of fractures and to compare the ductilities an unbroken specimen is included. Two specimens show typical cleavage fracture, though after differing amounts of prior deformation. The cleavage fractures are seen to occur in a plane perpendicular to the specimen axis. The ductile fracture on the other hand is seen to have occurred after correspondingly much greater deformation and considerable necking. No fracture stress measurements were made on specimens giving ductile fractures.

For the larger grain size specimens which gave cleavage fractures at 20°C, the σ values, plotted against $\frac{1}{\sqrt{d}}$, are shown by circles in Fig. 24. The values lie on a straight line which does not pass through the origin if the effects of deformation on σ are not taken into account. If, however, a correction is made on the basis of Fig. 18 to obtain the hypothetical σ values for zero deformation, then the values of σ shown by crosses on Fig. 24 are obtained. The corrected σ values again lie on a straight line through the origin. The gradient of the dotted line corresponding to the σ values corrected for zero deformation in Fig. 24 is also represented in Fig. 15 which can now be considered to give the $\sigma \propto \frac{1}{\sqrt{d}}$ relationship for the various temperatures of test. The increase of line gradient with temperature is shown in the plot of $\sigma\sqrt{d}$ against T in Fig. 26.

Metallographic examination of the cleavage facets in the fractures at 20°C showed that for the three specimens of largest grain size, which fractured after less than 0.07 neutral strain, the cleavage facets were crossed by striations due to twinning and were very similar to the one shown in Fig. 11. The remaining specimens which fractured after greater deformations had facets comparable with that of Fig. 17 and marked "river-like" structures were developed. Fig. 27 shows a typical cleavage facet of a specimen which was fractured during a test at 20°C. The natural strain occurring prior to fracture was 0.175. It is of interest to compare Figs. 17 and 27 at x 250. The facets are very similar. It appears that twinning which causes stepping on the cleavage facets is inhibited during fracture if the specimens have undergone neutral strains greater than about 0.07, though some twins are seen in the basal plane in Fig. 27. The twin markings are mutually at 60° with each other. Unfortunately, in the present work, close observations of the cleavage facets could not be made under very high magnifications since it was difficult to view any particular facet without other cleavage facets prohibiting the close approach of the microscope objective. To carry out studies such as those reported, for example, by Zappfe and others(67) it would be necessary to use specimens of larger grain size.

In the experiments at 20°C it was observed that, for the specimens giving typical cleavage fractures, the fractures invariably occurred before a natural strain of about 0.30 was reached. Specimens having sufficiently small grain size and greater ductility necked down completely after further elongation which proceded until a ductile fracture occurred. Cleavage was never found to occur in tests when the temperature was sufficiently high, or the grain size small enough to allow deformation to proceed to more than 0.30 natural strain without fracture occurring. Thus there would seem to be a difficulty in producing cleavage fractures in zinc which has been deformed by more than 0.30 natural strain.

In view of this a specimen of small grain size was elongated to 0.40 natural strain at 20°C and then tested to fracture at -196°C. Fig. 28 shows the typical results of such a test. The rather striking observation isthat cleavage is found to occur at -196°C, outside the neck of the specimen where the natural strain is less than 0.30. The general conclusion is that deformations of more than 0.30 natural strain increase the true fracture stress of zinc, since there would then appear to be a loss of the capacity to show cleavage fracture. The experiments indicate that heavily worked specimens do not show cleavage fractures even at low temperatures. This links up with the observations in the higher temperature tests that if deformation can proceed to more than 0.30 natural strain then the specimens continue to deform and ultimately give ductile fractures.

These facts can be identified with the phenomenon termed "rheotropic brittleness" by Ripling and Baldwin[68]. They found that specimens of zinc and steels were less liable to fracture after heavy deformation than without prestrain. If high prestrains were given, then the ductility was actually found to be increased in tests at lower temperatures. Unfortunately they did not measure true fracture stresses or examine the types of fracture.

(3.7) The Effect of Strain Rate.

Over the range of strain rates which were available from 1 to 1/500 th % per sec., the influence of strain rate below $-77^{\circ}C$ was too small to be detectable. Its effect became increasingly marked at higher temperatures.

The elongation occurring prior to cleavage ε_c was markedly dependent on the strain rates employed and also on the grain size of the specimens under the given conditions of test. Thus in determining the influence of strain rate alone it was particularly necessary to choose specimens having grain sizes as nearly equal as possible. To obtain the required number of specimens the average grain size selected was 210 ± 30 grains/in.

In addition to the variation in grain size, a further error arose because of the bending of the Tensometer beam. This effectively caused a small non-uniform decrease in the values of the strain rates applied but the error in the extreme cases was calculated to be less than 4% and thus it could be ignored.

Tests were carried out over the range of strain rates $\dot{\varepsilon}$ available at the temperatures of 20°C, 10°C and 0°C and the natural strains ε_c which occurred prior to cleavage were measured. The results are given in the graph in Fig. 29. The graph is drawn of ε_c against log $\dot{\varepsilon}$ to give a more convenient distribution of points. The scatter of results is due to the small grain size variation and the curves at the three temperatures have been drawn as far as possible to those which would have been obtained for specimens of equal grain size. By comparing Table 5 with Fig. 29 it can be seen that the results for smaller grain sizes lie above the corresponding lines whilst those for larger grain sizes fall below them.

TABLE 5
(As illustrated in Fig.29)

Temperature T^oC.	Strain Rate $\dot{\varepsilon}$ % per sec.	$\frac{1}{d}$ grains/in.	Natural Strain to Cleavage ε_c
20	0.0106	228	0.284
20	0.0264	226	0.211
20	0.053	243	0.186
20	0.099	209	0.141
20	0.132	241	0.175
20	0.264	253	0.150
20	0.530	228	0.120
10	0.0106	232	0.193
10	0.053	230	0.127
10	0.264	240	0.101
0	0.0033	189	0.180
0	0.0053	211	0.167
0	0.0106	187	0.122
0	0.0170	198	0.120
0	0.0264	218	0.133
0	0.053	181	0.083
0	0.053	196	0.086
0	0.132	212	0.082
0	0.264	219	0.083
[illegible]	[illegible]	[illegible]	[illegible]

These results indicate that the elongations prior to cleavage ε_c in tests at fast strain rates at 20°C are of similar values to those obtained with slower strain rates at 0°C. An examination of the stress-strain curves indicated that the flow properties of approximate yield stress and strain hardening were also similar for specimens which showed similar elongations prior to fracture. It was evident that various combinations of temperature T and strain rate $\dot{\varepsilon}$ could produce similar flow and fracture conditions.

The form of the curves obtained and presented in Fig. 29 suggested that an "activation type" of process might be occurring. From the general theory of "Rate processes" the strain rate $\dot{\varepsilon}$ can be considered to be equivalent to a function of the temperature T given by an equation of the form:-

$$\dot{\varepsilon} \equiv A e^{-\frac{Q}{KT}}$$

where A is a constant, depending on the conditions

K is Boltzmann's constant

and Q is a fundamental constant for the process taking place and is known as the "Activation Energy".

It is seen that the above equation can be written:-

$$\log \dot{\varepsilon} = \log A - \frac{Q}{KT}$$

To test whether the equation was relevant to the present phenomena a plot of $\frac{1}{T}$ against $\log \dot{\varepsilon}$ was made for the condition of a constant elongation prior to fracture. Fig. 30 shows the corresponding values of $\frac{1}{T}$ and $\log \dot{\varepsilon}$ taken for the values $\varepsilon_c = 0.11$ and 0.20, from the graph of Fig. 29. For each value of ε_c the three points fall on a straight line. From the gradients of the lines an estimate can be made of the value of the "Activation Energy" Q. From the above equation, replacing K by R the gas constant to obtain Q in cals/gm. molecule it is seen that

$$Q = R \frac{\partial}{\partial(\frac{1}{T})} \left\{ \log_e \dot{\varepsilon} \right\}$$

Using the value for the mean gradient of the two lines in Fig. 30 and the known value of R = 2 cals./gm. mole/deg.C.

$$Q = 2 \times \frac{\log 10}{0.00022} = \frac{2 \times 2.3}{0.00022}$$

$$= 21 \times 10^3 \text{ cals/gm. molecule}$$

$$= 21 \text{ K.cals/gm. molecule}$$

This value cannot be regarded as being a precise value for an Activation Energy because of the numerous sources of error in its determination. The combined errors are difficult to assess, but the true value of Q can be expected to lie within the range 21 ± 5 K.cals/gm.molecule. This determination of Q is not sufficiently accurate to enable very specific suggestions to be made concerning the processes to which it refers. It may be that a number of processes are involved. Some significance may be attached, however, to the fact that Q is of a similar value to the activation energy for self diffusion. The values given in the "Metals Reference Book"(69) are:-

20.4 K.cals/gm. molecule. parallel to the c axis

31.0 " " perpendicular to the c axis

It would seem that the processes indicated by the above equations are not merely specific to the fracture process. Specimens stress-strain curves. It would seem that the processes are related to the flow properties and, because of this, to the cleavage mechanism. Ideas will be developed later of cleavage occurring as a consequence of the mode of deformation.

For a further examination of the processes involved, a greater range of strain rates would be necessary so that a more accurate value of Q could be obtained over a wider range of temperature. Nevertheless, from the present work it appears that the effects of temperature T and strain rate $\dot{\varepsilon}$ on the flow, elongation prior to cleavage and true fracture stress can be combined in a single parameter of the form $f(\dot{\varepsilon}\, e^{\frac{Q}{KT}})$ at least near the transition temperature.

(3.8) The Transition Temperature.

In previous studies of fracture many definitions have been given of the term "transition temperature". Usually, however, the term has been used in notched bar impact tests and it has been defined most often as a temperature at which a certain energy has been absorbed in fracturing by the change of fracture type. Such a transition temperature was first quantitatively reported by Agnor and Shank for zinc[62].

In the present work the transition temperature was found to be clearly defined in the tests in simple tension. Moreover the change from typical cleavage to ductile fracture was very precise and, for specimens of the same grain size and tested at the same strain rate, it occurred over a temperature range of only a few degrees. Fig.20 shows a series of typical true stress-natural strain curves for specimens of approximately constant grain size of about 250 grains/in. The elongations prior to cleavage increased markedly with increasing temperature and, as was mentioned previously, if the temperature was sufficiently high for a natural strain of 0.30 to be reached without fracture, then the specimens continued to deform and considerable necking took place, terminating in a ductile fracture which represented fracture above the transition temperature.

The effect of grain size variation was clearly apparent. For any given temperature, the smaller the grain size the more ductile the specimen. When the temperature was high enough the specimens with the smallest grain size continued to deform to give~~n~~ ductile fractures. The specimens with larger grain size, on the other hand, gave cleavage fractures at the same temperature. This was observed in the experiments at 20°C. Put another way the transition temperature was higher for specimens of larger grain size. Decrease in grain size lowered the transition temperature.

From the effects of strain rate variation already discussed it was seen that faster strain rates were equivalent to lower temperatures and vice-versa. Hence the transition temperature was raised in tests at faster strain rates. The results of the previous section gave quantitative data that "shock" or rapid stressing increases the likelihood of cleavage failure.

(4) A DISCUSSION OF EXPERIMENTAL RESULTS

To summarise, the results for which an explanation is sought are:

(1) The low value of the cleavage stress σ

(2) The existence of the sharp transition temperature.

(3) The relation $\sigma \propto \frac{1}{\sqrt{d}}$

(4) The increase of σ with increase in T.

(5) The effects of strain on fracture.

(6) The mutual relation of temperature and strain rate.

The low cleavage fracture stresses measured for zinc have been shown to be less than the theoretical value of the cohesive strength σ_m by a factor of the order of 100 to 1,000. All materials which show cleavage would appear to have similarly low fracture stresses. This must remain essentially the fundamental problem of cleavage. It is a consequence of this that cleavage requires little energy and as a result many failures of metals in service have been traced to cleavage fracture.

It was pointed out in the discussion of theories of cleavage that thermodynamic reasons have been given for the low values of cleavage fracture stresses. Calculations of the σ values from thermodynamic theories give results of about 100 to 1,000 times less than σ_m and are thus of the correct order of magnitude, but they fail completely to account for the effects of any of the variables which the present work has shown to be important. It would not be true, however, to conclude that the thermodynamic theories are completely wrong. It would rather appear that they require to be developed in much greater detail before they can be of use in guiding further experiment. The great objection to the thermodynamic theories in their present form is that they are unable, as yet, to take into account the fact that fracture is essentially a structure sensitive process and true fracture stress is therefore a structure sensitive quantity. In the present work this has been clearly brought out by the strong dependence of σ on grain size.

It appears necessary, then, to look to "flaws" in the material which might be a possible cause of cleavage. The problem is thus in many ways closely analagous to the problems of the low stresses which cause plastic flow. The Griffith Theory has been shown to be of great significance in showing how certain kinds of "flaws", namely cracks, can raise the value of the internal stress over small volumes to values equal to the theoretical cohesive strength σ_m.

It was shown how the Griffith Theory could be used directly to explain the results obtained in the present work at $-196^{\circ}C$ where the zinc fractured without overall deformation. The results were approximately numerically correct and the theory also indicated the correct $\sigma \propto \frac{1}{\sqrt{d}}$ relation as shown in Fig. 13. Nevertheless, as shown by Elliott's analysis[50] the idea that pre-existing cracks are present in a metal is untenable on theoretical grounds and the present work has also provided experimental data which would be difficult to reconcile with simple theory of pre-existing cracks. In the first place, the effects of strain on fracture are too complex to admit interpretation on simple crack mechanisms, and, secondly, the fact that σ has been found to increase with temperature T in the absence of deformation as shown in Figs. 15 and 26 is markedly against the predictions of Griffith Theory. In the present work the increase of σ with T has been determined for large grained specimens which fractured without overall deformation over the range $-196^{\circ}C$ to $-77^{\circ}C$, thus the result is independent of the validity of hypotheses concerning the effects of deformation. This would appear to be the first instance in the investigation of fracture in polycrystalline materials where it has been found that σ increases as T increases in the absence of deformation.

However, data on other materials is extremely meagre and the only other comparable investigation known is the work of Eldin and Collins[15] on a steel which they tested in tension down to $12^{\circ}K$. Their result was opposite to that now found for zinc. They found that σ decreased with increasing T in the range $12^{\circ}K$ to $61.5^{\circ}K$ where fracture occurred without reduction in area.

There are objections to the direct application of Griffith Crack Theory to pure metals since it is wrong to assume that cracks can always be present. The problem of cleavage in metals is to search for a mechanism whereby the high internal stresses can be built up to cause cracks which can be propagated under the applied stress. The problem is thus to determine the origin of the cracks which ultimately lead to fracture since they cannot be considered to exist before the stress is applied.

A promising line of approach to the problem of crack formation was indicated in the discussion of the application of dislocation theory. The theory will now be considered in further detail and reviewed in the light of the results obtained in the present work.

(4.1) <u>The Application of Dislocation Theory to Experimental Results.</u>

Elementary ideas of crack formation were indicated in Fig.4 when dislocations can become sufficiently close to coalesce. In a pure polycrystalline metal this condition is most likely to be developed at the end of a slip line, when the dislocations, moving in the slip direction under the applied stress, are halted by the grain boundary and thus a pile up and coalescence of dislocations may be expected. Further coalescence would lead to a growth of crack size and the complete opening up of the crack would then depend on whether it could grow to the critical size necessary for self-propagation which would depend on the applied stress. The critical crack size $2c$ for propagation under an applied stress σ has been previously shown to be given by the Griffith formula $\sigma = \sqrt{\frac{\alpha E}{2c}}$. Conditions must now be examined under which cracks of the critical size can be produced in metals.

The critical shear stress in many metal single crystals has been shown to be of the order of 50 gms/m.m^2 which is of the order of 1/30th ton/sq.in. Thus it might be expected that some Frank-Read sources would be brought into operation in producing dislocations in the grains of a polycrystal when the shear stress is greater than this value. The fact that the overall deformation of polycrystals is extremely small until much higher stress levels are applied indicates that the free movement of dislocations is obstructed by the grain boundaries.

In the present experiments on zinc at -196°C, though the overall deformation in the specimens was too small to be detected, many slip bands must have been formed and their progress halted at the grain boundaries before fracture occurred. If the stoppage of the slip bands caused cracks of the size of the grain diameter, these would close spontaneously until the applied stress was sufficient to cause a stress concentration at their tip of the theoretical cohesive strength σ_m so that they could then be propagated to cause fracture. Thus the criterion for fracture would be reduced to the Griffith condition. It was mentioned previously that the Griffith Theory could be used to predict the $\sigma \propto \frac{1}{\sqrt{d}}$ relation and also give the correct numerical values if a crack size $2c$ could be identified with the mean grain diameter d. The dislocation mechanism presented here indicates how cracks of the size of the grain diameter can be expected to arise.

(4.11) A Calculation from Dislocation Theory.

In Section II(5) a method was outlined for calculating the number of dislocations n which can be piled up in a length L of slip plane between the Frank-Read source and a barrier under an applied shear stress σ_s. If μ is the shear modulus and b the strength of a dislocation, the formula given was

$$n = \frac{2 L \sigma_s}{\mu b}$$

Now a more detailed calculation of the same problem has been made by Eshelly, Frank and Nabarro[70] and the positions of the dislocations along the slip direction have also been worked out. The distribution was shown to be similar to that in Fig. 5 with the density of dislocations greatest near the barrier. From the complete analysis a slightly more accurate formula for the number of dislocations n was found to be

$$n = \frac{L \sigma_s}{2 A}$$

where $A = \dfrac{\mu b}{2\pi(1-\nu)}$ for edge dislocations

ν being Poisson's Ratio.

An analysis by Koehler[71] has shown that, in addition to the large shear stresses produced by the pile up of dislocations, large tensile stresses are also produced. He calculated the tensile stress σ_i across a plane perpendicular to the slip direction and obtained a relation

$$\sigma_j = k n \sigma_s$$

where k is a constant of the order of unity.

On substituting for the value of n from the first equation a relation can be obtained for the tensile stress σ_j produced near the tip of the dislocation array under the applied shear stress σ_s. We find

$$\sigma_j = \frac{K L \sigma_s^2}{2A}$$

If this tensile stress is equal to or greater than the theoretical cohesive strength σ_m, then fracture should occur.

Thus the criterion for fracture is $\sigma_j \geqslant \sigma_m$.

$$\text{or} \quad \frac{K L \sigma_s^2}{2A} \geqslant \sigma_m.$$

Since K, A and σ_m are constants, the shear stress σ_s necessary for fracture is seen to be dependent on the length L available for the dislocation pile up, or on the grain diameter d, if the array of dislocations can be assumed to occupy the entire length of a slip line in a grain. Furthermore, since the shear stress σ_s in the most favourably orientated grains is equal to the overall applied tensile stress σ divided by $\sqrt{2}$, we obtain the relation

$$\sigma = \sqrt{2}\,\sigma_s \propto \frac{1}{\sqrt{L}} \propto \frac{1}{\sqrt{d}}$$

Thus, on dislocation theory a formula is obtained which gives the experimentally observed relationship $\sigma \propto \frac{1}{\sqrt{d}}$. The present work has provided results from which the formula may be tested numerically. The results are equally valid for specimens of all grain sizes because of the prediction of the correct relationship between σ and d.

Considering a specimen of grain size d of 0.01 cms., the shear stress σ_s in the most favourably orientated grains at fracture at -196^{o}C is of the order of 10^{9} dynes/cm^{2} as shown in Fig. 13

$$\text{Then } L \sim 10^{-2} \text{ cms.}$$

$$\sigma_s \sim 10^{9} \text{ dynes/cm}^{2}$$

$$A \sim 10^{4} \text{ dynes/cm}$$

$$K \sim 1$$

$$\frac{KL\sigma_s^{2}}{2A} = \frac{1 \times 10^{-2} \times 10^{18}}{10^{4}} = 10^{12} \text{ dynes/cm}^{2}$$

The tensile stress magnification is thus of the order of magnitude of the theoretical cohesive strength σ_m. Hence the formula provides approximately correct numerical values. To calculate the number of dislocations which are required to be piled up on the slip lines of specimens of this grain size, we have

$$n = \frac{L\sigma_s}{2A} = \frac{10^{-2} \times 10^{9}}{10^{4}} = 10^{3}$$

On the dislocation mechanism it would be necessary to cause a pile up of 1,000 dislocations between the source and grain boundary in a specimen of mean grain diameter 10^{-2} cms.

On the theory of Eshelby, Frank and Nabarro[70] a formula has also been given for the distance q between the two leading dislocations of the array.

$$q = 1.84 \frac{A}{n\sigma_s}$$

$$= 1.84 \times \frac{10^{4}}{10^{3} \times 10^{9}} \sim 10^{-8} \text{ cms.}$$

Hence for the required stress magnification to be produced the leading dislocations must coalesce.

Recently a similar theory has been applied by Petch(72) to the problem of cleavage in steel. He found that the fracture stress σ was given by an equation

$$\sigma = \sigma_o + f\,d^{-\frac{1}{2}}$$

where σ_o and d are constants.

The relation between true fracture stress σ and grain size d is thus similar to the results found in the present work for zinc, except for the additional term σ_o. A comparison of the line gradients of the curves of σ against $\frac{1}{\sqrt{d}}$ for steels and zinc indicates that the gradient for steels is about twice as great. This might be anticipated from the estimates of the theoretical cohesive strength σ_m for steels and zinc(73). Thus the formulae which have been shown to be relevant and numerically of the correct order of magnitude for zinc can be applied to the case of cleavage in steels if the σ_o term can be attributed to a frictional force which effectively reduces the value of the applied stress.

(4.12) Further Developments of Dislocation Theory

The suggested application of dislocation theory, though giving a realistic picture of the build up of high internal stresses, requires much further development before many of the phenomena can be satisfactorily understood. The calculation given above which suggests the correct relationship between σ and d as well as providing numerical values of the correct order of magnitude still remains open to criticism. The calculation has merely considered the outlines of the problem. It was assumed that the Frank-Read sources in a grain acted independently of each other and gave the number of dislocations consistent with the relaxation of elastic strain at the source. In addition the dislocations were assumed to fill completely the entire slip line of the grain and provide a back stress which exactly cancelled the applied stress at the source. Further developments of dislocation theory must be awaited before any of these assumptions can be fully justified.

For a better understanding of the process of crack formation it is desirable to know more of the dynamic properties of dislocations. Their speed of travel would be likely to have considerable influence on the coalescence necessary to cause a crack. The calculation of Eshelby, Frank and Nabarro[70] might be expected to be considerably modified if the additional influence of the dislocation momentum in piling up was considered. This would effectively increase the stress σ_i at the tip of the dislocation pile up and the calculated value of σ_i may therefore be too small. However, in giving the value of the applied shear stress σ_s in the grains it was assumed that this value was not reduced by the operation of neighbouring Frank-Read sources. In view of this the actual value of σ_s might be considerably less than the value used in the calculation. A lower value of σ_s would have the effect of reducing σ_i. Thus the two effects of dislocation momentum and of slip on neighbouring slip planes would have opposite effects on the calculation of the stress concentration σ_i but the present experimental results indicate that if a complete theoretical analysis were possible then the final result should not differ markedly from the one deduced in the preceding section.

On the proposed dislocation mechanism for cleavage two reasons can be advanced to explain the increase of true fracture stress σ with the temperature T. In the first instance, if the pile up of dislocations at the boundary occurred sufficiently slowly, then at the higher temperatures the increased mobility of the dislocations might allow them to diffuse out of their slip plane into the grains and the grain boundaries. Hence it would be necessary to apply a higher overall stress σ before the pile up was large enough to provide the stress concentration necessary for cleavage. A second reason is that increased stress relaxation from neighbouring sources is to be expected at the higher temperatures. Hence, the conditions are less favourable for crack growth. This is a consequence of the fact that the critical shear stress for yielding decreases with increasing temperature.(74) If the operation of the sources is affected by nitrogen then the temperature effect on yielding is likely to be very large. At the lower temperatures the sources locked by nitrogen atmospheres will be unable to act to relieve the stress concentrations, but with increasing temperature increasing ease of operation will be possible. The observations described in the Appendix indicate that the zinc contains some nitrogen.

In addition the above reasoning indicates why flow would be expected to occur at the higher temperatures. A general yielding of the grains would take place before an internal stress concentration of the magnitude required for cleavage could be built up. It is of some interest that the flow and fracture properties have been found to obey an activation law. The activation energy was found to be 21 ± 5 K.cals/gm.mole and about the same value as that found for self diffusion(69). It would appear that the build up of internal stress and crack formation at the higher temperatures is only possible at sufficiently high strain rates where the time allowed for self-diffusion is short. A complete calculation from dislocation theory must take this effect into account.

A direct interpretation can now be given of the existence of the transition temperature. At the higher temperatures the mobility of dislocations in moving out of their slip planes and the stress relief from the yielding in neighbouring slip planes and adjacent grains caused by the decrease of yield stress with increasing temperature would prevent a dislocation pile up from producing a crack of the size necessary for cleavage. An additional factor is that considerable deformation takes place at the higher temperatures. It would also seem that after heavy deformation slip lines of sufficient length do not exist on which the number of dislocations necessary for cleavage could be piled up. Hence continued deformation would occur without fracture.

The observed effects of strain on true fracture stress are difficult to explain at present. The rather complex effects of prestrain on σ shown in Figs. 18 and 19 may possibly only occur in this form for zinc, though details for other metals are extremely meagre. The only comparable results have been obtained for steels[66] but they showed a continuous rise of true fracture stress with the amount of prestraining. The curves for the effect of prestrain where the zinc is ductile on the σ value at $-196^{\circ}C$ must be explained in terms of the structures obtained after varying amounts of deformation. The effects are clearly too complex to admit of interpretation on a simple theory of pre-existing cracks. On dislocation theory the effect of prestrains in the range 0.05 to 0.25 in decreasing the σ value at $-196^{\circ}C$ must be explained either by assuming high residual stresses produced during the prestraining or by assuming that the cold-worked structure of the material allows an easier growth of cracks of the size required to propagate a cleavage fracture.

In the present work twinning was shown to be associated with fractures which occurred after small deformations. However, after larger deformations the cleavage facets were relatively twin-free. After about 0.07 strain prior to fracture an increasing number of grains in the fracture surface were free from twins and it might be considered that the decrease in σ caused by strain was associated with the lack of twinning at fracture. Apart from this observation no further effects were found of the connection between twinning and fracture. The whole problem of the effect of strain on true fracture stress in zinc is likely to be very complex in interpretation. The marked elastic anisotropy of the individual crystals in the specimens and their modes of deformation by slip, twinning, kinking and grain boundary slip must all be taken into account.

In conclusion, it appears that the proposed dislocation mechanism for the build up of large internal stresses leading to cracks which cause cleavage can be successfully applied to explain a number of phenomena in the cleavage of polycrystalline zinc. The explanation of further details requires further developments from dislocation theory.

V - EXPERIMENTS ON THE FRACTURE OF PURE POLYCRYSTALLINE MAGNESIUM IN SIMPLE TENSION

(1) INTRODUCTION

In view of the results obtained for the cleavage of pure polycrystalline zinc, a point of some importance is to determine whether similar properties can be found in metals having a similar crystallographic structure. The metals most closely related to zinc in this respect are magnesium and cadmium. All three are of hexagonal close packed structure and have similar plastic properties.

A survey of the literature indicated that cleavage has been observed in single crystals of magnesium. The (0001) $(10\bar{1}1)$ $(10\bar{1}2)$ $(10\bar{1}0)$ have all been suggested as possible cleavage planes[1] though it is probable that twinning obscured the true identity of the planes and it appears that the basal plane (0001) is the most likely cleavage plane. However, there appear to have been no investigations of the tensile properties of pure polycrystalline magnesium with the object of studying the factors affecting the cleavage fracture. In the present work the effects of temperature and grain size variation on cleavage were studied to determine whether these could be correlated with the results obtained for the fracture of polycrystalline zinc.

A continued survey of the literature did not reveal any instances of the observation of cleavage in cadmium. Studies of single crystals[75] indicated that shear fractures occurred after considerable deformation. Reports of tensile tests on the polycrystalline metal[76] likewise indicated high ductility and a ductile fracture even at $-183^{o}C$. Cadmium has also been found to remain ductile in a notch impact bend test at $-196^{o}C$[77]. A study of the fracture properties of cadmium was not made in the present work since there is ample evidence that cleavage fractures cannot be obtained, at any rate above $-196^{o}C$.

A comparison of the crystallographic and plastic properties of zinc, magnesium and cadmium can be obtained from Table 6.

TABLE 6

	Zn.	Mg.	Cd.
Structure	all hexagonal close packed		
$\frac{c}{a}$ axial ratio	1.856	1.624	1.886
Slip plane	(0001)	(0001)	(0001)
Slip direction	$[11\bar{2}0]$	$[11\bar{2}0]$	$[11\bar{2}0]$
Twin plane	$(10\bar{1}2)$	$(10\bar{1}2)$	$(10\bar{1}2)$
Young's Mod. (kg/mm^2) along the c axis at angle to c axis	 3,560 12,630 at 70^{o}	 5,140 4,370 at 53^{o}	 2,880 8,300 at 90^{o}
Melting point	$419^{o}C$	$650^{o}C$	$321^{o}C$
Cleavage planes	(0001)	(0001)$(10\bar{1}0)$ $(10\bar{1}2)$$(10\bar{1}1)$	None

The Table shows the similarity of plastic properties. On a dislocation mechanism of cleavage it would be expected that the three metals should also show a similarity of cleavage properties. The investigations on magnesium were carried out to determine whether this was the case in comparing the results with those obtained for zinc.

(2) MATERIAL AND SPECIMEN PREPARATION

The magnesium was obtained in the form of extruded rods $\frac{7}{8}$ths. inches diameter from "Magnesium Elektron Ltd." It was of 99.95% purity. Analysis gave the chief impurities:-

Zn.	Al.	Si.	Cn.	Mn.	Fe.	Sn.	Ni.
%	%	%	%	%	%	%	%
0.05	0.05	0.03	0.01	0.005	0.002	0.03	0.04

Tensile specimens were cut from the extruded rods and machined to the dimensions shown in Fig. 10 . The final polish of the gauge length was carried out using 000 emery paper cooled with paraffin.

It was necessary to obtain a good polish on the ends of the specimens in order to count the grain size. A number of methods of polishing were initially used, but the method giving most satisfactory results was a simple "chemical polish" as recommended by Kehl[63]. The polishing solution was 8% nitric acid in alcohol. The specimens were first polished on papers down to the grading 400C cooled by running water. They were then placed in the polishing solution for about 30 secs. and best results were obtained when the specimen was slightly agitated. The specimens were not etched since a reasonably good grain contrast was obtained when they were observed under polarised light. The grains were found to be heavily twinned due to the machining. To remove the surface grains the specimens were alternately polished on the 400 C papers and dipped in the chemical polish until relatively twin-free grains could be viewed and a grain size count made under polarised light.

The method of grain size counting was similar to that previously described for zinc and the number of grains intersecting a line of known length were counted. The grain size across any given cross section of the extruded magnesium rods was not so uniform as that found for the zinc. The small grain magnesium specimens used in the experiments had grain sizes within the range 300 $\pm$ 50 grains/in.

(3) <u>EXPERIMENTAL RESULTS</u>

(3.1) <u>The Variation of True Fracture Stress with Temperature</u>

A series of specimens of approximately equal grain size chosen from the extruded rods were tested in tension between -196°C and 70°C. The method of testing and the temperature bath and apparatus used were the same as those described in the zinc experiments. The results are shown in Fig. 31, indicated by circles for a grain size of 300 $\pm$ 50 grains/in. Cleavage occurred throughout the range of temperature. The cleavage facets in all the specimens were distorted and stepped by twinning. The brightness and reflectivity of the cleavage facets diminished rapidly on exposure to air.

Stress-strain curves showed that deformation had occurred in the specimens prior to fracture at all temperatures of test. The elongation prior to cleavage in shown in Fig.32. Even at $-196^{\circ}C$ the specimens showed 0.04 ductility but the ductility did not increase with increasing temperature up to about $0^{\circ}C$. Similarly a variation of strain rate from 1 to 1/500 th % per sec. did not affect the ductility or true fracture stress. Above $20^{\circ}C$ the fractures were found to be partly intergranular and partly cleavage. Some small cracks were also found on the gauge length and between the grain boundaries.

(3.2) The Variation of True Fracture Stress with Grain Size

A point of some significance was to determine the dependence of true fracture stress on grain size since a clear relationship was found in the experiments on zinc. The extruded rods of magnesium did not show a marked variation of grain size along their length. To obtain the required variation it was necessary to give annealing treatments to produce grain growth. Since magnesium is well known to be easily oxidised in air the specimens were annealed in vacuo for 2 hours at $550^{\circ}C$.

For the annealing treatments the specimens were each placed in pyrex tubing of 1 in. external diameter. One end was sealed off and the other end drawn down to a narrow neck. A further neck was also made at a short distance along the tubing. The neck at the end of the tubing was connected to a backing pump and when the system was evacuated the section containing the specimens was sealed off without allowing the admission of air.

The ends of the specimens were polished and the grain size was found to lie within the range 40 $\pm$ 10 grains per in.

Tests were carried out at $-196^{\circ}C$, $-77^{\circ}C$ and $20^{\circ}C$ and the results are shown by crosses on Fig. 32. The ductility prior to fracture in each case was about 0.03. The results indicate that a specimen of large grain size is slightly stronger at $-196^{\circ}C$. There appears to be a steady decrease of true fracture stress with increasing temperature for the specimens of large grain size. At $20^{\circ}C$ some intergranular fracture also occurred. The variation of grain size is seen to have relatively little influence on the cleavage properties of pure magnesium. No correlation is possible between the cleavage characteristics of magnesium and zinc.

(4) CONCLUSIONS

From the present investigation of pure polycrystalline magnesium it is apparent that the factors affecting its cleavage are very different from those in the case of zinc.

A low cleavage stress was again found but it was not strongly dependent on variation of grain size, temperature or strain rate. On an elementary dislocation mechanism of cleavage it would appear difficult to understand the great difference in properties of the hexagonal metals. As shown in Table 6 the chief difference between magnesium and zinc lies in the much greater anisotropy of the zinc crystal in the elastic properties and in the c/a axial ratio which is greater than that which would correspond to perfect close packing. It may be that these differences must be considered in a complete analysis of the crack formation problem.

From Table 6, cadmium is shown to be more nearly akin to zinc in the anisotropy of the crystal as well as having closely similar plastic properties. It is still possible that cadmium could possess similar cleavage properties to zinc if tests could be made at temperatures considerably lower than -196°C.

VI - SOME GENERAL CONCLUSIONS FROM THE PRESENT SERIES OF EXPERIMENTS

The present experiments and the review of previous work have shown a considerable diversity of the effects of a number of variables on cleavage.

In view of the well-defined cleavages which have been reported in α-iron, tungsten and molybdenum, a similar cleavage might have been anticipated in β-brass. Moreover, β-brass might have been expected to be an ideal material on which to study the mechanism of cleavage in a pure body centred cubic metal, since α-iron of the required purity is difficult to obtain and pure tungsten and molybdenum in sufficient bulk are not readily available. The cleavage of body centred cubic metals remains an important practical problem because of the necessity to eliminate cleavage failure in steels. The influence of a number of alloying elements in reducing the susceptibility to cleavage would still appear difficult to understand theoretically. The present investigations of β-brasses down to $-196^{\circ}C$ did not give any indication of cleavage failure and thus no characteristics of cleavage could be revealed in the experiments on this material.

The three metals zinc, magnesium and cadmium of hexagonal structure have been shown to have very different cleavage properties. A survey of the literature indicated that cadmium remains ductile even in notched bar impact tests down to $-196^{\circ}C$. The present investigations showed that pure polycrystalline magnesium gave cleavage fractures in simple tension after a short elongation. The ductility and true fracture stress were relatively unaffected by variation of grain size, temperature and strain rate. This was in marked contrast to the effects of these variables on zinc. The results obtained for zinc were interpreted on the basis of a dislocation mechanism for the build up of high internal stresses leading to crack formation. The theory was shown to be capable of interpreting many of the phenomena but it was not nearly sufficiently developed to account for the differences in the cleavage characteristics of the different metals. However, the proposed theory is capable of considerable flexibility which would be necessary to explain such a diversity of phenomena. Further advances in dislocation theory must be awaited before a more satisfactory theory of the cleavage process can be developed.

A point of interest is that the recent experiments of Petch[72] on steels have revealed a relationship between the true fracture and grain size similar to the one obtained for zinc in the present experiments. It would seem desirable to determine whether this relationship has a wider range of application extending to other materials which show cleavage fractures. However, the present investigations have shown that the relationship does not hold for pure magnesium.

The explanation of the true fracture stress-grain size relation on the proposed dislocation theory of cleavage would further indicate that if some means could be obtained of preventing a pile up of dislocations along the complete length of a grain diameter then an increase in true fracture stress and an increased ductility would result. The investigations of Kenneford[78] on the properties of extruded zinc base alloys would seem to confirm this. He found that the addition of 2% aluminium to zinc increased the ductility prior to fracture as well as increasing the yield stress and true fracture stress if the alloy was quenched and tempered to obtain spheroidisation of the aluminium particles. Thus the suggested theory indicates a means of obtaining better properties in metals which show cleavage fracture. A further confirmation arises from the experiments of Ripling and Baldwin on zinc and steels[68][79] in which they found a lowering of the transition temperature by previous heavy cold-working at a temperature where the metals were ductile. This can be explained by the disorientation produced in the grains effectively reducing the lengths of the slip lines available for the piling up of dislocations and so preventing a stress concentration of the magnitude which causes a crack leading to cleavage.

The present work has shown the very close relationship which exists between fracture and plastic flow. Plastic flow occurs by dislocation creation and motion under the applied stress causing slip and other forms of deformation in one grain leading to flow in neighbouring grains and throughout the specimen. Fracture, on the other hand, is caused when the dislocations can be held by a barrier and pile up under the applied stress to cause a crack which grows to the critical size for cleavage before stress relief can occur. When the process of cleavage is considered in this way the hypotheses of Ludwik[12] are seen to be erroneous. He considered that fracture occurred if the brittle strength B was less than the yield stress Y. Conversely, if Y was greater than B the metal was ductile. On Ludwik's theory, factors increasing the yield stress Y would tend to promote brittleness. On the present theory some yielding, or dislocation movement, is always necessary before fracture occurs. Hence factors tending to increase the yield stress can also increase the true fracture stress.

The dislocation theory of cleavage can also provide an interpretation of the phenomenon of notch-brittleness which does not depend on the Orowan theory[13] of increase of effective yield stress at the notch. The strain rate at a notch, caused by the stress concentration at its root, is likely to be very much greater than the overall strain rate in the specimen. Thus there is less time for stress relief in the dislocation pile up and conditions are more favourable for crack formation.

The dislocation mechanism of cleavage presented here leaves many questions unanswered. Nevertheless its application to the present experimental results obtained for zinc indicate that the theoretical approach to the problem is on the correct lines. The problems of cleavage have been shown to be closely analagous to those of plastic flow. The further development of cleavage theory must await further advances in the general theory of dislocations. Theoretical problems requiring solution are:

The growth of cracks in a crystal lattice by coalescence of dislocations;

The mode of operation and interaction effects of Frank-Read sources;

The speed of travel of dislocations and the effect of their momentum in crack formation;

The structure of the cold-worked lattice and the build-up of internal stress concentrations;

The effect of solute atoms in inhibiting stress relief;

The distribution of fine precipitate particles on dislocation pile up;

The possible creation and positions of sessile dislocations in reducing the length available for pile up;

and no doubt many more problems will require solution before the problems of cleavage can be adequately understood.

Fundamental experimental data on the characteristics of cleavage is still meagre. For polycrystalline materials there is a need for more true fracture stress measurements, preferably in simple tension at low temperatures where the σ values can be measured directly. Studies of the effects of grain size variation, temperature, strain and strain rate should also prove useful. The effects of alloying elements and heat treatment should be of interest. There is also much scope for studies of single crystal cleavage. Metallographic studies of cleavage facets and the effects of orientation, temperature and alloying elements on the fracture stress can all be expected to give results of importance to aid in the development of a complete theory.

It is concluded that dislocation theory can be developed to account for large internal tensile stresses which can arise to cause cleavage fracture. The theory has been shown to be capable of explaining a number of the properties of cleavage but further developments of the theory are required before many of the phenomena can be fully understood.

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A P P E N D I X

THE STRESS - STRAIN CURVE AT 50°C.

The specimens prestrained at 50°C prior to test at -196°C gave stress - strain curves at 50°C similar to the one shown in Fig. 33. The specimens were elongated at a constant strain rate of 0.053% per sec. and the stress measurements indicated that discontinuous yielding was taking place. It would appear that this phenomenon was associated with solute atmospheres of nitrogen in anchoring the dislocations. Cottrell and Wain[80] have shown that the presence of nitrogen in single crystals of zinc causes a yield point with similar properties to those obtained in mild steels and in some other materials. However, no yield points were observed in polycrystalline zinc at low temperatures. Strain ageing treatments were carried out but still no low temperature yield point could be developed.

The present observations at 50°C might be regarded as an example of "blue-brittleness" analagous to the effect found for steels about 300°C in normal tensile tests.

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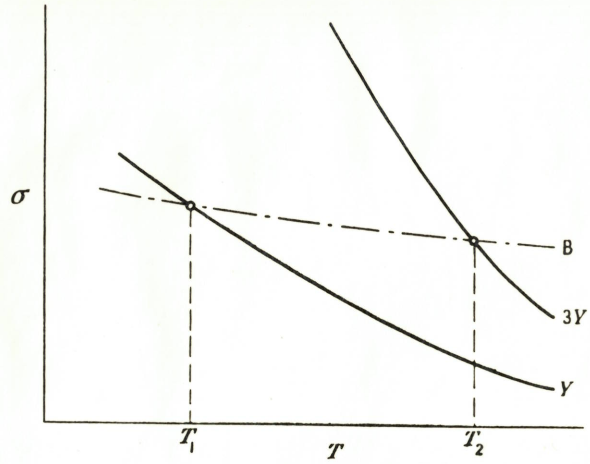
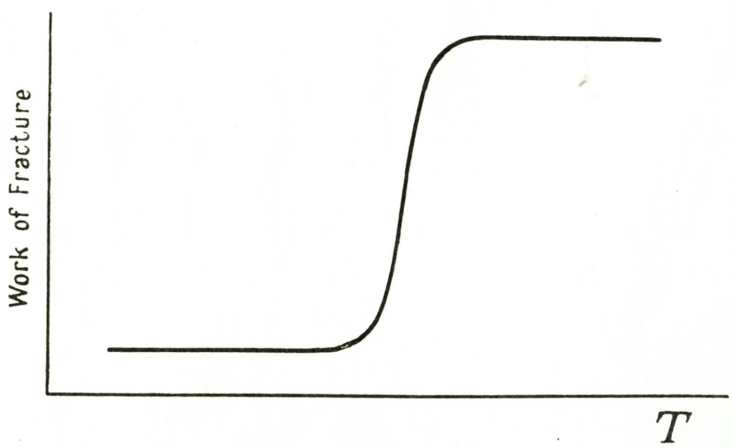


Explanation of the transition temperature. Regions of full brittleness (O to T_1), notch brittleness (T_1 to T_2), and full ductility (above T_2).

Fig. 1

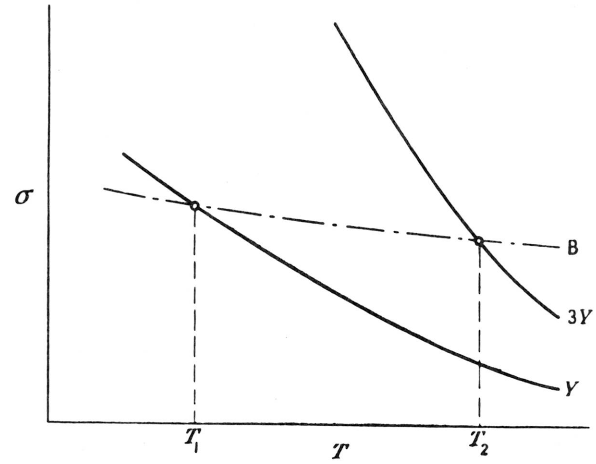
(after Orowan)



The transition temperature: the "notch impact value" (work of fracture) as a function of the temperature.

Fig. 2

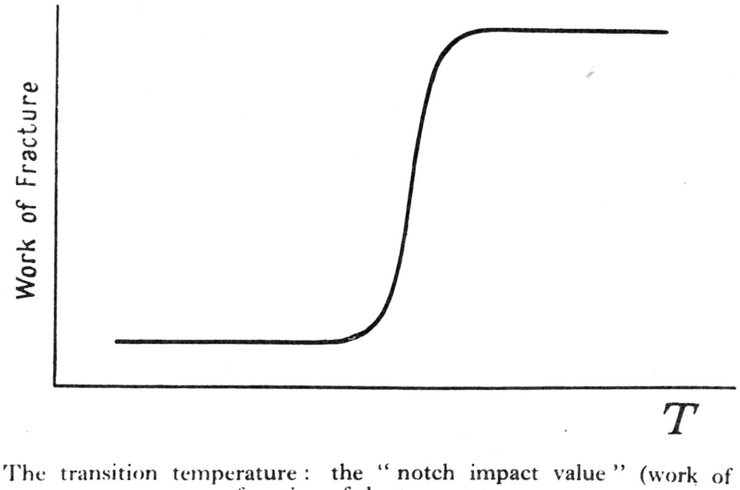
(after Orowan)



Explanation of the transition temperature. Regions of full brittleness (O to T_1), notch brittleness (T_1 to T_2), and full ductility (above T_2).

Fig. 1

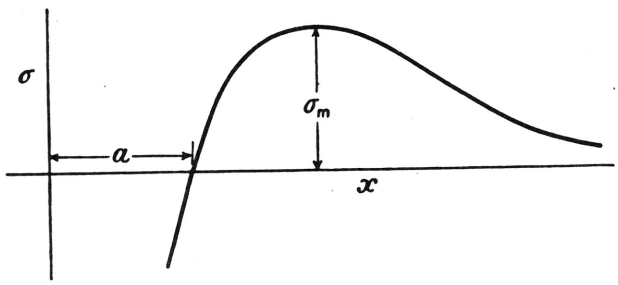
(after Orowan)



The transition temperature: the "notch impact value" (work of fracture) as a function of the temperature.

Fig. 2

(after Orowan)



Cohesive forces as a function of the interatomic spacing.

Fig. 3

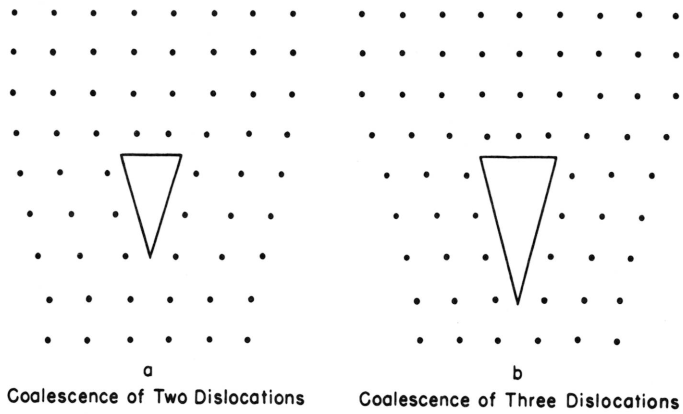


Fig. 4

Crack formation by dislocations
(after Zener).

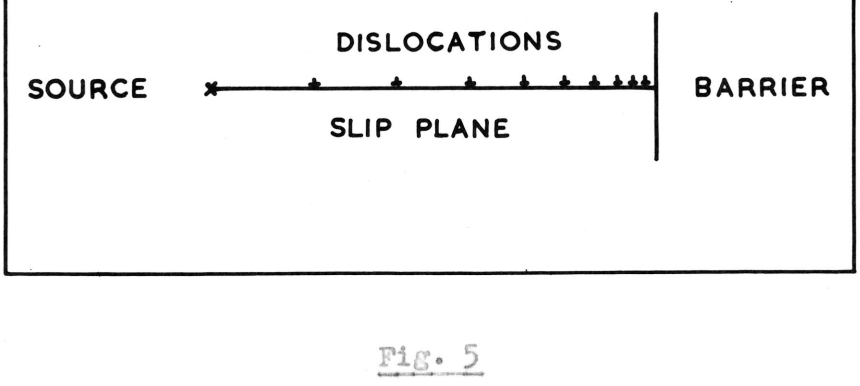


Fig. 5

An array of dislocations, piled
up in a slip plane.

Fig. 6 The central portion of an electro-polished β-brass Charpy specimen before fracture.

x 4.

Fig. 7 The same specimen after fracture.

x 4

Fig. 8 An "end-on" position showing the fracture surface of the specimen.

x 4

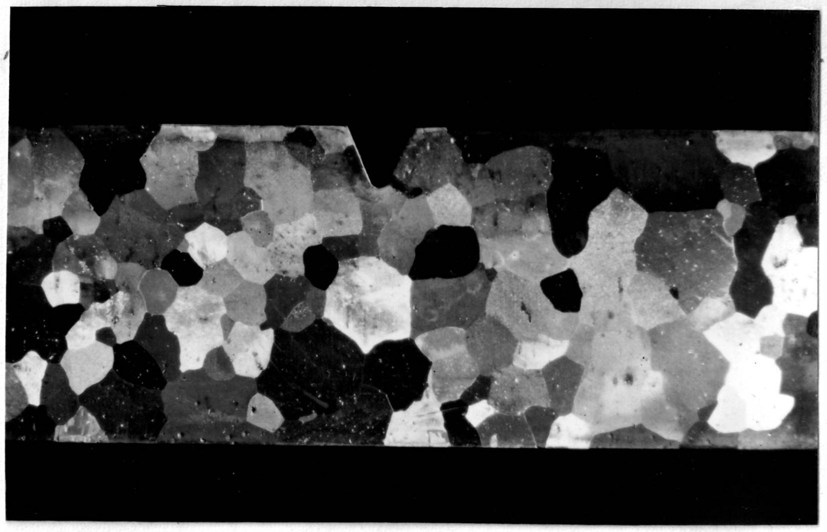


Fig. 6

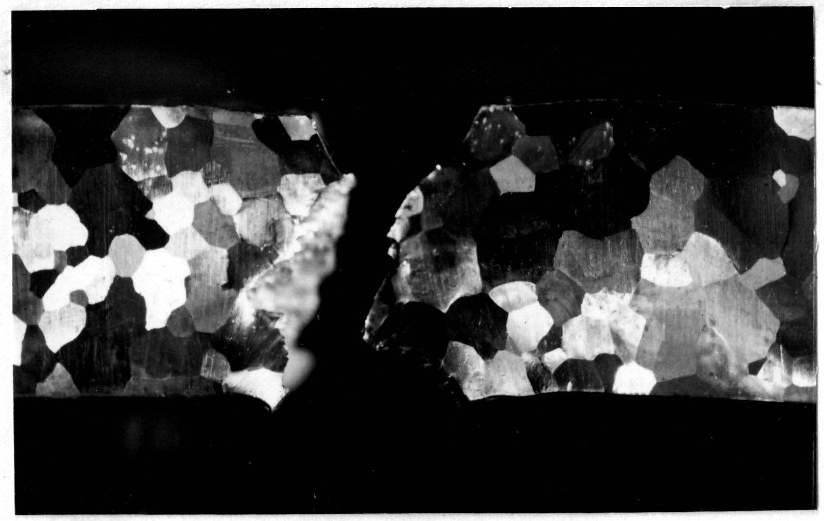
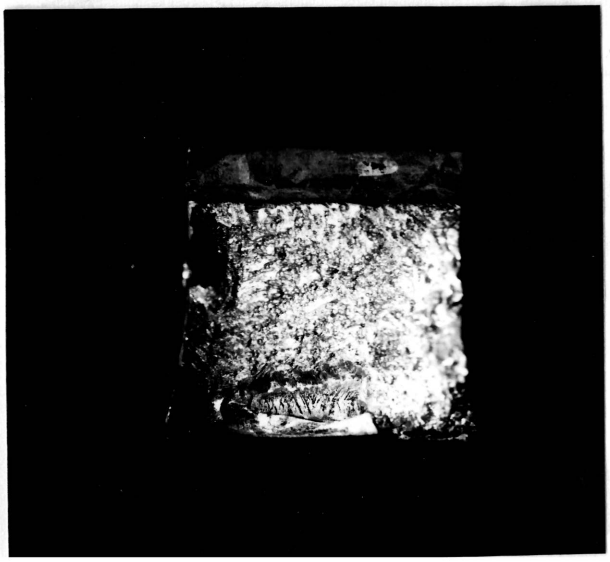


Fig. 7



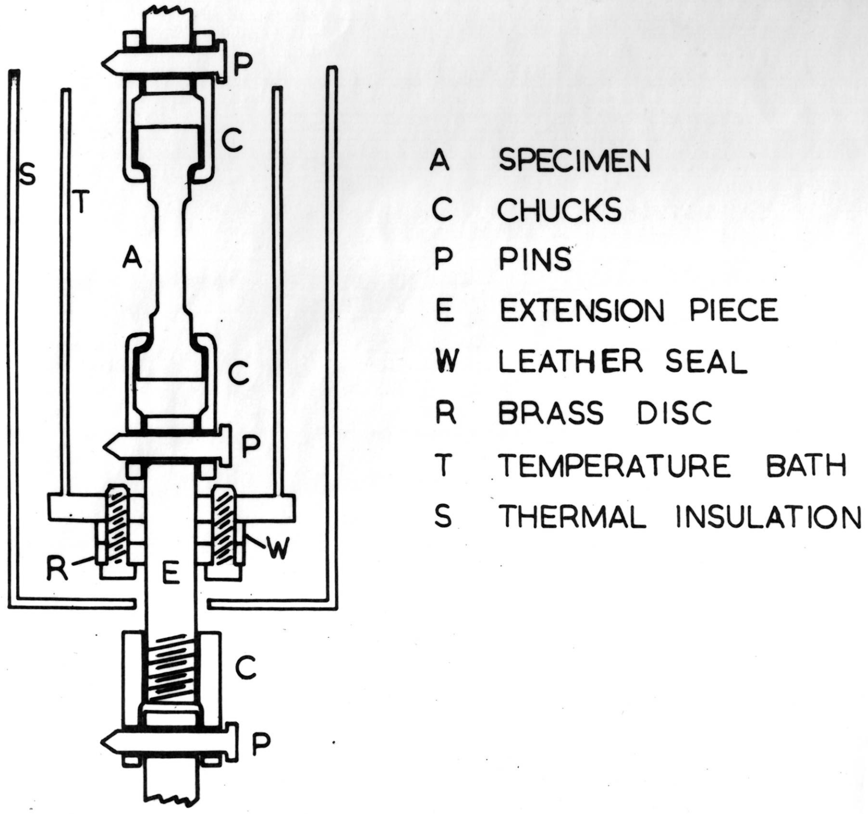


Fig. 9

The Tensile Specimen Mounting and Temperature Bath.

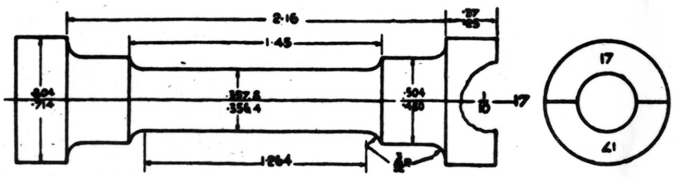


Fig. 10

The Tensile Specimen dimensions.

Fig. 11 The cleavage facet of a zinc specimen fractured at $-196^{\circ}C$.

x 200

Fig. 12 The gauge length of the specimen, electropolished after fracture. Some twins are present near the fracture surface S though the remainder of the gauge length is twin-free.

Fig.11

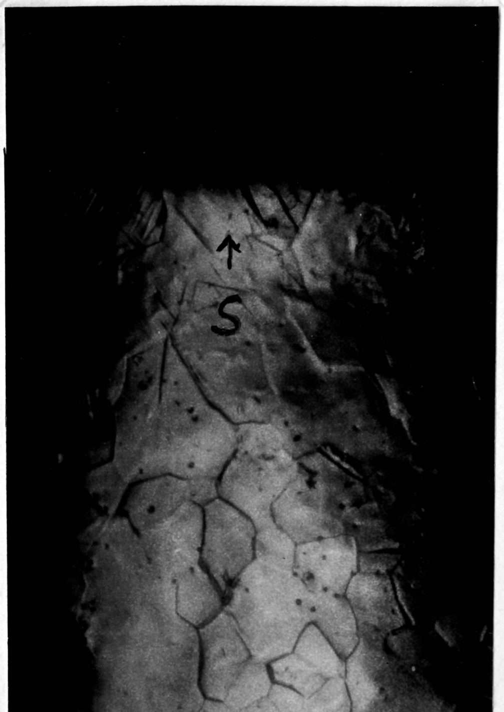


Fig.12

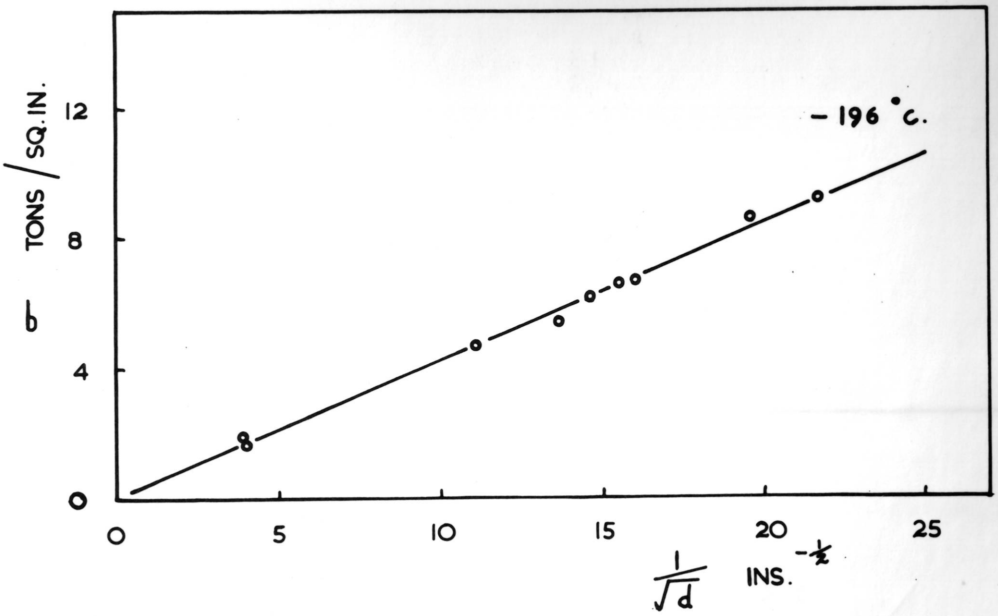


Fig. 13

The relation between true fracture stress σ at −196°C and mean grain diameter d.

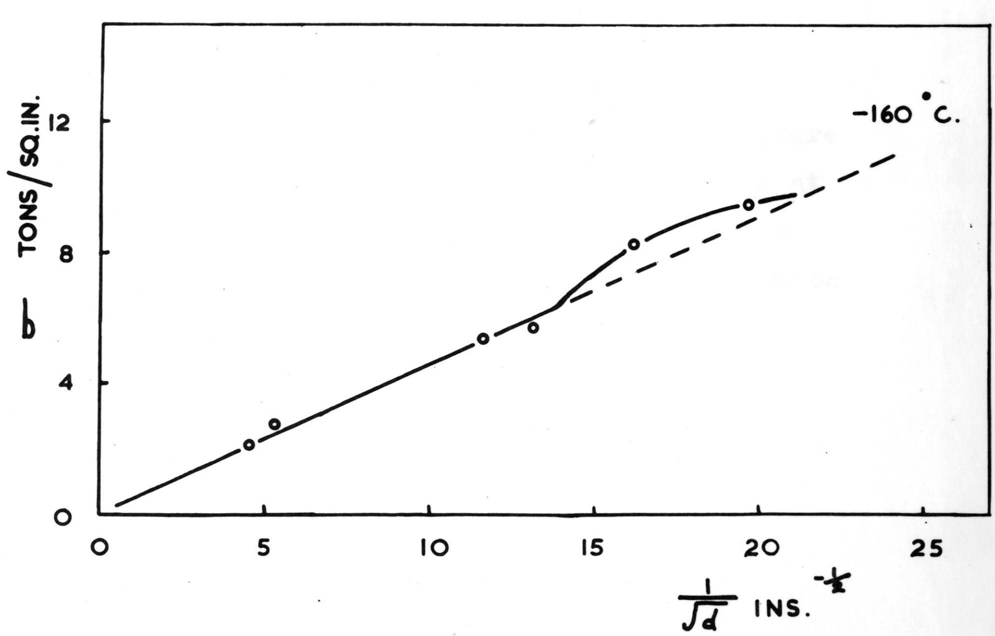


Fig. 14

The relation between true fracture stress

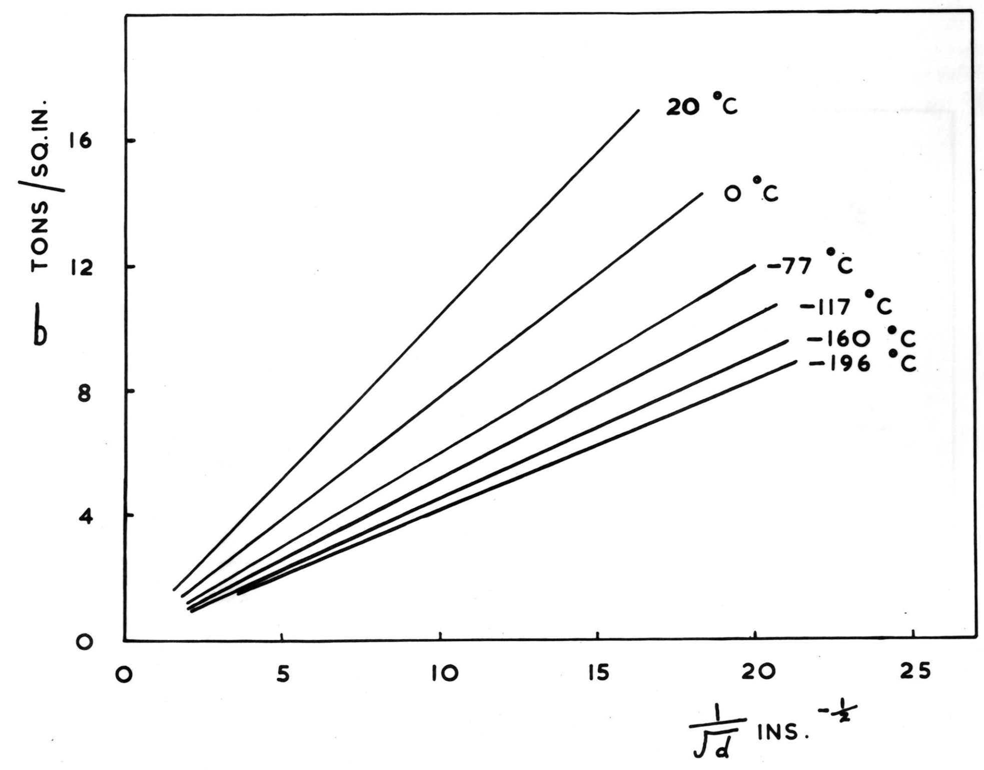


Fig. 15

The relation between true fracture stress and mean grain diameter at a series of temperatures when a correction is made for deformation occurring prior to fracture.

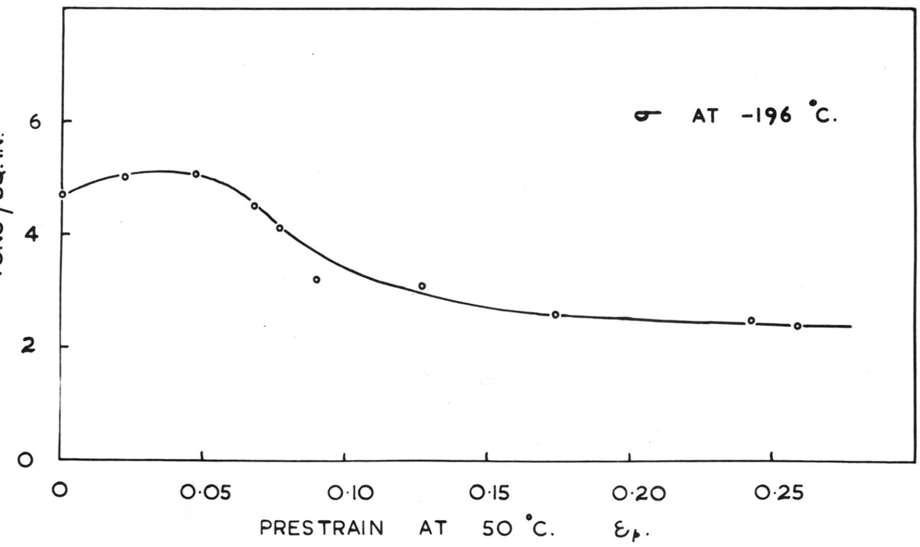


Fig. 16

The variation of true fracture stress at $-196^{\circ}C$ of specimens of nearly equal grain size of about 120 grains/in. with prestrain ε_p given at $50^{\circ}C$.

<u>Fig. 17</u> A cleavage facet of a specimen fractured at $-196^{\circ}C$ after a prestrain of 0.174 given at $50^{\circ}C$. "River-like structures" are seen. There are no striations due to twinning which were shown in Fig. 11.

x 250

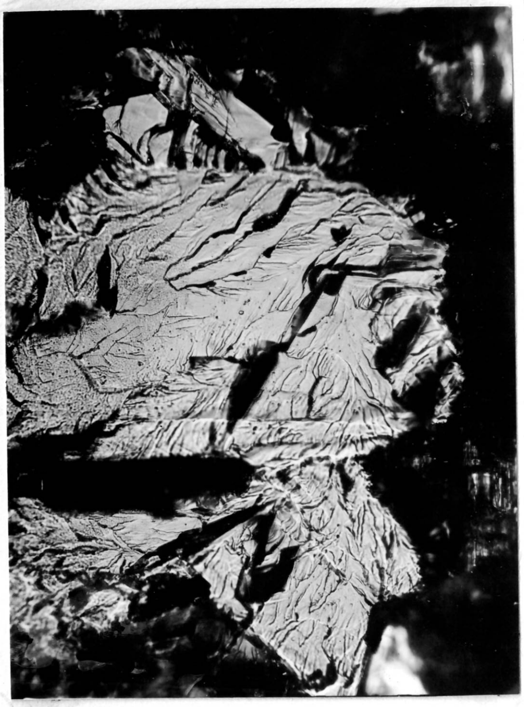


Fig.17

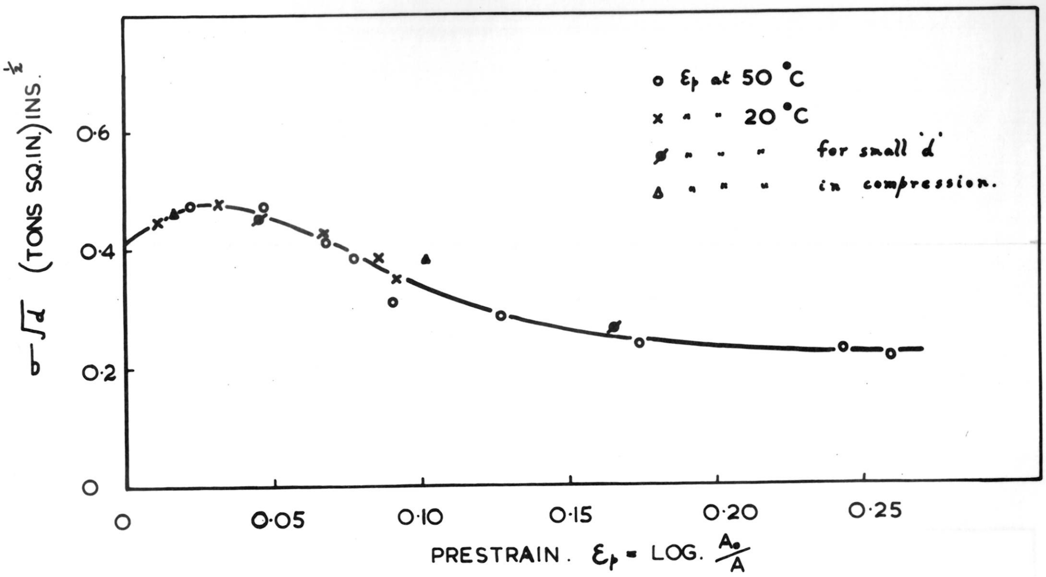


Fig. 18

The effect of prestrain on true fracture stress at -196°C for specimens of various grain sizes.

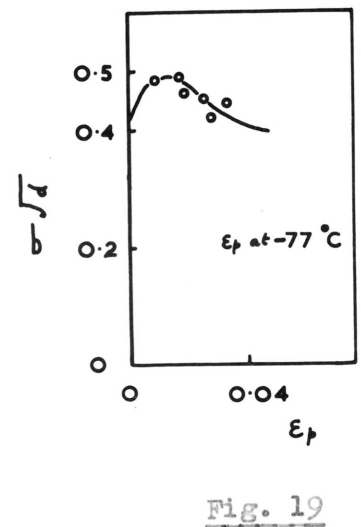


Fig. 19

The effect of prestrain at -77°C on true fracture stress at -196°C.

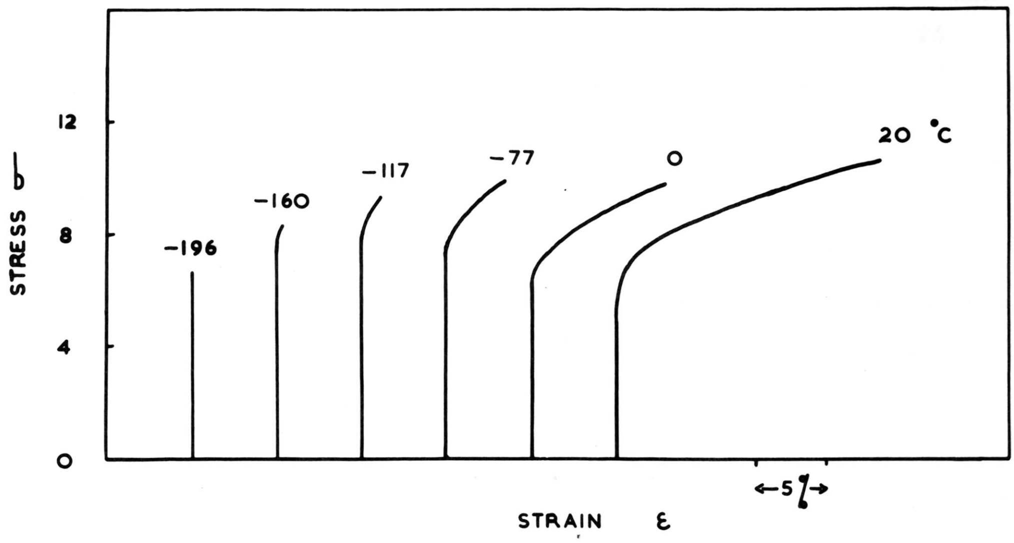


Fig. 20

True Stress Strain curves to fracture at various temperatures for specimens of nearly equal grain size of about 250 grains/in.

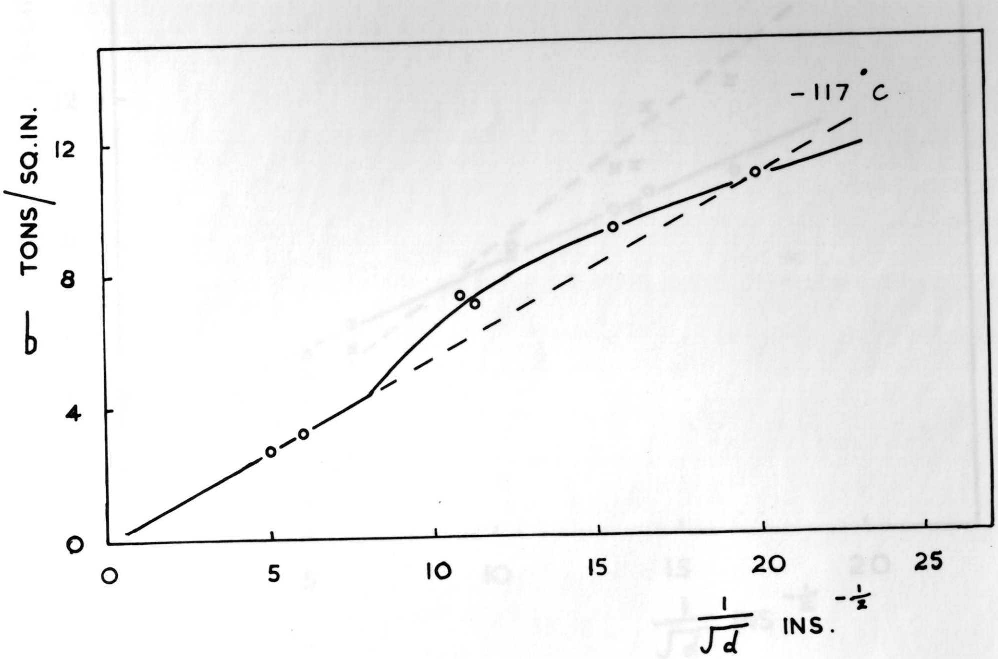


Fig. 21

The Relation between true fracture stress and mean grain diameter at -117°C. Only the two specimens of largest grain size fractured without measurable overall deformation.

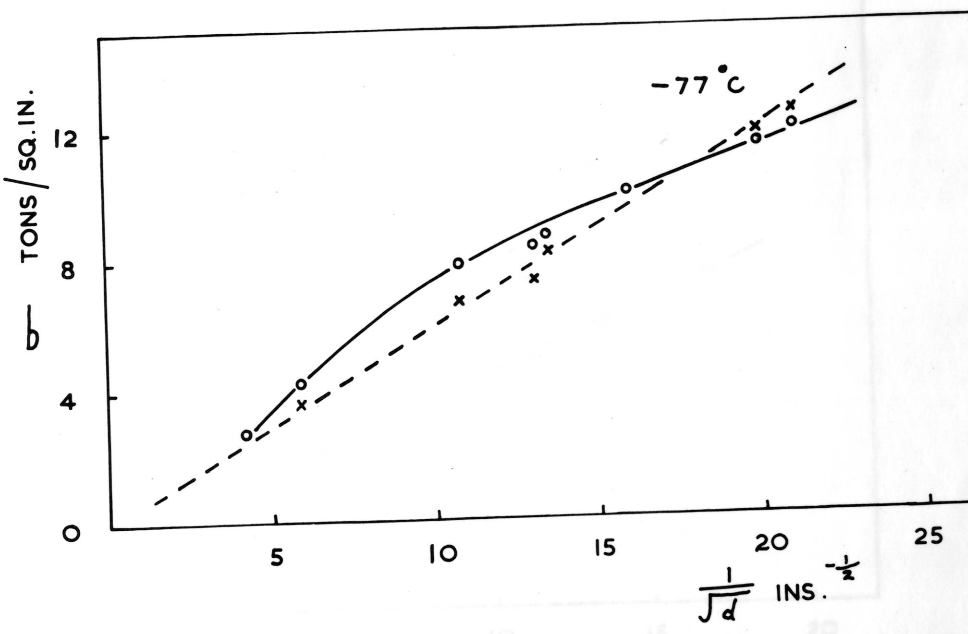


Fig. 22

The Relation between true fracture stress and mean grain diameter at -77°C. The

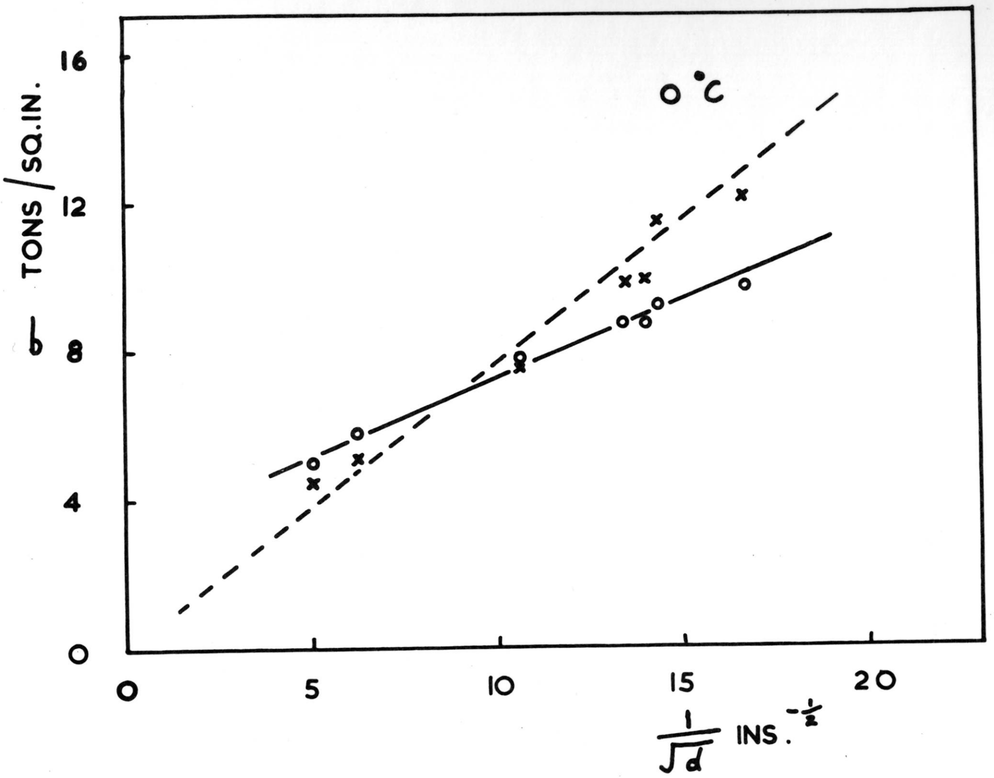


Fig. 23

The Relation between true fracture stress and mean grain diameter at 0°C. Measured σ values are shown by circles. Values corrected for deformation (from Fig.18) are shown by crosses.

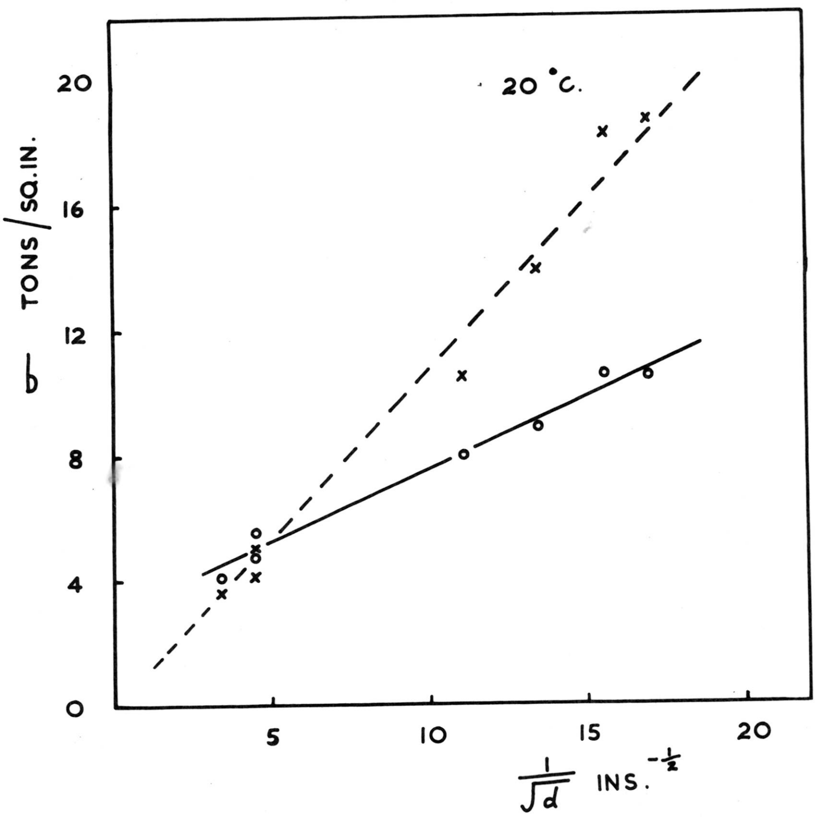


Fig.24

Fig. 25 Tensile specimens.

Showing a specimen before fracture, a specimen giving a ductile fracture above the transition temperature, and two specimens with cleavage fractures after differing amounts of deformation.

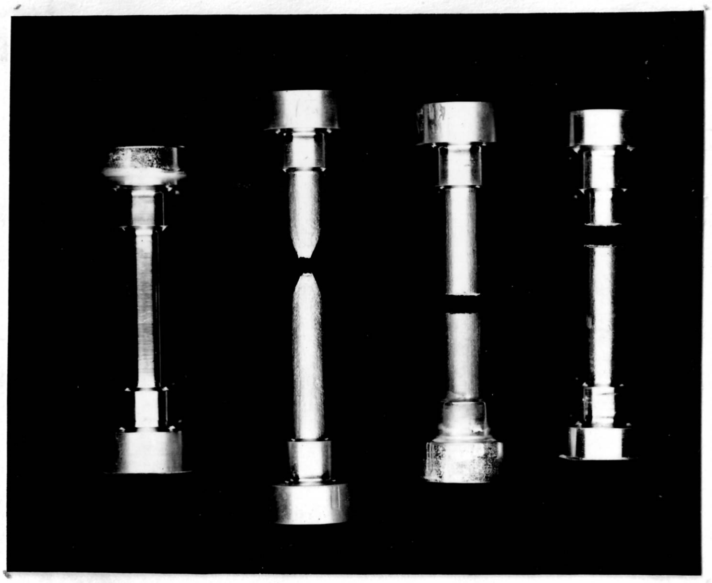


Fig. 25

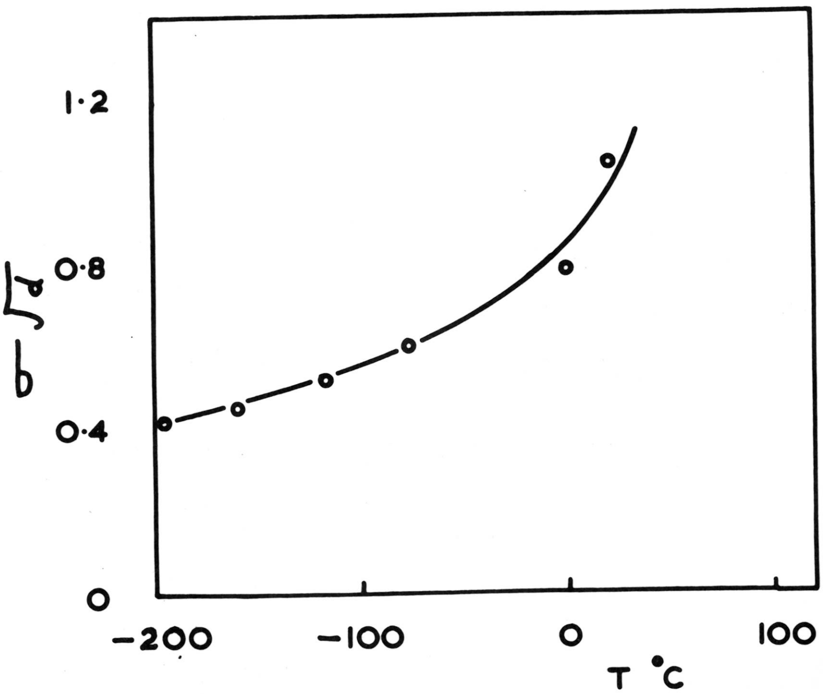


Fig. 26

The variation of true fracture stress, corrected for deformation, with temperature, taken from the line gradients of Fig. 15.

Fig. 27 A cleavage facet of a specimen which fractured at 20°C after undergoing 0.175 natural strain prior to fracture. This is comparable with Fig. 17, from a specimen which was given a similar strain at 50°C and fractured at -196°C.

x 250

Fig. 28 A specimen fractured at -196°C after heavy prestrain at 20°C. The cleavage fracture has occurred outside the neck.

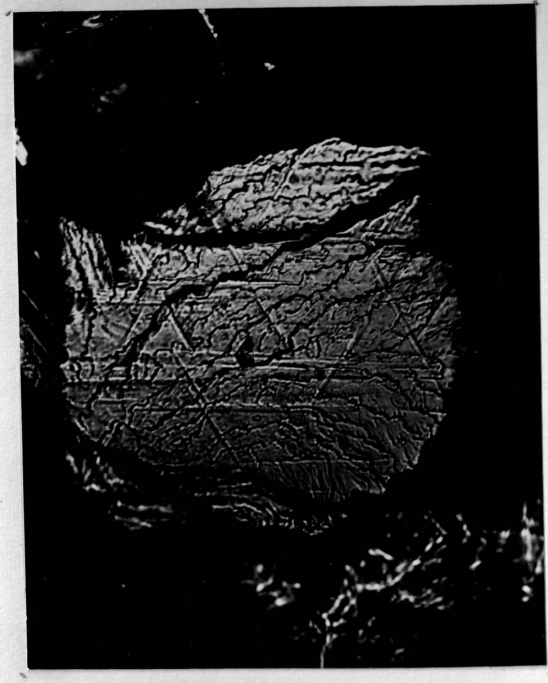


Fig.27

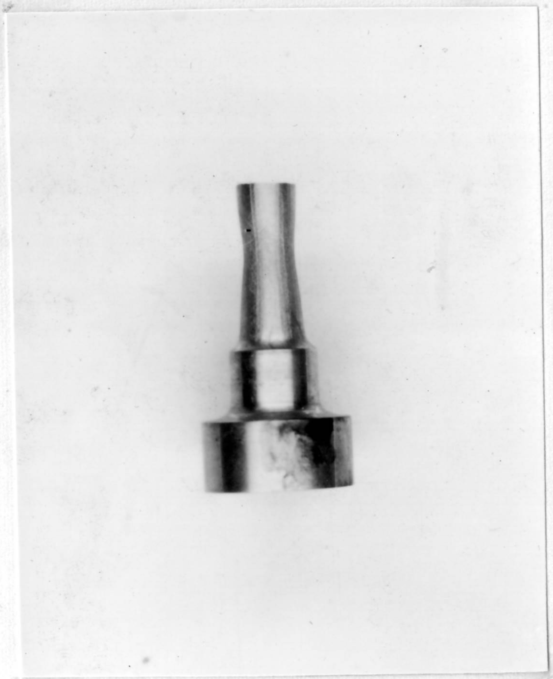


Fig.28

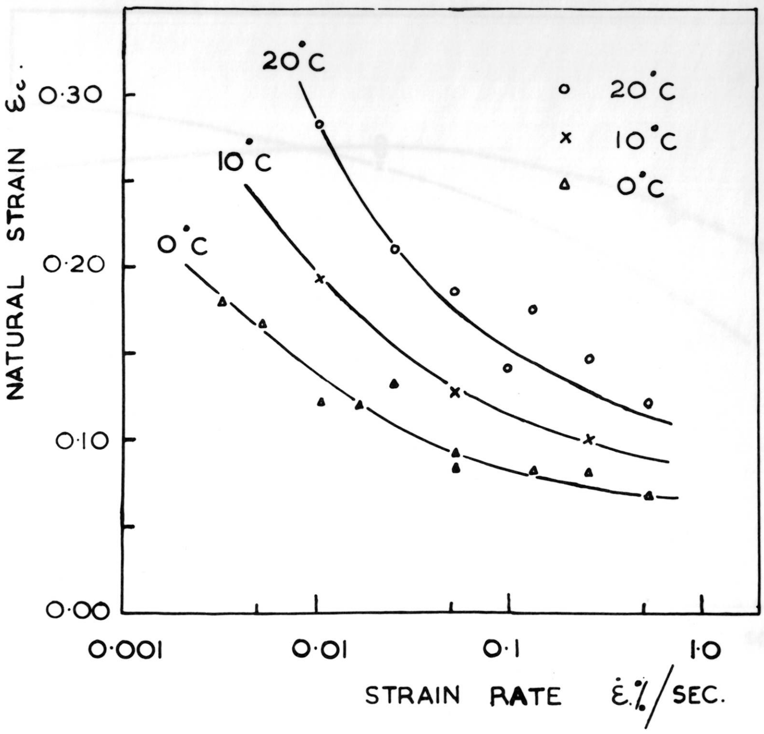


Fig. 29

The variation of elongation prior to cleavage ε_c at three temperatures, with the strain rate $\dot{\varepsilon}$.

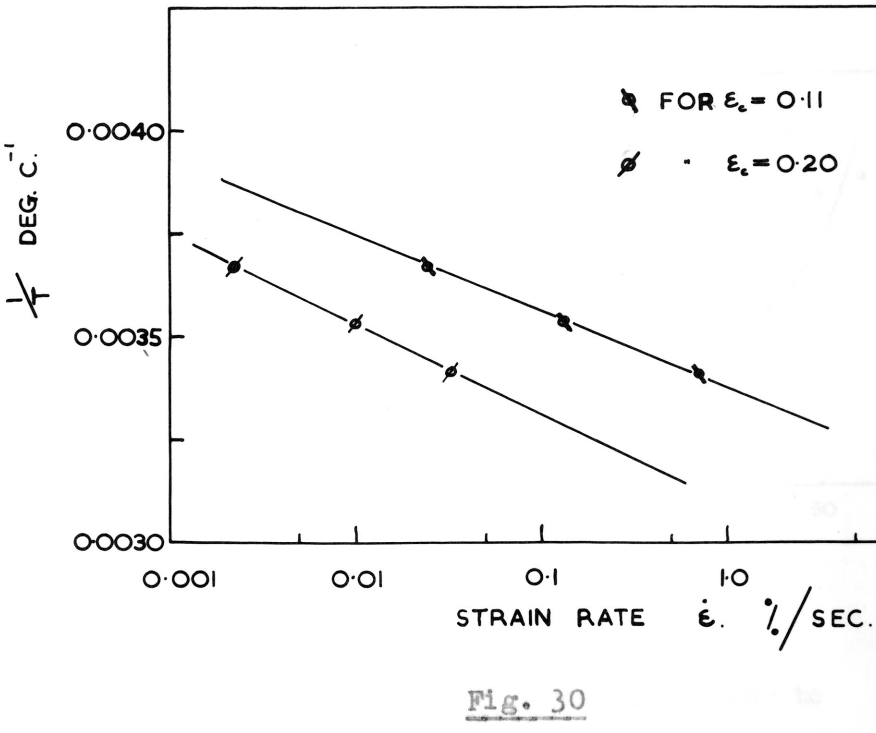


Fig. 30

An "Arrhenius plot" to determine an

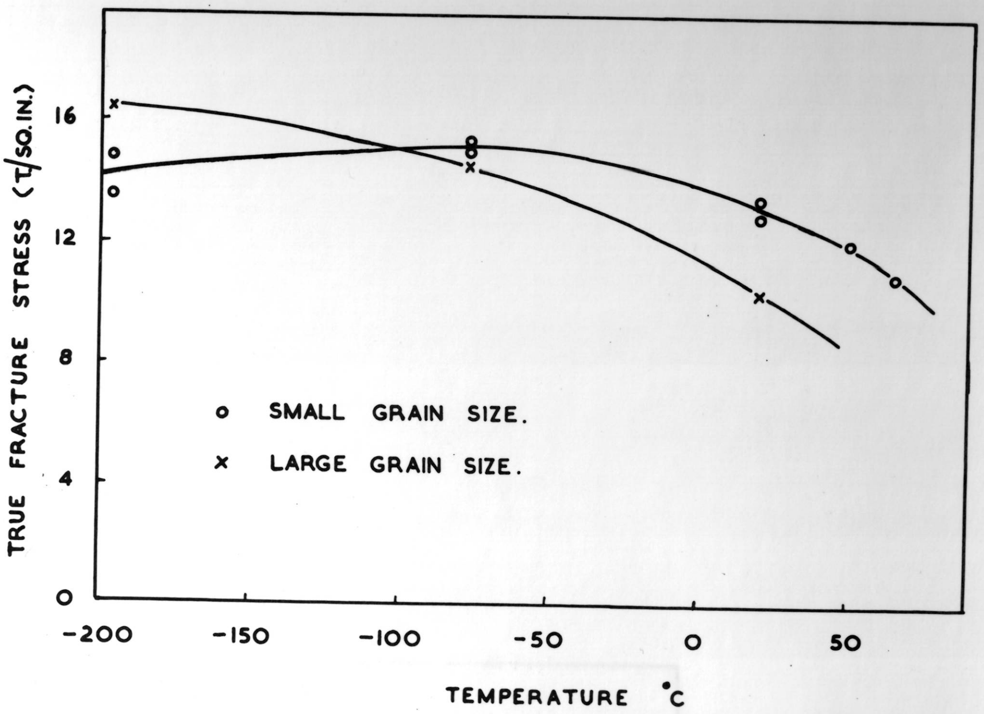


Fig. 31

The variation of true fracture stress with temperature and grain size for magnesium.

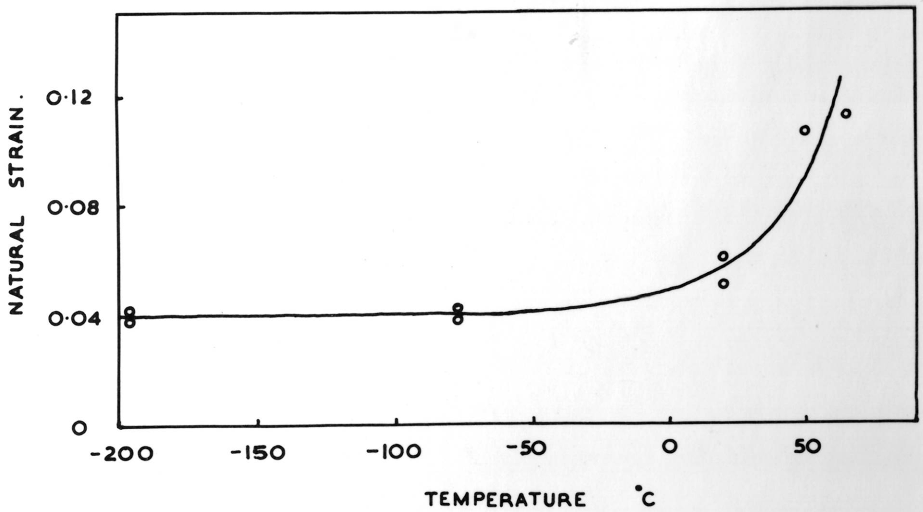


Fig. 32

The variation of elongation prior to cleavage with temperature for magne-

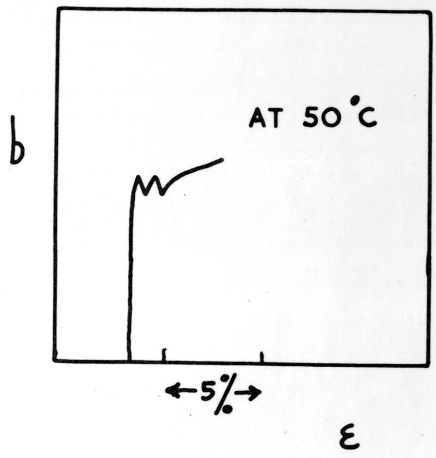


Fig. 33

A typical stress strain curve at 50° C.