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The Analysis of Fracture Surfaces by Electron Microscopy

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ABSTRACT

The fracture surfaces of metals and alloys exhibit markings and a topography which are characteristic of the modes of fracture operating during the initiation and the propagation of the crack. These markings can be easily and best differentiated by electronmicroscopy.

This work reviews and illustrates the characteristic features of the following modes of fracture:

Transgranular fracture which can take place either

- by cleavage along crystallographic planes
- or by the formation and coalescence of microvoids which leave concave depressions called "dimples" on both fracture surfaces,
- or by the slow progression of a fatigue crack by a mixed ductile - cleavage mode of fracture.

Intergranular fracture which is either

- brittle when there is a perfect separation of the grains along the grain boundaries,
- or ductile when this separation is associated with the plastic formation of void along the grain boundaries.

NOTE: In order to have a clear and concise discussion of the different modes of fracture, the complete descriptions of the different illustrations are given with the figures rather than in the text. The general direction of crack propagation is always indicated by an arrow.

I. INTRODUCTION

The systematic development of the structural analysis of fracture surfaces was begun several decades ago. A powerful means of investigation has recently been added with the use of the electron microscope. Electron micrography is used in the form of a special technique which is called "microfractography" or "electron fractography". This can be considered as an extension of the techniques of fractography used by metallurgists and exemplified by the work of Zapffe.[1]

The low depth of field (~ 0.2 microns) of the optical microscope makes photomicrography of non-planar surfaces almost impossible. The electron microscope with a considerably larger depth of field, a high resolving power and a large range of magnifications is an ideal tool to study deformed and fractured surfaces. The depth of field is of the order of 3 microns for a resolving power of 30 Å. When the resolving power is limited to 100 or 200 Å by the replicating technique the depth of field can reach 10 to 20 microns. This large depth of field of the electron microscope also makes possible the recording of stereoscopic views which are very helpful in understanding the microscopic topography of a fracture surface.

The fracture features which can be observed by electron microscopy may range in size and height from 50 Å (limit of the replicating techniques) to 25 microns (limit of the field of view at 2000X). A complete understanding of these features requires a basic knowledge of the different mechanisms of fracture and also some information on the nature and microstructure of the material. In the case of test specimens, the knowledge of testing variables such as temperature, load, stress at the tip of the crack, stress conditions (plane stress or plane strain), strain rate, environment etc., help considerably in understanding the microfeatures of the fracture surfaces. In the investigation of a service failure most of these variables will not be known; however, it is always possible to compare the features observed to similar features present on fracture surfaces of the same material tested under known conditions.

This document reviews and illustrates the characteristic topographies and features of different fracture surfaces resulting from tests where the microscopic modes of fracture were known. It is not intended to make a complete survey of the published work on microfractography, however, the outstanding contribution of Crussard, Plateau, and Henry[2,3] should be mentioned.

II. EVALUATION OF THE EXPERIMENTAL PROCEDURES

Optical Microscopy

In the study of fracture surface the use of the optical microscope should not be neglected in spite of its low depth of field. Observations at magnifications ranging from 3X to 30X yield the following information:

Localization of the origin of the crack,

Maximum crack length and profile of the crack front before fast propagation took place,

relative roughness of the different zones of the fracture surface (such as chevron markings in the region of fast propagation).

relative importance of the shear lips or cone fracture with respect to the total fracture surface.

These observations will help in deciding which regions should be replicated.

High power optical microscopy, with the use of a tilting stage in order to get the specimen in proper focus fills the magnification gap between low power microscopy and electron microscopy. For instance, Forsyth and Ryder[(4)] have done most of their excellent investigations on fatigue crack propagation by using the optical microscope at magnifications up to 1500X.

A cross-section of the fracture surface always provides some complementary information very valuable for the understanding of the topography of the fracture surface. In the case of a test specimen the cross-section can be prepared after stopping the test before complete fracture or separation occurs, i.e. at the stage of slow crack growth. The open fracture faces should be sealed or plated (with nickel in the case of an iron base material) to avoid deformation of the edges during sectioning and polishing.

Optical microscopy at 1000X or 2000X reveals features such as depths of microcavities, heights of steps on the fracture face, interaction of the crack front with grain boundaries, second phase particles, secondary cracks, etc.

Electron Microscopy

The only way to look directly at a fracture surface in the electron microscope is by using the techniques of reflection electron microscopy. Some work has been done using this technique to study fatigue fracture surfaces.[5] The results are very poor, partly because of the limited resolving power (300 Å) and also because the image is distorted since the fracture surface is viewed at a glancing angle. Furthermore, reflection electron microscopy is a long and difficult procedure. Consequently, the study of fractures with the electron microscope are based on the preparation of faithful replicas of the fracture surface. The different techniques for producing suitable replicas are summarized in Appendix I.

Artifacts

The one step relica, such as the direct evaporation of carbon on the fracture surface in vacuum, is the most reliable technique. It assures an exact reproduction and a proper interpretation of the fracture features. The two step replica presents the problem that artifacts are always present. An artifact can be defined as follows, "any property of a scientific method that can lead to incorrect results or misinterpretation can be considered as an artifact." Two types of artifacts are common in the two step replica: the first one is the result of an incomplete filling of the details of the fracture surface by the viscous plastic material; the second type of artifact is due to the damage done to the impression during separation from the specimen. A proper identification of artifacts is the basis of any good scientific procedure. In microfractography this identification can be done by comparison with the direct carbon technique, by preparation of many replicas of the same region, or even by replicating the same region on the two opposite halves of the fracture surface.

Orientation of the Replica

The transposition of a replica from the fracture surface into the electron microscope creates two main problems: First, it is difficult to locate exactly the area being photographed at high magnification with respect to the origin or a given reference point. For instance, it is possible to measure accurately the spacing of fatigue arrest lines (crack growth rate) but the corresponding crack length can be given only with an accuracy of $\pm$ .003" unless a complex technique of scribing reference markers is used. The second problem is due to the fact that it is difficult to know from the replicas and the micrographs the exact orientation of the macroscopic direction of crack propagation.

This problem can be partly solved by shadowing the replica in a direction parallel to the direction of crack propagation.

Interpretation of the Topography on the Micrographs

A beginner is very often confused by the topography of the fracture surface as seen on a micrograph. Most of the time the shadows are such that by rotating a print by 180°, what looked like a cavity becomes a mount and vice versa. The use of a stereo pair of pictures may also lead to a certain confusion since by changing the relative positions of the two pictures (left to right, right to left) the stereoscopic effect is inverted. However, a short study of the contrast resulting from shadowing the replica (Figure 1) shows that if the direction of shadowing and the type of replica (one step or two steps) are known, the interpretation of the topography is unambiguous. On a given micrograph it is always possible to find the direction of shadowing by looking carefully at the shadows of some small details. In order to study stereo micrographs in the best conditions, it is recommended to use a stereo viewer which can accommodate prints as large as 8" x 10".

III. MODES OF FRACTURE

Under a large tensile stress the atoms of a perfect crystal may separate by sliding past one another or they may pull apart.[24] In general, the fracture of metals and alloys takes place by processes which all stem from these two theoretical modes of crystal rupture. These processes of fracture can be classified as follows:

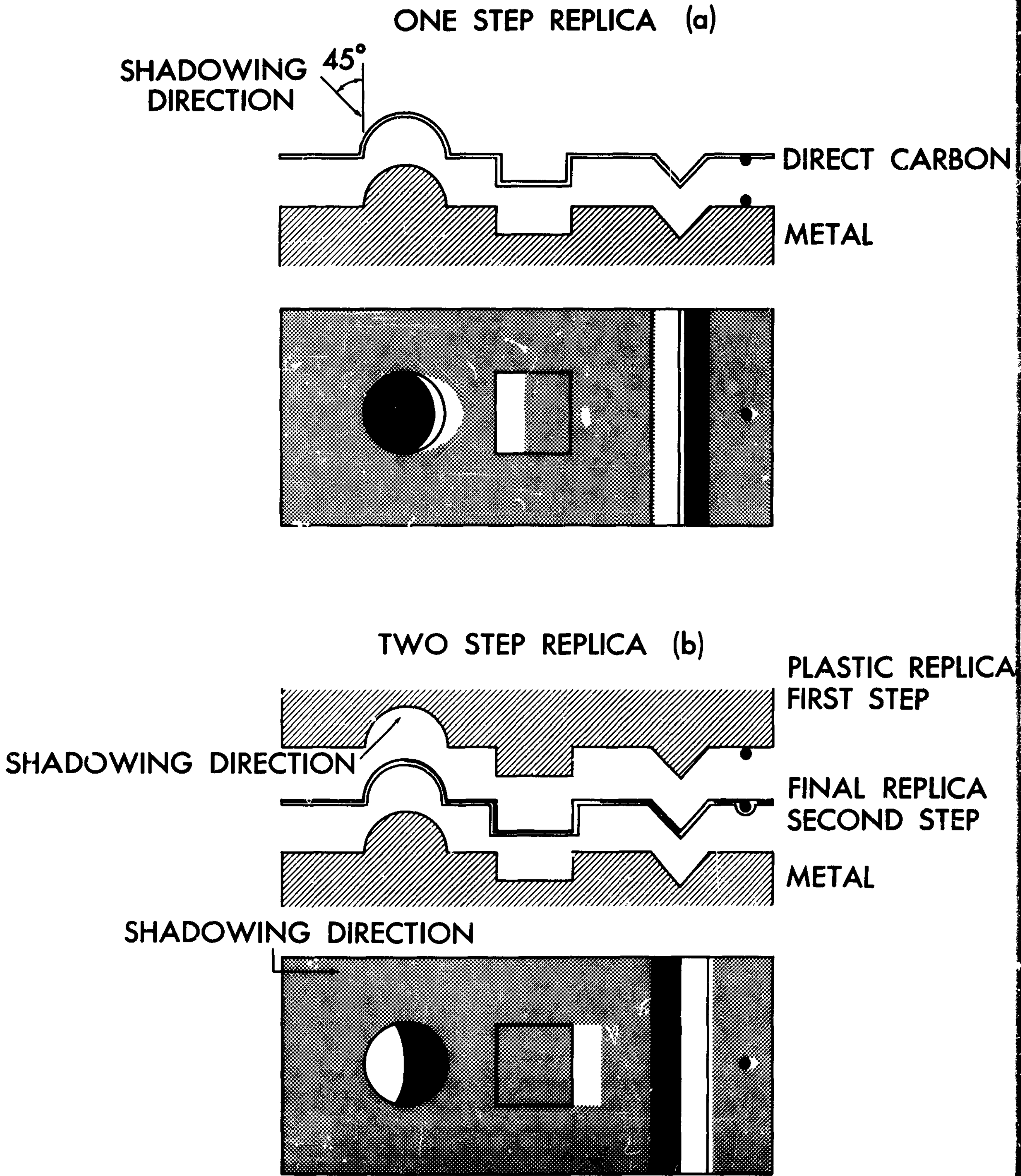

Figure 1 (a,b) - Illustration of the two types of replica. It can be seen that when the direction of shadowing and the type of replica are known, the topography (hills or valleys) of the fracture surface can be determined without ambiguity.

1. Cleavage Fracture

A typical example of a perfect cleavage fracture is the fracture of crystals of mica which split along their weakly bonded layer of planes. Many other crystalline substances including metals with body-centered cubic or hexagonal structures may fracture by separation normal to crystallographic planes of high atomic density. In the face-centered cubic structure, fracture by normal separation does not appear to be crystallographically controlled but it may take place on a very small scale in planes subjected to maximum tensile stress. It should be pointed out that cleavage fracture of metal crystals is always accompanied by some plastic deformation[(25)] which means that it is not a simple process of separation along atomic planes.

2. Plastic Fracture

(a) Fracture by uninterrupted plastic deformation

Metals that do not work harden much, some single crystals and also some polycrystalline metals draw dov- almost to a chisel edge or a point before breaking apart; this can hardly be termed "fracture" in the normal sense. It is usually referred to as "rupture" because the process arises from prolonged shear on slip planes within the worked region of the crystals which at one point finally shear apart.

(b) Fracture by formation and coalescence of microvoids

In most of the ductile tensile failures the reduction of area does not reach 100% although the overall deformation before fracture may be extensive. This is due to the fact that there is formation and coalescence of microvoids in the necked down region of the test specimen. The coalescence of the voids takes place by a process of internal and localized necking. It was observed by electron fractography that this process of plastic fracture by formation and coalescence of microvoids is very common in metals. It has been referred to as a "ductile fracture" (and also a "dimple" fracture") in opposition to the brittle cleavage fracture. The expression "ductile fracture" leads to some confusion since numerous fractures which would be referred to as "brittle fractures" after a macroscopic identification (no overall deformation, low impact energy) are found to be the result of a plastic rupture when they are carefully studied by electron microscopy. In the following discussion, the term "plastic fracture" will refer to a fracture which results from the growth and coalescence of transgranular voids.

3. <u>Intercrystalline Fracture</u>

The two preceding modes of fracture are observed in single crystals and polycrystalline materials: in all cases the fracture is transcrystalline. Polycrystalline materials exhibit another mode of fracture which is the separation of crystals from each other along the grain boundaries. This is a distinct type of fracture as it does not involve the fracture of the actual crystals; it is referred to as an "intercrystalline fracture".

In practice, it is rare when the fracture surface of a polycrystalline metal exhibits only one of the preceding modes of fracture; usually, one mode is predominant, but any of the others may have played a role in the initiation and propagation of the fracture. Consequently, the types of fracture are usually classified according to their cause and their appearance. This classification separates the fractures into two classes, "ductile fractures" and "brittle fractures", according to the amount of deformation and distortion which is present in the neighborhood of the fracture surfaces.

Two typical modes of ductile fracture are usually encountered:

(a) a fibrous fracture occurs when the fracture surface is normal to the direction of maximum tensile stress. A typical example is the cup in the "cup and cone" fracture of a round tensile specimen. In the case of a plate, the fibrous fracture is characterized by the presence of chevron markings which indicate the direction of crack propagation. A fibrous fracture usually takes place under plane strain conditions.

(b) A shear fracture takes place when the fracture surface makes approximately an angle of 45° with the direction of the maximum tensile stress. The cone in a "cup and cone" fracture of a round tensile specimen is a typical shear fracture; so is the shear lip in the fracture of a plate. A shear fracture is the result of a plane stress condition.

The expression "brittle fracture" covers the following modes and causes of fracture:

Cleavage which occurs below the transition temperature in most of the B.C.C. metals, particularly in iron and steels and also in some hexagonal metals.

Intergranular fracture, which is due to inter-crystalline weakness caused by embrittlement of the grain boundaries or by creep at a temperature above the equicohesive temperature and in some cases by low temperatures.[6]

Fatigue fracture is always characterized by a smooth fracture surface with well defined macroscopic and microscopic striations also called crack front arrest lines. Fatigue fracture is usually transgranular with a mixed plastic cleavage mode of fracture which is not well understood.

Stress corrosion fractures are caused by the embrittling effect of an environment. Hydrogen embrittlement, also called "static fatigue", can be classified as a form of stress corrosion failure. Stress corrosion fractures are intergranular or transgranular or both. Transgranular fractures usually follow a well defined crystallographic plane.

It should be repeated again that the expression "brittle fracture", as it is commonly used, refers to a fracture which occurs without detectable macroscopic deformation. The use of the electron microscope shows that many fractures classified as "brittle", are in fact, the result of a localized plastic rupture as defined precedently. A proper re-evaluation of the fracture vocabulary and classifications will be needed in order to keep up with the progress and understanding which result from the use of more sophisticated means of investigation.

IV. CLEAVAGE FRACTURE

The best example of a cleavage fracture is the fracture of alkali halide single crystals such as sodium chloride or lithium fluoride. One would expect the two surfaces created to be perfectly plane, but a single crystal is never perfect and the cleavage crack does not follow a single crystallographic plane. It is broken up along many cleavage planes. The steps between the different planes form a river pattern often called "river markings" which is characteristic of a cleavage fracture. The river lines tend to run together, giving the local direction of crack propagation, either cancelling each other or producing a larger cleavage step. The screw dislocations present in the crystal or created by plastic deformation at the tip of the crack are supposed to be responsible for the formation of cleavage steps on the crack surface. The best illustration of this explanation is the sudden increase in the number of cleavage steps that occur when the crack crosses a region of high screw dislocation density such as a twist-boundary (line ABC in Figure 2a). The steps or "river markings" between each cleavage surface are often the result of a fracture along a secondary cleavage plane. However, in many cases, when there is a small amount of plastic deformation associated with cleavage the river markings are just plain tear ridges formed by shear.[7]

In the case of polycrystalline materials the orientation of the cleavage plane changes across the grain boundary from one grain to the other. If the orientation change is not too large the crack front crosses the boundary with no change except for

2a - The river markings along ABC probably originate at a sub-boundary. The river markings are clearly due to cleavage along a secondary cleavage plane.

2b - Typical examples of river markings originating at a grain boundary.

Figure 2 (a,b) - CLEAVAGE FRACTURE. Typical river markings on a cleavage plane in an Alnico alloy (two step replicas).

the appearance of large cleavage steps originating at the boundary. This is well illustrated at the boundaries EF and GH on Figure 3. When two adjacent grains have a large orientation difference the crack front very often follows the grain boundary in order to go from one cleavage direction to another. The details of how individual grain cleavage cracks are joined along grain boundaries are in many cases obscured by the collapse of the replicating film. Crussard(2) has shown that in polycrystalline aggregates cleavage fracture is a discontinuous process, many different cleavage cracks being nucleated ahead of the main crack and often propagating in opposite directions in adjacent grains. The junction of these microcracks with the main crack front takes place by cleavage along secondary cleavage planes or separation along grain boundaries. It may also happen that some grains are so poorly oriented for cleavage that they deform extensively and fail by plastic rupture.

Figures 2 and 3 illustrate the cleavage fracture of an Alnico alloy. Figure 4 represents the brittle cleavage fracture surface of a Charpy impact bar or a pearlite free steel broken at 78°K. These three figures illustrate most of the main features of a cleavage fracture; that is:

(a) change of orientation of the cleavage plane from one grain to another;

Figure 3a - CLEAVAGE FRACTURE. Typical cleavage fracture through many grains of an Alnico alloy. The grain boundaries EF and GH are clearly at the origin of the formation of river markings. In grains A and B fracture was intercrystalline (two step replica).

Figure 3b - CLEAVAGE FRACTURE. Cleavage fracture in a TZM molybdenum alloy. The large cleavage "feathers" are made up of many cleavage facets. Extensive tear takes place between the large regions of cleavage (two step replicas).

Figure 4 - CLEAVAGE FRACTURE. Fracture surface of a pearlite free steel broken by impact at 78°K. ABC is a grain boundary; BD is a typical river marking. The facet F is due to the cleavage of a deformation twin. Fracture in G is intergranular. (one step replica).

(b) indication of the local direction of crack propagation by the river markings present on each cleavage plane;

(c) variability of the direction of crack propagation from grain to grain;

(d) regions of high deformation and/or of intergranular fracture along the grain boundaries;

(e) presence of small cleavage facets or "tongues" on the fracture surface of the steel specimen. The facets are due to the cleavage of deformation twins formed by plastic deformation at the tip of the propagating crack.

The features which characterise the cleavage fracture of two-phase aggregates are not so well understood and not so easily differentiated. Low and Turkalo[8] made a systematic study of the low temperature brittle fracture surfaces of the various decomposition products of the austenite of a 0.50% carbon steel. They observed that for pearlite or upper bainite the fracture path follows the cleavage plane of the ferrite phase through several pearlite colonies or bainite grain. For tempered lower bainite and tempered martensite the fracture surface is made up of many cleavage facets related to the bainite or martensite needle size. In the case of untempered martensite they observed that 50% of the fracture surface is due to an intercrystalline fracture along the austenite grain boundaries.

Figure 5 illustrates the brittle fracture surface of a Charpy impact test bar of A.I.S.I. 4340 steel broken at 78°F. Thecleavage facets are small and oriented in all directions and they do not seem to follow the martensite needles. Some coarse and short river markings can be observed on each facet. The facets are joined by highly distorted regions. Some intergranular fracture appears to be always present.

V. PLASTIC FRACTURE

As it was pointed out in the preceding classification of the different modes of fracture, a plastic fracture is the result of the growth and coalescence of microvoids formed during the last stages of deformation and during crack propagation. These microvoids produce the numerous concave depressions observed on the two opposite faces of the fracture. Crussard and his co-workers[2] were the first to observe these depressions with the electron microscope. They were named "cupules" or "dimples", the work "dimple" being more commonly used. Crussard[3] explains the mechanism of formation of the dimples as follows: "in the course of deformation and as a result of differences in elastic and plastic properties, microcracks are produced around the particles ahead of the main crack." Dimples have been observed on the fracture surfaces of a large variety of materials such as carbon and alloy steels, austenitic steels, aluminum titanium alloys and copper alloys.

Figure 5 - CLEAVAGE FRACTURE. Fracture surface of 4340 steel broken by impact at 78°K. The cleavage planes are broken up into small facets with coarse and ill defined river markings. The regions between each grain are highly distorted. In the center of the micrograph, it is difficult to differentiate between a cleavage fracture and an intergranular fracture (one step replica).

The dimples are characterized by their shape and their average sizes. By and large, the shape seems to depend essentially on the conditions of fracture while the size is more a function of the microstructure of the material.

Conditions of fracture and shapes of the dimples

Beachem[9] has recently published an excellent analysis of this problem which had also been studied partially by Crussard[3] and Tiner.[10] It is necessary to distinguish between a normal fracture (cup of a tensile fracture), a shear fracture (cone or shear lip of a tensile fracture) and a tear fracture (crack propagation in a plate under plane strain conditions). The sketches of Figure 6 illustrate the different shapes of dimples resulting from the different modes of coalescence of the microvoids.

Normal plastic fracture - The maximum principal stress is normal to the fracture surface and the rate of crack propagation is slow. The dimples are equiaxed as shown on sketch 6a and as illustrated on Figure 7. In many cases a particle,inclusion or precipitate, is observed near the bottom of the dimple where the void was nucleated.

Shear plastic fracture - When the state of stress is such that extensive shear takes place by plastic deformation during the formation and coalescence of the microcavities, the dimples are elongated and have the shape of parabola pointing in the direction of shear on each fracture surface. This is illustrated on

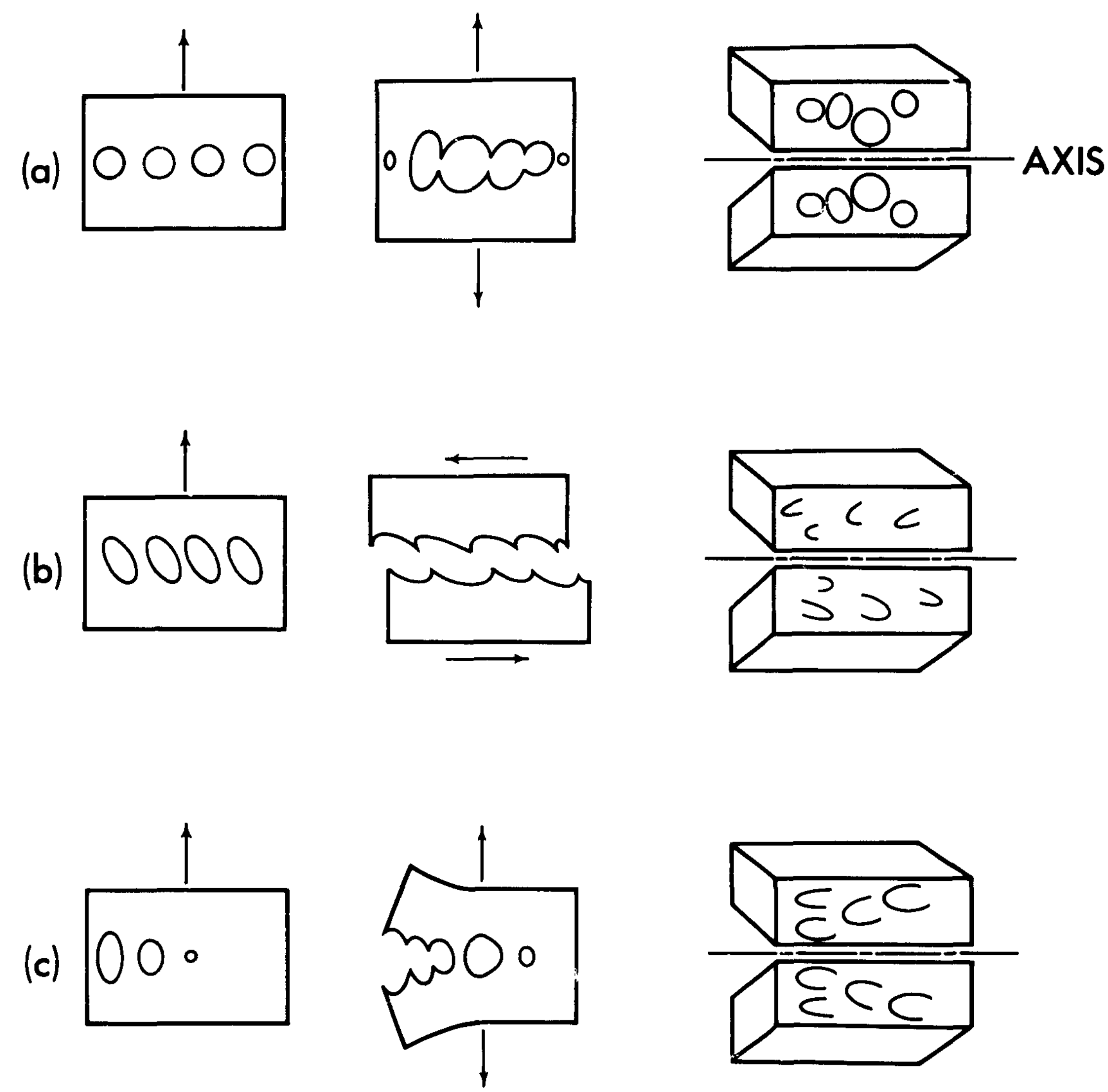

Figure 6 - PLASTIC FRACTURE (sketches from ref. 9)
a - normal plastic: formation of round dimples
b - shear plastic: formation of elongated dimples pointing in the direction of shear on each fracture surface,
c - tear plastic: formation of elongated dimples pointing in the direction opposite to the direction of each propagation.

Figure 7 - PLASTIC FRACTURE - NORMAL MODE
Formation of round, equiaxed dimples in a normal plastic fracture of a 4340 steel. Some inclusions or second phase particles can be observed at the bottom of the dimples (one step replica).

sketch 6b and Figure 8. A shear fracture surface also shows, besides regions covered with elongated dimples, some very flat areas. According to Crussard, these flat areas are due to fracture along slip planes which have been weakened by deformation. This hypothesis would need further confirmation.

Plastic fracture by tear - Sketch 6c shows the formation and orientation of the dimples in the case of a tear fracture. Like in a shear fracture, the dimples have the shape of parabola, but now on both surfaces they point in the direction opposite to the direction of crack propagation. A good example of tear dimples is shown on Figure 9.

Beachem(9) considers that the elongated shape of the dimples is due to the extensive plastic deformation taking place when the microcracks link up with the main crack front. Although this factor plays an important role, it is felt that the parabolic shape of the dimples is the result of a difference in the rates of propagation of the microcracks along the longitudinal and transverse directions of the parabola. This shape can also be explained simply by the fact that the part of the microcrack which is the farthest away from the crack front, would have more time to propagate in a transverse direction than the part closest to the main front. It is interesting to note that similar parabola-shaped dimples appear on the fracture surfaces of non-crystalline materials such as lucite or plexiglass as shown on Figure 10. In this case however, the dimples are very large, probably because of the

Figure 8 - PLASTIC FRACTURE - SHEAR MODE
Formation of elongated dimples and of a flat region of glide decohesion in a shear fracture of a 4340 steel. (one step replica). The arrow indicates the shear direction.

Figure 9 - PLASTIC FRACTURE - TEAR MODE
Elongated dimples resulting from a tear plastic fracture in a 4340 steel. See sketch 6C (two step replica).

Figure 10 - FRACTURE MARKINGS ON PLEXIGLASS
Matching fracture surfaces. Note the matching features A, A' and B, B' on the two fracture faces. The parabola markings are similar to the plastic dimples observed in a tear ductile fracture of a metallic material.

limited number of crack nucleation sites present ahead of the main front. Irwin[11] showed that these macroscopic markings on a fracture surface are the result of the differences in the speed of propagation of a multitude of separately initiated components of fracture. The same phenomena seems to take place on a microscopic scale in the process of ductile fracture of metals and alloys. The shape and size of the dimples will, therefore, depend on the overall velocity of propagation of the main crack front which is, in turn, governed by the rate and distance at which advance initiations can occur and the rapidity with which the microcracks can be linked together.

Size of dimples

The second phase particles and inclusions distributed in a metal or alloy play an important role in the initiation of the microcracks, and therefore, in the mechanism of fracture. This is confirmed by the fact that very pure and clean metals, solid solutions and even quenched supersaturated solid solutions such as Al-Cu when pulled in tension break with 100 percent reduction in area. This role of the second phase particle initiating fracture, explains why there is a relationship between the size and distribution of the particles and ductility or reduction of area. Plateau[12] has recently derived a mathematical expression for this relationship and shown that a large amount of experimental data fits his theoretical curve. However, no work so far has attempted to relate the size of the dimples to the interparticle spacing.

This would be rather difficult because of the complex shape of the dimples and only a statistical distribution of sizes would be meaningful.

Figures 11, 12, 13 and 14 illustrate the different sizes and shapes of dimples observed in some common materials. In age hardening alloys such as the aluminum alloys or the maraging steels where most of the second phase particles are coherent with the matrix, it is evident that the size of the dimples is much larger than the spacing between the precipitates. In steels such as 4340, it has been observed that the higher the fracture toughness, the larger the dimples. This shows that only a limited number of inclusions of precipitates act as crack nucleation sites ahead of the main crack front, and that this number depends on the overall velocity of propagation of the crack. Irwin[11] concludes from his observations that an increase in crack velocity is accompanied by an increase in the number of microcracks which are initiated closer and closer to the main crack front. For steels, it is known that the transition from a ductile to a brittle (cleavage) fracture corresponds to a marked increase in the speed of propagation of the crack front. Although it is easy to identify by electron fractography, a true cleavage fracture or a perfect plastic fracture, the features of a ductile-brittle fracture are not very well understood. The expression "quasi-cleavage" is usually employed to describe this type of fracture illustrated by Figure 15. Beachem[7] shows that i is

Figure 11A - PLASTIC FRACTURE - Example of tear plastic dimples in a 7178 aluminum alloy. The average size of the dimple is of the order of microns.

Figure 11B - PLASTIC FRACTURE - Example of plastic dimples in a 2219 aluminum alloy. Note the range of sizes of dimples.

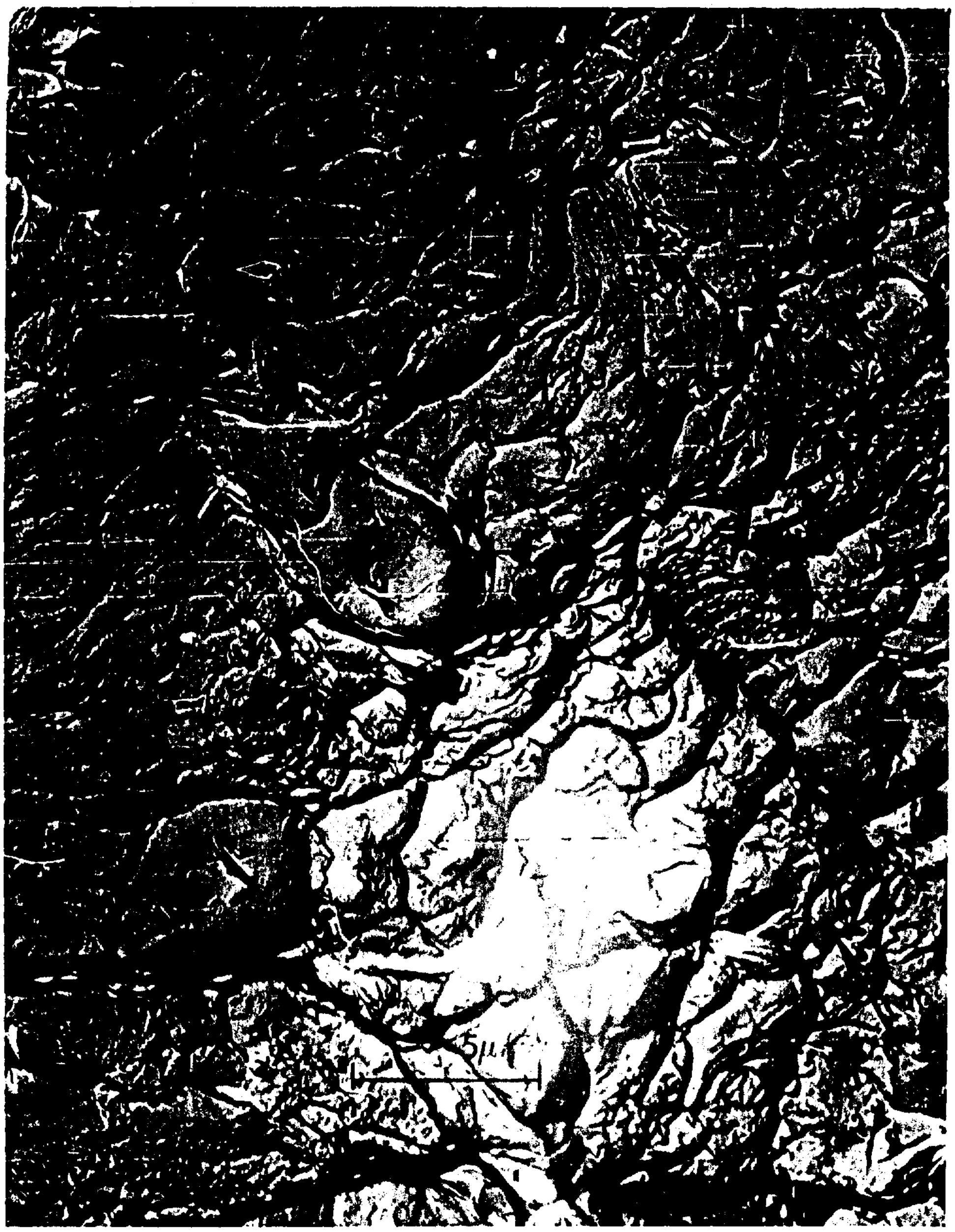

Figure 12 - PLASTIC FRACTURE - Example of plastic dimples in a maraging steel (two step replica).

Figure 13 - PLASTIC FRACTURE - Large dimples in a 70-30 brass resulting from a tear fracture. Many of the dimples may have originated at dislocation pile-ups or sub-boundaries (two step replica).

Figure 14 - PLASTIC FRACTURE - Example of large plastic dimples in a titanium alloy (Ti - 6Al - 4V) (two step replica).

Figure 15 - QUASI CLEAVAGE - Fracture surface of a 4335 steel showing quasi-cleavage. The surface is made up of shallow dimples and small cleavage facets,(two step replica).

easy to differentiate between true cleavage and quasi-cleavage facets by finding the origin of the local fracture. In the quasi-cleavage facet, the origin is contained within the facet, whereas in true cleavage, the initiation point is at the edge of the facet. A quasi-cleavage facet could be described as a ductile dimple approaching a condition of fracture by cleavage. A better understanding of the micromechanisms of fracture in the ductile-brittle transition zone will require further work.

Finally, Rogers[13] pointed out that inclusions are not necessarily the only sites of initiation of a plastic fracture. Microcracks may form at dislocation pile-ups or certainly along subboundaries. The smooth, slightly polyhedral dimples observed in 70-30 brass on Figure 13 may result from the decohesion along a slip plane or a subboundary. Dimples may also be initiated at grain boundaries. However, except in the case of ductile intergranular fractures which will be described later, it is, in general, impossible to distinguish the grain boundaries and the grain size on a fracture surface showing extensive dimple formation.

VI. INTERGRANULAR FRACTURE

Intergranular fracture is a separation of the crystals from each other along the grain boundaries. A schematic picture of the three dimensional network formed by the grains of a polycrystalline metal is shown on Figure 16 which represents a three-dimensional model of a stack of tetrakaidecahedrons.[14] Figure

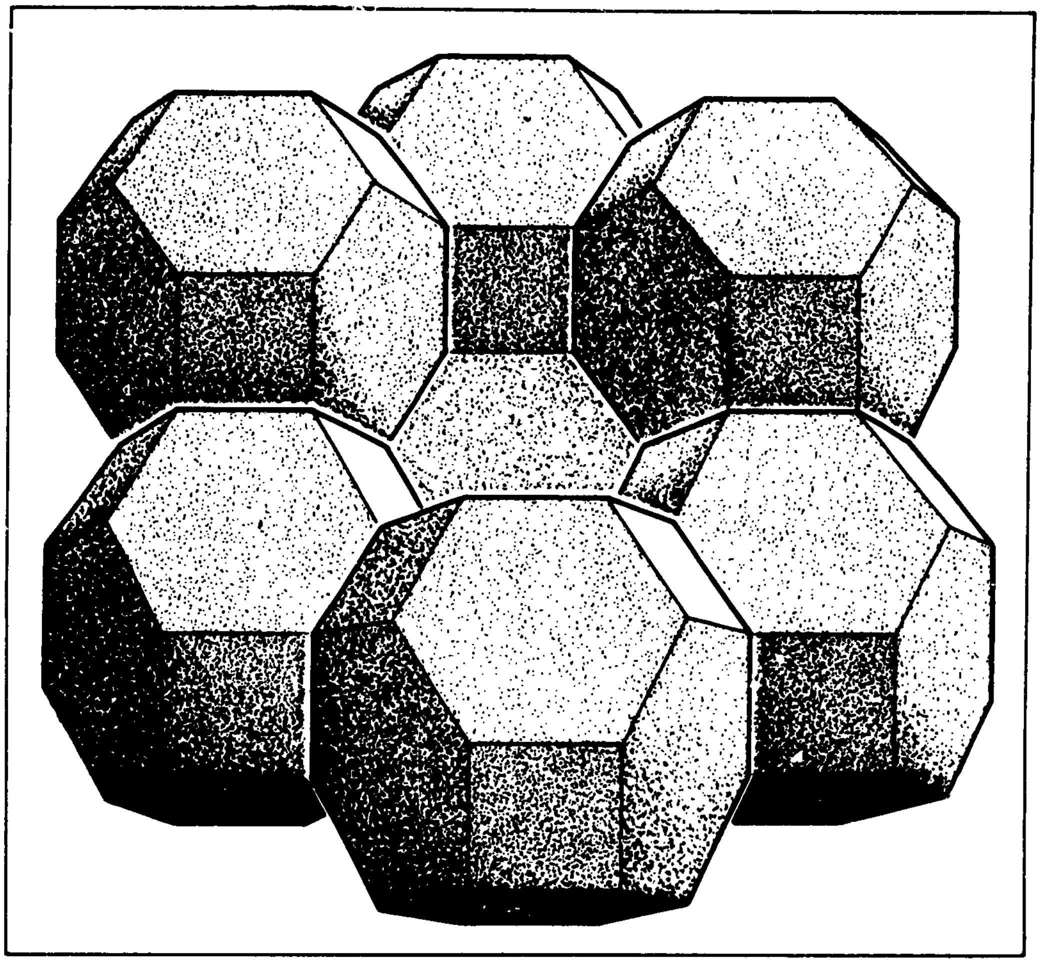

Figure 16 - INTERGRANULAR FRACTURE - Stack of tetrakaidecahedrons (14) representing the ideal assembly of crystals or grains in a polycrystalline metal. (two step replica).

17 shows that a real intergranular fracture looks almost like the theoretical picture described on Figure 16. On the real fracture each facet of the grains is slightly curved which shows that the conditions for metastable equilibrium of the grain boundary network were satisfied at the austenitizing temperature. The small "hairlines" on each facet are unaccounted for and may be related to the second phase particles or to the mechanism of separation of the grains. Another example of a perfect intergranular fracture is shown on Figure 18 which represents the fatigue fracture surface of a 70-30 brass. An intergranular quench crack in a 4340 steel is shown on Figure 19. Figure 20 represents an intergranular stress corrosion fracture in 7075 aluminum alloys.

All these figures are good illustrations of a brittle intergranular fracture, the term brittle referring to the total lack of deformation associated with the final separation of the grains. This type of fracture is observed at room temperature in materials where the grain boundaries have been embrittled by a film of a brittle phase or by the segregation of an impurity without the appearance of a second phase. This seems to be the case of the temper brittleness and hydrogen embrittlement of high strength steels.

Plateau, Henry and Crussard(15) have described a number of cases of intergranular brittle fracture resulting from the formation of film-like precipitates at grain boundaries. Notable among these were several austenitic chromium nickel steels which

Figure 17 - INTERGRANULAR FRACTURE - Brittle intergranular fracture in a 4340 steel. This intergranular fracture at the origin of a service failure was probably due to stress corrosion and hydrogen embrittlement. Note the hairlines along the grain boundaries facets and the microcavities in A and B (two step replica).

Figure 18 - INTERGRANULAR FRACTURE - Brittle intergranular fracture in a 70-30 brass. This fracture is the result of a slow crack propagation under cyclic load in a centrally notched panel of brass. (Two step replica).

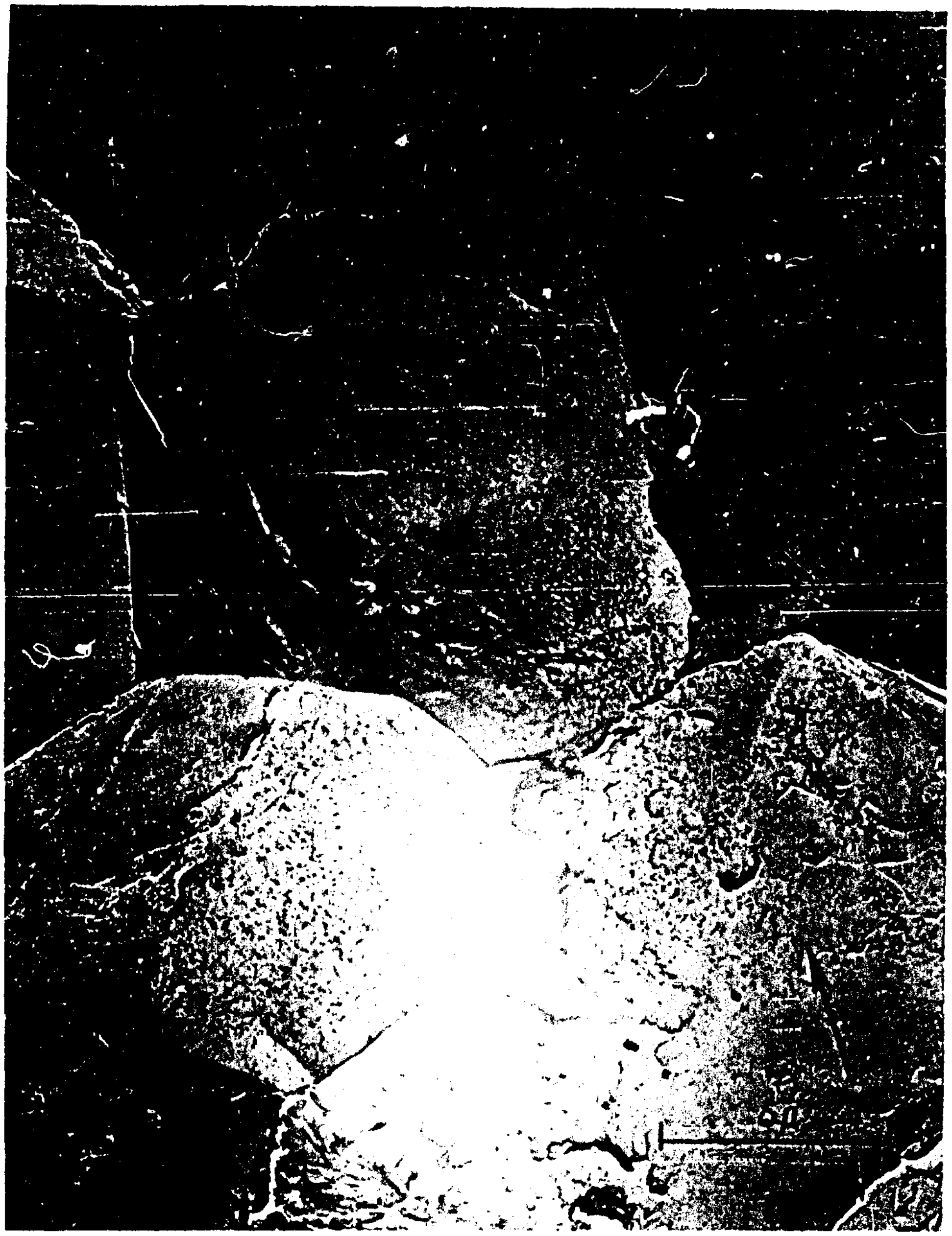

Figure 19 - INTERGRANULAR FRACTURE - Brittle intergranular fracture in a 4340 steel. This fracture took place during quenching. (two step replica)

Figure 20 - INTERGRANULAR FRACTURE - Brittle intergranular fracture due to stress corrosion in a 7079 T6 aluminum alloy. (Two step replica)

had been embrittled by intergranular carbide precipitation. They found that the grain boundary film does not have to be continuous to produce intergranular fracture. The fracture was observed to occur at the carbide matrix interface or sometimes by cleavage at the carbide plate. Figure 21 shows an example of an intergranular fracture in AM355 due to $M_{23}C_6$ carbides.

Stress corrosion fracture in steels and aluminum alloys is usually intergranular; but in most cases, the fracture surface is so badly corroded that a detailed identification of the modes is usually very difficult.

The best known examples of intergranular fracture are the result of creep at temperatures above the equicohesive temperature ($\gtrsim 0.5$ absolute melting point). Grant[16] and Gifkins[17] have given some excellent reviews of this mode of fracture. A very small amount of fractography work has been done in this case. The grain boundaries often show a striated appearance probably related to the serrations observed by optical microscopy which are due to grain boundary sliding. This is shown on Figure 22. In opposition to creep fracture, it should be mentioned that Backofen[6] has recently observed intergranular fracture in pure aluminum at very low temperatures, which would indicate the existence of another equicohesive temperature.

Although grain boundary cracking corresponds in general to a slow rate of crack propagation, it has not been possible so far to determine the exact mechanisms of propagation. There are

Figure 21 - INTERGRANULAR FRACTURE - Brittle intergranular fracture and network of carbides along the grain boundary facet in AM335. (two step replica)

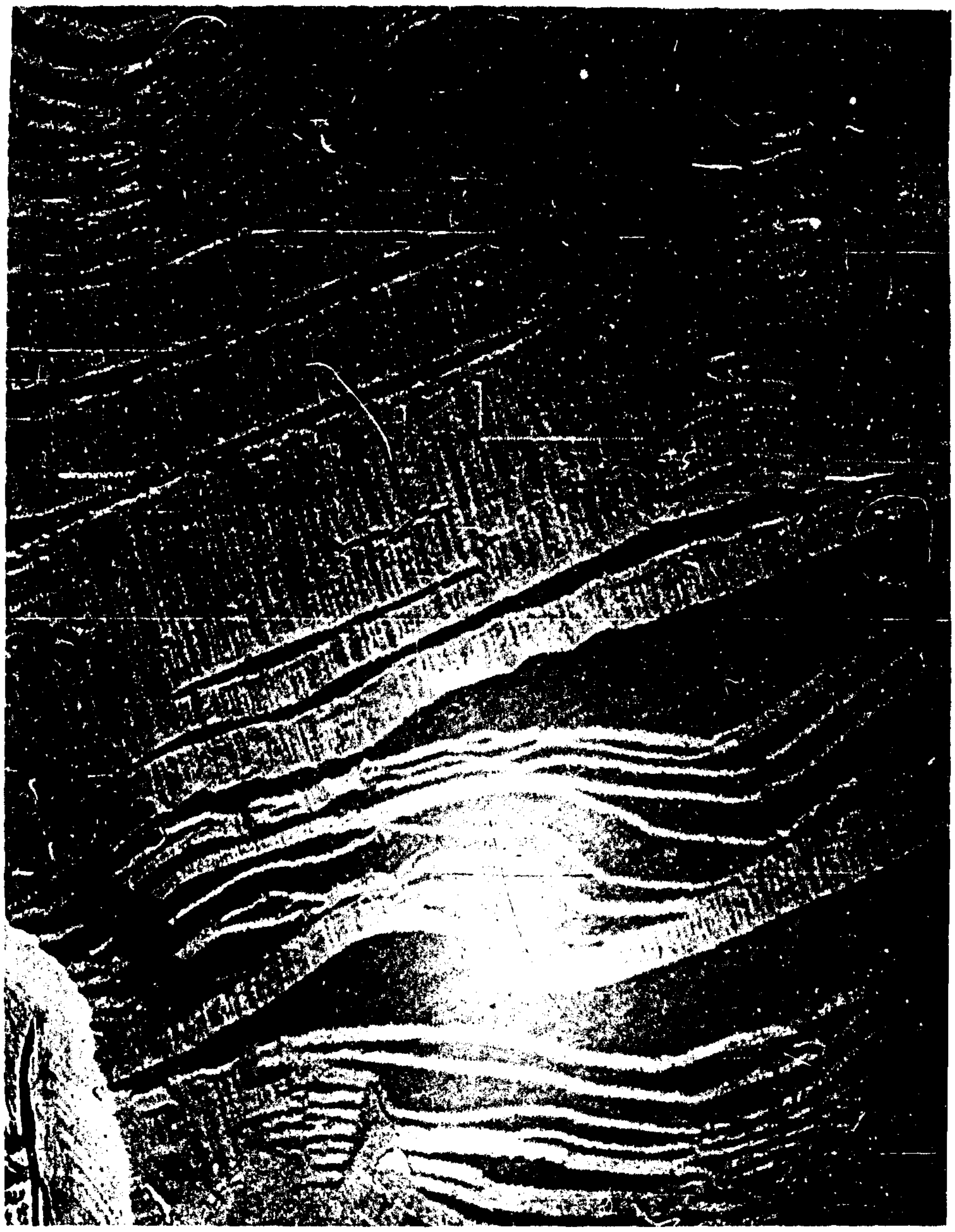

Figure 22 - INTERGRANULAR FRACTURE - Brittle intergranular fracture due to high temperature creep in a Nimonic alloy. The striated appearance of the grain boundary facet is probably due to grain boundary sliding and formation of serrations during creep. (two step replica)

no clearly visible features such as crack front arrest lines or nucleation sites. Some controversial microcavities, supposedly due to hydrogen condensation, have been observed in some cases in steels.[18] We feel that the "hairlines" observed on the grain boundary facets of Figure 17 are certainly connected with the initiation and propagation of the fracture. However, this conclusion is purely qualitative.

The preceding examples seem to show that an intergranular fracture should be easy to identify. In order to avoid any doubt, it is always recommended to compare the grain sizes on the fracture surface and on a cross-section. In many cases of fracture of high strength steels, it was found that intergranular cracking was always mixed with ductile fracture; a good example of this behavior is shown on Figure 23. In such a case, it is sometimes difficult to differentiate between a grain boundary face and a cleavage or grain-cleavage facet.

A second mode of grain boundary separation is the plastic intergranular fracture. The separation along the grain boundaries is accompanied by an extensive shear deformation and formation of elongated shear dimples. Figures 24 and 25 show a plastic grain boundary fracture in a 2219 aluminum alloy. This type of fracture is usually connected with the well-known delamination fracture of some aluminum alloys which result from a depletion of second phase precipitates along the grain boundaries. Another example of plastic intergranular fracture takes place during creep at high temperatures when voids form along grain boundaries by diffusion of vacancies.

Figure 23 - MIXED PLASTIC - INTERGRANULAR FRACTURE
Example of juxtaposition of two modes of fracture: plastic and intergranular in a 4340 steel (two step replica).

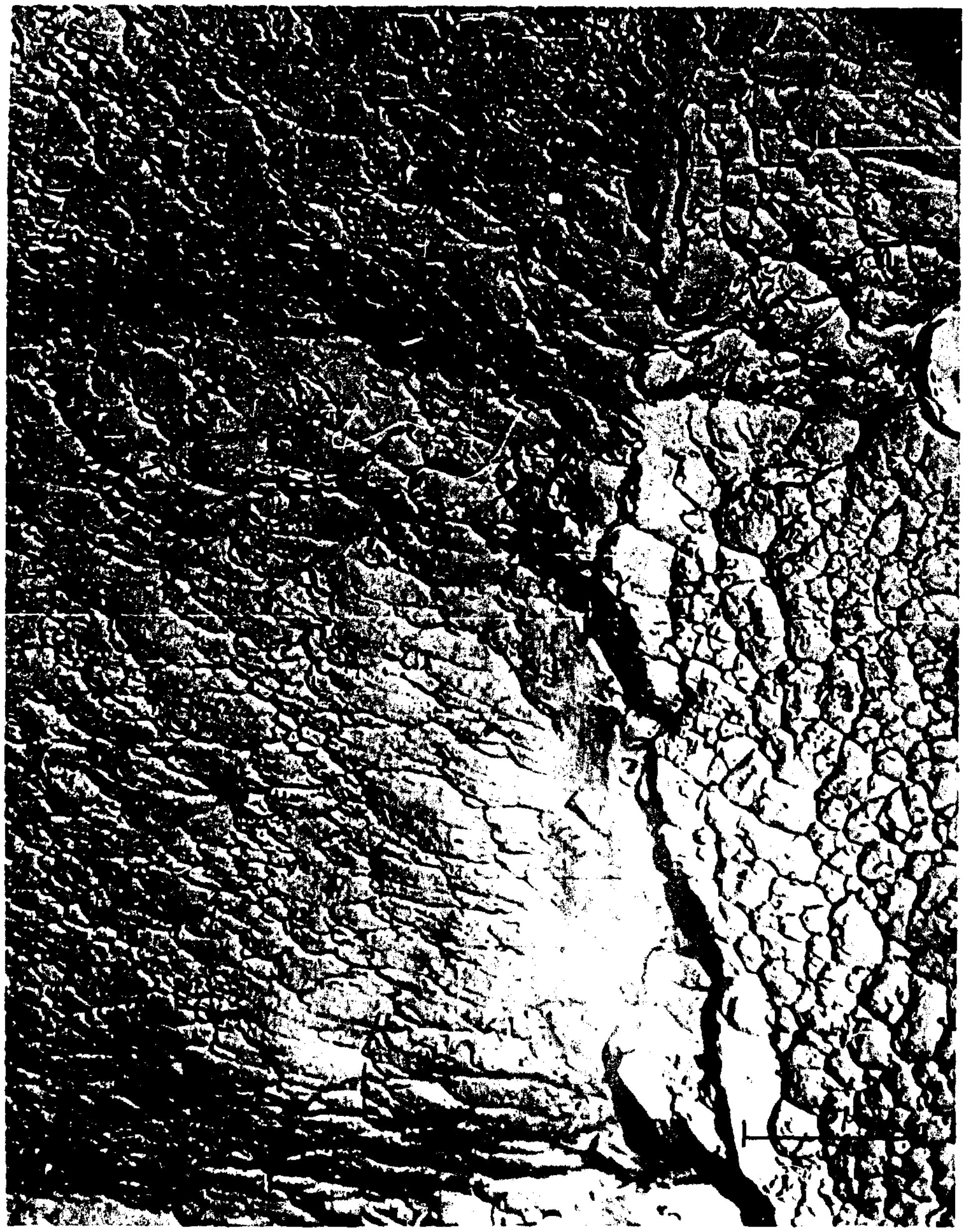

Figure 24 - INTERGRANULAR FRACTURE - Plastic intergranular fracture in a 2219 alloy. The difference in orientation of the grain boundary facets is well marked and the shear direction is given by the direction of elongation of the dimples. (two step replica).

Figure 25 - INTERGRANULAR FRACTURE - Plastic intergranular fracture in a 2219 alloy. The difference in orientation of the grain boundary facets is well marked and the shear direction is given by the direction of elongation of the dimples. (two step replica)

IV. FATIGUE FRACTURE

The examination of the fatigue fracture surface of an aluminum alloy by optical microscopy shows dull and bright regions. The dull zones are depressions usually with a large inclusion visible at the bottom of the depression. The bright zones show a series of regular striations as illustrated by Figure 26. The examination of the same surface by electron microscopy shows that the depressions are made up of dimples characteristic of a rapid, plastic fracture. The striations of the bright zones are clearly resolved (Figure 27) and in the case of a fatigue test under a constant stress level, the spacing of the striations is locally very uniform. The examination of program loaded fatigue fractures has demonstrated that each of the characteristic fatigue striations first observed by Zapffe and Worden[19] is produced by a single cycle of stress,[4] thus the striations also called crack front arrest lines, represent the successive positions of a transgranular front at each load cycle.

Forsyth[20] has recently given an excellent survey of the mechanisms of fatigue crack propagation in aluminum alloys. Two general types of striations have been recognized, the brittle and the ductile types. Figure 28 taken from Forsyth, illustrates the differences between the two types of striations. The brittle striations are connected with what seems to be a cleavage fracture along sharply defined facets. Numerous river line markings separating these facets run normal to the striations as shown on Figure 29.

Figure 26 - FATIGUE FRACTURE - Optical micrograph of a fatigue fracture in a 7178 aluminum alloy. The regions out of focus are due to the small depth of field of the optical microscope. (two step replica).

Figure 27 - FATIGUE FRACTURE - Electron micrograph of a fatigue fracture in a 7178 aluminum alloy. By contrast with Fig. 26 this micrograph shows the larger depth of field of the electron microscope. The flat regions where the crack propagated in a cyclic manner are connected by ductile regions where fracture took place suddenly around inclusions or by shear.

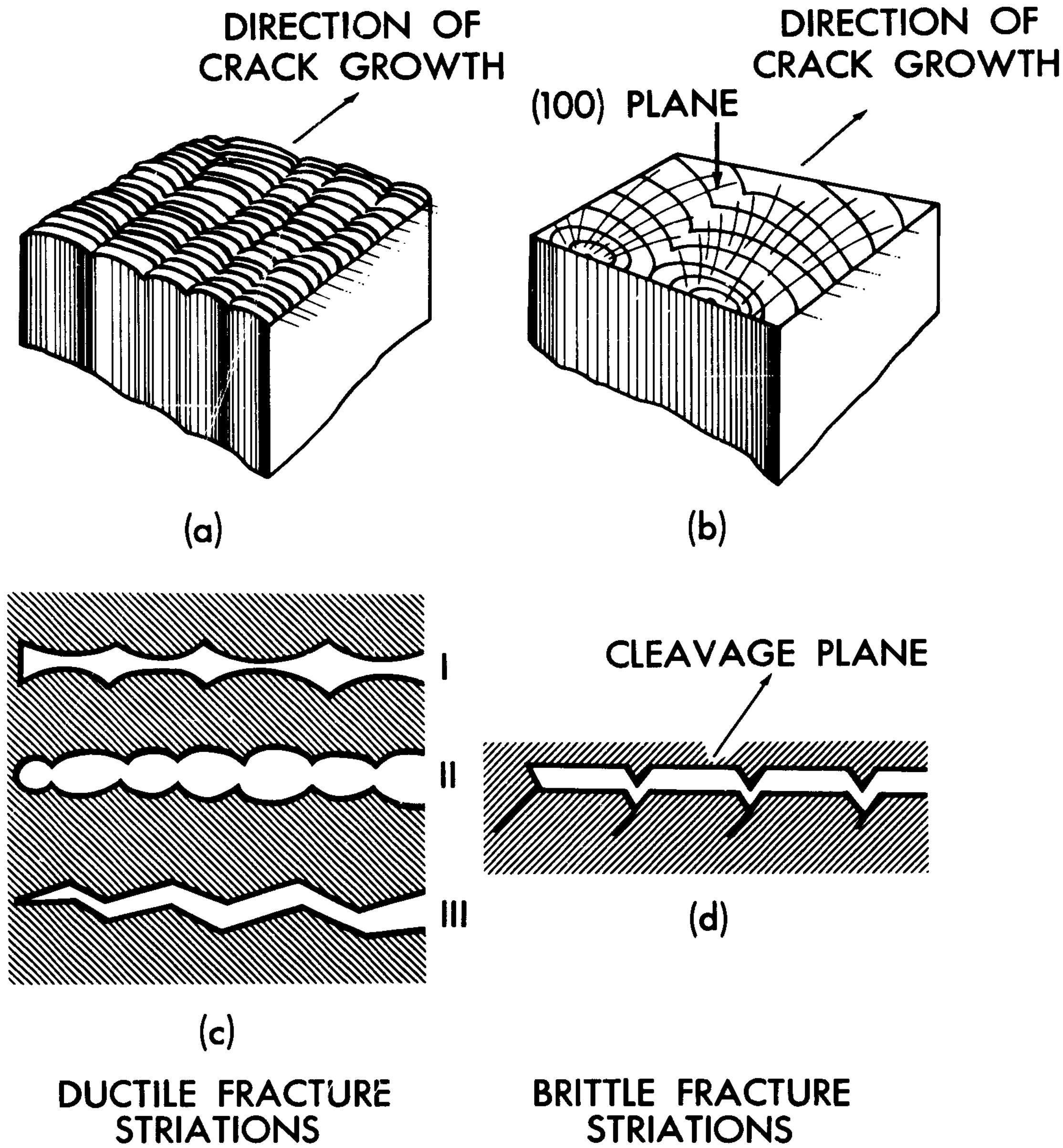

Figure 28 - FATIGUE FRACTURE - Different type of ductile and brittle fatigue striations. (Sketches a, b are taken from Forsyth (20)) (two step replica).

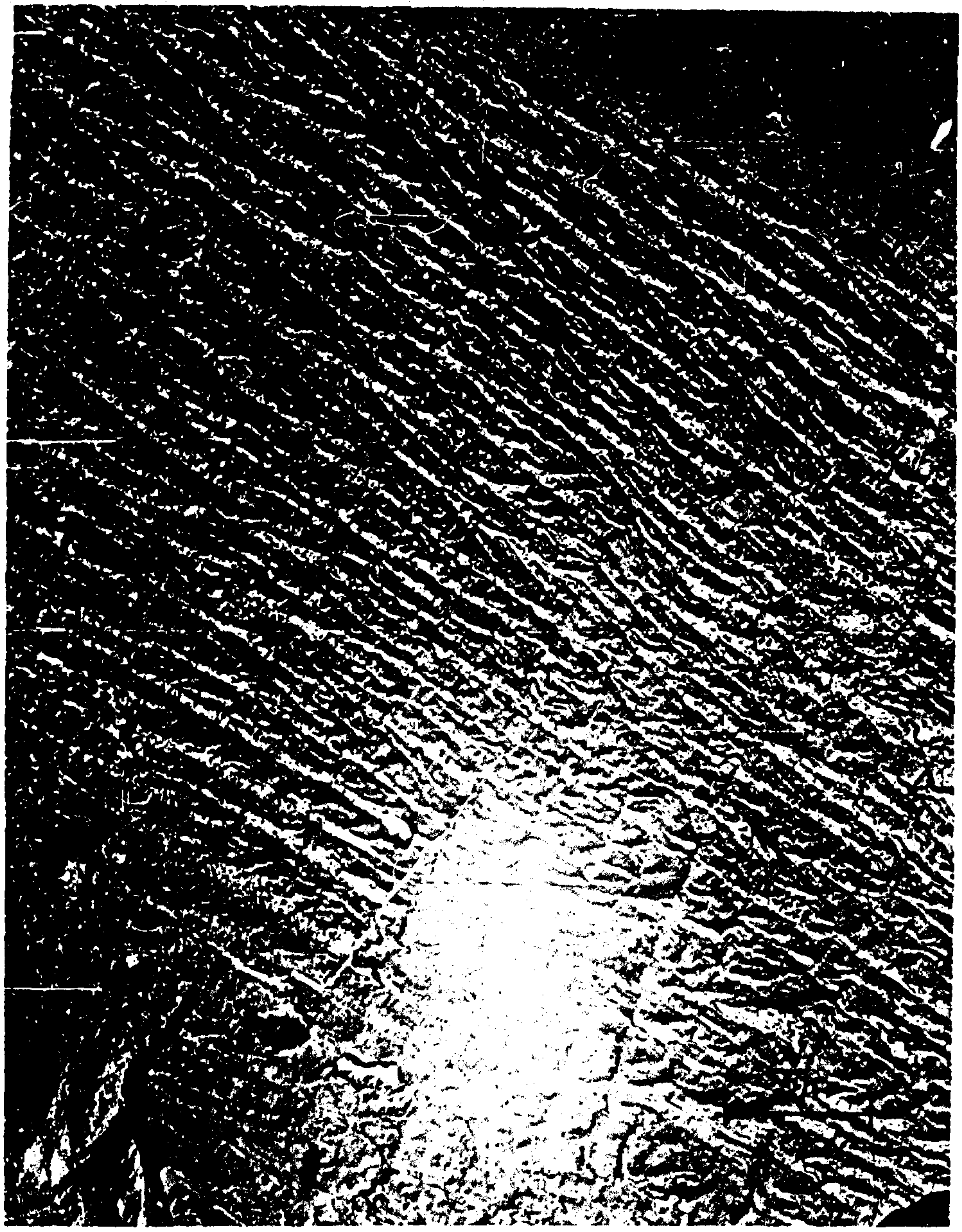

Figure 29 - FATIGUE FRACTURE - Example of brittle fatigue striations in a service failure of a 2014 aluminum alloy. Note the cleavage facets running parallel to the direction of crack propagation and normal to the fatigue striations. (two step replica).

The profile of a brittle striation is shown on sketch 28d. As far as it is known, brittle striations have been observed only in high strength aluminum alloys, and they are usually indicative of the presence of a corrosive media.

The ductile striations are more common and they have also been observed in polymers which would show that their formation is not necessarily related to the crystallographic nature of metals. Figures 27 and 30 give some good examples of ductile fatigue striations. The exact mechanism of formation of these striations is not clearly understood. Sketch 28e presents different profiles of fatigue striations. Smith and Laird[21] and McEvily[22] have shown that in the case of high stress fatigue, the profiles of the two fracture surfaces do not match. Under conditions of low stress fatigue, it is not clear at this stage, whether the profiles are of the type II or type III represented on Figure 28.

Grain boundaries play an important role in fatigue crack propagation. The different figures presented here show that in aluminum alloys, the crack front is held up along grain boundaries, and the orientation of the striations changes from one grain to the other. This could be accounted for by the fact that in high stacking fault energy materials, fatigue cracking is the result of deformation by cross-slip which is more extensive inside the grains than along the grain boundaries.

Under uniform load, the height and the spacing of the striations increase with the crack length. The spacing of the

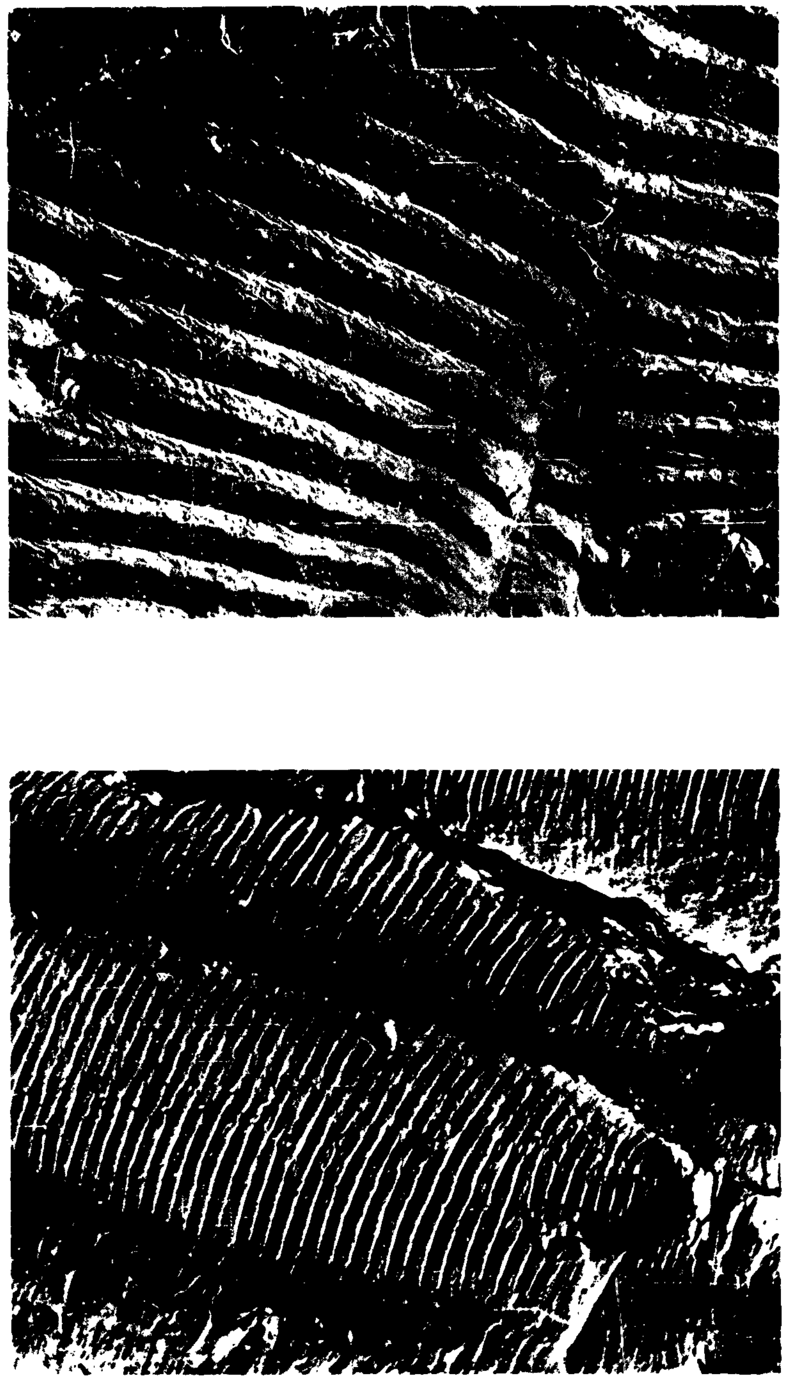

Figure 30 (a,b)- FATIGUE FRACTURE - Examples of ductile fatigue striations in a 7178 aluminum alloy. Figure 30b shows clearly the role of the grain boundaries in slowing down the progression of the crack front (two step replica).

striations is not always equal to the theoretical spacing or crack growth rate derived from measurements of crack length versus number of cycles. If the crack front does not move in a uniform manner over its whole length, the average spacing of the striations will be larger than the macroscopic growth rate. However, in general, brittle fracture of inclusionsand ductile tear around these inclusions result in a rapid local advance of the crack front. Consequently, the striation spacing is, in general, smaller than the macroscopic crack growth rate observed on the surface of a test panel. Figure 31 illustrates clearly the extensive brittle fracture of large inclusions. The spacing of the striations is also markedly dependent on the load.

Figure 32 shows that under a random load, the spacing of the striations may vary considerably. The well defined fatigue striations provide us with a perfect marker which should be used to measure the true influence of a peak overload on the rate of crack propagation.

Other examples of fatigue striations are shown on Figures 33, 34, 35 and 36. In general, the striations in different materials are not as sharply defined as they are in high strength aluminum alloys. In steels for instance, the successive positions of the crack front do not appear clearly defined; the striations are short and discontinuous. Very often, according to the ratio of the maximum to the minimum stress, the details of the fracture surface have been destroyed by the rubbing action of the two fracture faces.

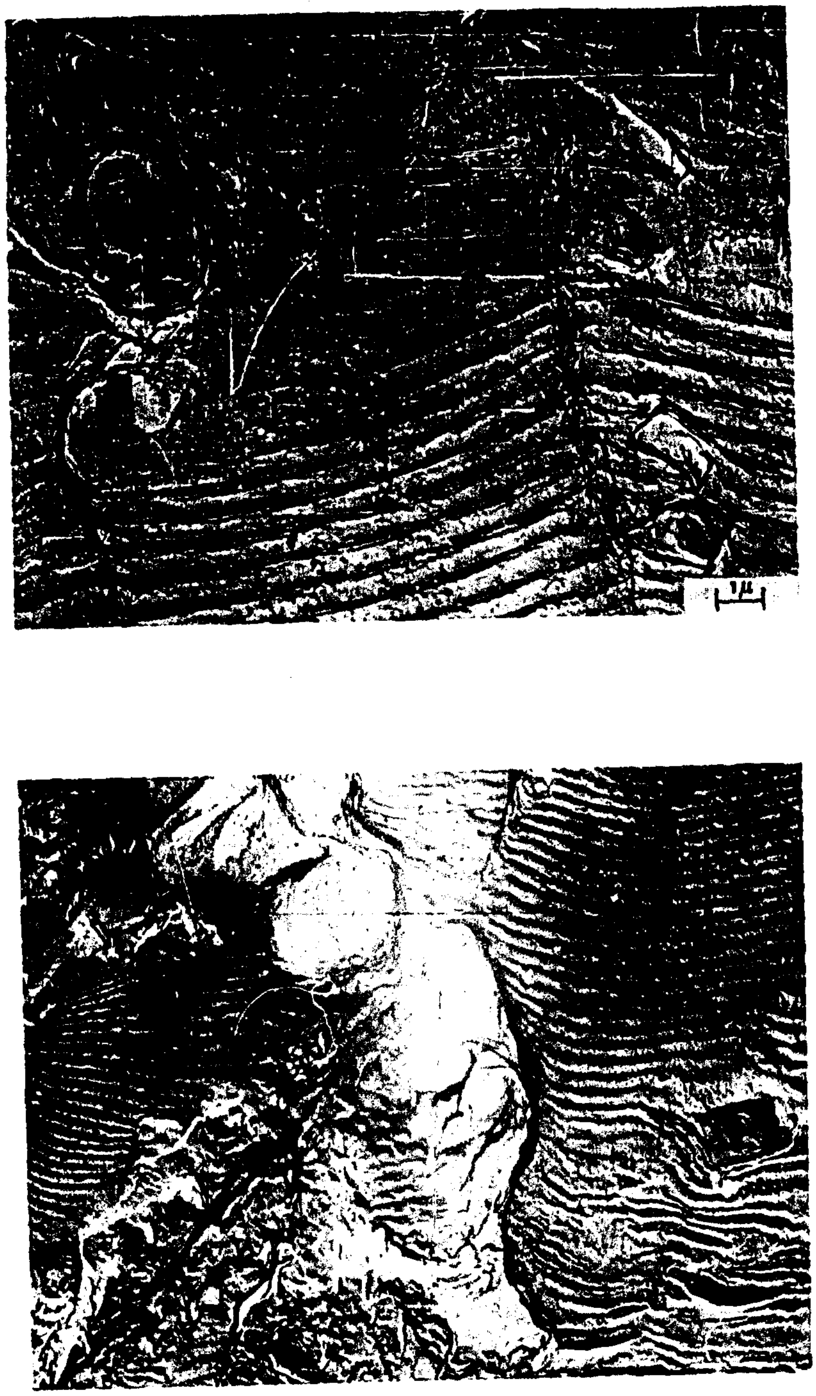

Figure 31 (a,b)- FATIGUE FRACTURE - Examples of ductile fatigue striations and brittle fracture of second phase particles in a 7178 aluminum alloy (two step replica).

Figure 32 - FATIGUE FRACTURE - Under a random load the spacing of the striations is not uniform. The influence of peak overloads on the rate of advance of the front is clearly indicated - 7178 aluminum alloy. (two step replica).

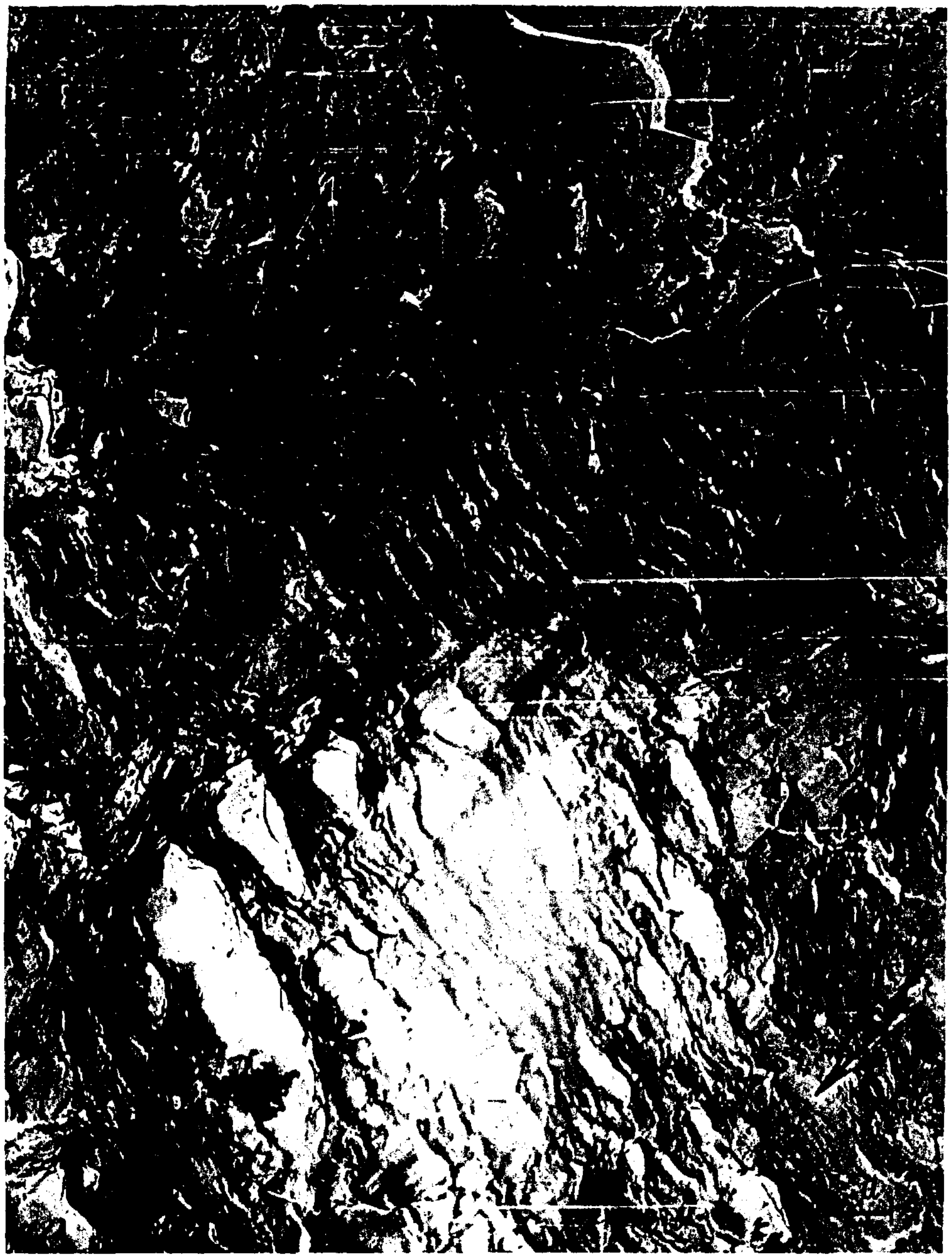

Figure 33 - FATIGUE FRACTURE - Fatigue striations in a 4340 steel. The striations are never well defined over the whole fracture surface (two step replica).

Figure 34 - FATIGUE FRACTURE - Fatigue striations in Ti - 6 Al - 4 V alloy (two step replica).

Figure 35 - FATIGUE FRACTURE - Fatigue striations in a welded zone of a 2219 aluminum alloy. The large second phase particles are probably eutectic particles of Al_2Cu (two step replica).

Figure 36 - FATIGUE FRACTURE - Fatigue striations in a zirconium alloy (two step replica).

This is often the case with service failure where a fatigue mode of fracture is identified by the presence of a few striations on a flat surface and the absence of other characteristic fracture features. Crack propagation under cyclic load is not always transgranular with a striated appearance. In some cases, fatigue cracks have been observed to propagate along grain boundaries. 70-30 brass was already given as an example of this type of behavior which can also take place in steels.

In many cases, a misinterpretation of some fracture features can lead to some confusion. For instance, Wallner's lines[23] should not be confused with fatigue striations. Wallner's lines are usually observed in very brittle materials; they are the result of an interaction between the advancing crack front and an elastic wave propagating at the same time. They appear as uniformly spaced striations but with an occasional criss-crossing network of lines (Figure 37). It very often happens that in a composite material, fracture takes place at the interface between large second phase particles and a matrix. Such a case is shown on Figure 38. The sharp lines and facets which are growth markings should not be confused with fatigue striations.

VIII. CONCLUSION

In summary this short review shows that the fracture surfaces of metals and alloys exhibit markings and a topography which are characteristic of the modes of fracture operating during the

Figure 37 - WALLNER'S LINES - Examples of Wallner's lines resulting from the brittle failure of a silicate particle in an Al-Si alloy (two step replica).

Figure 38 - FRACTURE ALONG AN INTERFACE - Cleavage fracture and separation along the interface of large particle in an Alnico alloy. The smooth and hexagonal steps are growth markings present on the particle before agglomeration (two step replica).

initiation and the propagation of the crack. These markings can be easily and best differentiated by electron microscopy.

The basic modes of fracture are separated into two classes: intergranular fracture and transgranular fracture. Figure 39 illustrates in conclusion, the different profiles of the fracture surfaces which result from these different modes of fracture.

Transgranular fracture takes place either:

- by cleavage along crystallographic planes,
- or by the formation and coalescence of microvoids which leave concave depressions called "dimples" on both surfaces of the fracture,
- or by the slow progression of a crack front under cyclic loads.

This last mode seems to be only partially controlled by the crystalline nature of metals.

Intergranular fracture is either:

- brittle when it is the result of a perfect separation of the grains along the grain boundary planes,
- or ductile when this separation is associated with the plastic formation of voids along the grain boundaries.

In general, one, two or more of these modes of fracture may be juxtaposed on a given fracture surface according to the nature

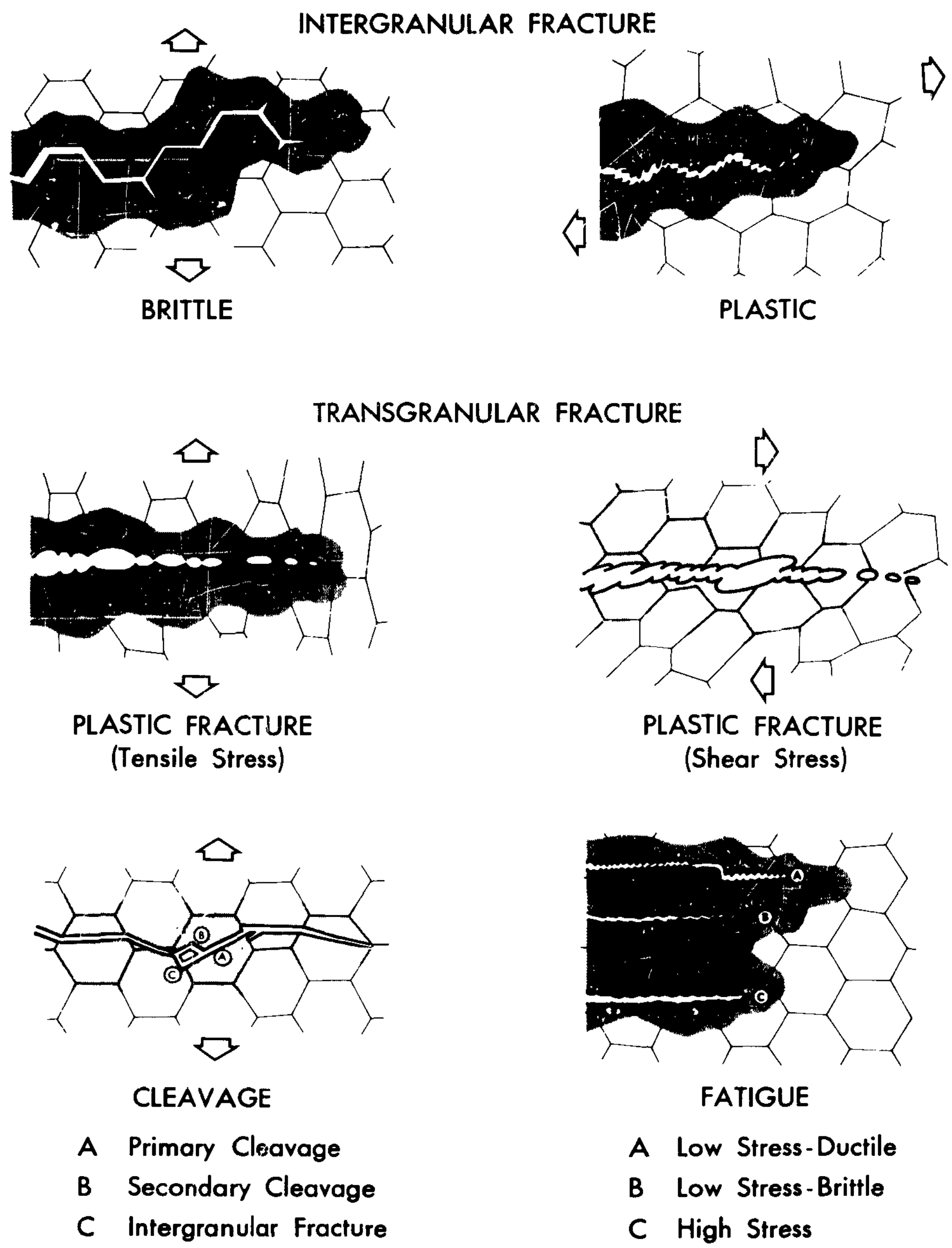

Figure 39 - MODES OF FRACTURE - The most common modes of fracture are represented on sketches a to f. In these sketches the crystallographic unit is represented by the grain. The same modes of fracture are true for smaller crystallographic units such as the twins, or the martensite needles or the second phase crystals.

of the material and the fracture conditions.

The results obtained so far by the techniques of electron fractography show the great possibilities offered by using the electron microscope to study fracture surfaces. In the analysis of service failures these techniques have been, and are today, a most valuable tool. It is now possible to determine on a microscale the local modes of fracture, the main origin and the sites of secondary crack initiation and also to follow the direction of crack propagation. The influence of crystal defects and precipitates on the initiation and propagation of the crack can be studied directly and easily at high magnifications.

Although electron fractography is becoming a common tool among the metallurgical laboratory techniques, a large amount of basic research remains to be done in order to get a complete understanding of the mechanisms of fractures which lead to the formation of fracture surface markings. This research should have a very high yield to investment ratio, the replicating techniques are simple, reliable and cheap; the electron microscopy work does not require a high resolving power and can be carried out with the less sophisticated electron microscopes. Finally, there will be a lot of fractures available for a long time.

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General Outlook on Electron Fractography

The study of the features and topographies of fracture surfaces is only a part of the general research program aimed at understanding the mechanisms of fracture. So far, this study has been, in general, limited to a purely qualitative evaluation and classification of the modes of fracture. It is time now to try to relate the size, shape and distribution of the fracture features to the quantitative measurements of fracture properties such as fracture strength, toughness and ductility. In order to coordinate this research effort, a subcommittee on microstructure and fractography was created recently within the A.S.T.M. Committee on fracture testing of high strength metallic materials. This subcommittee will have to face the following tasks:

1) evaluate the different techniques used in electron fractography and standardize the procedures of preparation and viewing of fracture surfaces or of replicas,

2) define an unambiguous nomenclature to describe the topography and features of fracture surfaces,

3) prepare an unambiguous classification of the modes of fracture based on a differentiation between fundamental fracture mechanisms,

4) plan and coordinate with the discipline of fracture mechanics, a general research program of the mechanisms of fracture.

APPENDIX

Replicating Techniques

ONE STEP - DIRECT CARBON REPLICA[1]

The fracture surface is cleaned two or three times with a film of cellulose acetate or by some other technique and carbon is evaporated directly on the surface in vacuum. The carbon film is removed by one of the three following procedures:

Electropolishing - The specimen is electropolished at a low current density until the carbon film floats in the solution. In the case of fracture surfaces, the film usually breaks at the sharp edges. It is difficult to obtain a large replica.

Dissolution of the metal in a solution of 10% of bromine[2] - in alcohol takes place without any gas evolution. The carbon film floats off the surface after 2 or 3 hours.

Etching - The metal is removed by the same etching reagent used for a micrographic attack and the carbon replica should float to the surface. Very often, second phase particles and carbides are extracted with the carbon replica and can be analyzed by electron diffraction.

TWO STEPS REPLICATING TECHNIQUE USING A PLASTIC FILM

The plastic film most commonly used in electron microscopy to replicate metal surfaces is a cellulose acetate film. To prepare the replica, a piece of acetate film is applied to the fracture surface which has been previously coated with a replicating solution made up of acetone containing some cellulose acetate in solution.

The acetone partly dissolves the film which is then pressed onto the surface forming a plastic negative. The film is allowed to dry under pressure for 5 minutes and is then stripped off very carefully. The positive replica is prepared by shadowing this plastic negative replica with germanium (at 45°) and then carbon (normal to the surface) in a vacuum evaporator. The replica is oriented in such a way that the shadowing direction of germanium is parallel to the general direction of crack propagation. This procedure will help in the orientation of the electron micrograph and in the understanding of the features observed.

Small squares (1/8") are cut off the replica in the regions of interest and the cellulose acetate is dissolved slowly in a mixture of acetone and distilled water progressively enriched in acetone. The replica is allowed to wash for a few hours in pure acetone and is then ready for viewing.

1. E. Smith and J. Nutting, Brit. J. Appl. Phys., 7, (1956) pp. 214

2. G. Henry and J. Plateau, Proc. Fourth Int. Conf. on Electron Microscopy, Berlin, 1958 (Springer-Verlag, 1960).

REFERENCES

1. C. A. Zapffe and M. Clogg, Trans. Am. Soc. Metals, 34, 71 (1945).

2. C. Crussard, R. Borione, J. Plateau, Y. Morillon and F. Matray, J. Iron Steel Inst. (London), 183, 146 (1956)

3. C. Crussard, J. Plateau, R. Tamhankar, G. Henry and D. Lajeunesse, A Comparison of Ductile and Fatigue Fractures, Fracture (Swampscott Conf., 1959), John Wiley and Sons

4. P. Forsyth and D. Ryder, Aircraft Engineering, 32, 96 (1960)

5. C. Laird, G. C. Smith, Phil. Mag. 7, 847 (1962)

6. G. Y. Chin, W. F. Hosford, W. A. Backofen, M.I.T. Reprint 16C - 184. Metallurgy Deparrment.

7. C. D. Beachem, B. F. Brown, A. J. Edwards, NRL Memorandum Reports 1432, June 1963.

8. J. Low, A Review of the Microstructural Aspects of Cleavage Fracture, Fracture (Swampscott Conf, 1959), John Wiley and Sons

9. C. D. Beachem, Transactions ASM, 56, 318, (1963)

10. N. A. Tiner, Am. Soc. for Test. Mat., 82 (1961)

11. J. A. Kies, A. M. Sullivan, G. R. Irwin. J. Appl. Phys. 21, 716 (1950)

12. J. Gurland, J. Plateau, Trans. ASM, 56, 442, (1963)

13. H. C. Rogers, Discussion in Fracture, Technology Press, M.I.T. Cambridge, Massachusetts, 1959, 46, 446, 559

14. Metal Interfaces, ASM, Cleveland, 1952, Figure 19, pp. 90

15. J. Plateau, G. Henry, C. Crussard, Revue Metallurgie 54, 200 (1957)

16. N. J. Grant, Intercrystalline Failure at High Temperature, Fracture (Swampscott Conf., 1959), John Wiley and Sons

17. R. C. Gifkins, Mechanics of Intergranular Fracture at Elevated Temperatures, Fracture (Swampscott Conf., 1959), John Wiley and Sons.

REFERENCES (Cont'd)....

18. A. Phillips and G. Bennett, Metal Progress, Vol. 79, 5, 97 (1951)

19. C. Zapffe and C. Worden, Trans. ADM, 43, 958 (1951)

20. P. J. E. Forsyth, Acta. Met. 11, 703 (1963)

21. C. Laird, G. C. Smith, Phil. Mag. 7, 847 (1962)

22. A. J. McEvily, R. C. Boettner, T. L. Johnston paper presented at the 10th Sagamore Army Materials Research

23. H. Schordin, Velocity Effects in Fracture, Fracture (Swampscott Conf., 1959) John Wiley and Sons.

24. A. H. Cottrell, Proc. Roy. Soc. Series A, 1364, Vol. 276, p.1

25. G. T. Hahn, B. L. Averbach, W. S. Owen, M. Cohen, Initiation of Cleavage Microcracks in Polycrystalline Iron and Steel. (Swampscott Conf., 1959) John Wiley and Sons.