SSC-74

ELECTRON MICROSCOPE TECHNIQUES FOR STUDY OF FRACTURED SURFACES OF SHIP PLATE STEELS

by

A. Revere

and

R. Jaccodine

SHIP STRUCTURE COMMITTEE

SHIP STRUCTURE COMMITTEE

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ADDRESS CORRESPONDENCE TO:

Secretary
Ship Structure Committee
U. S. Coast Guard Headquarters
Washington 25, D. C.

January 23, 1959

Dear Sir:

The Ship Structure Committee has sponsored a study at the Stevens Institute of Technology directed toward utilizing the electron microscope to further the understanding of the micromechanism of fracture in ship plate steels. Herewith is the Final Report, SSC-74, of this project, entitled "Electron Microscope Techniques for Study of Fractured Surfaces of Ship Plate Steels" by A. Revere and R. Jaccodine.

This project has been conducted under the advisory guidance of the Committee on Ship Steel of the National Academy of Sciences-National Research Council.

This report is being distributed to individuals and groups associated with or interested in the work of the Ship Structure Committee. Please submit any comments that you may have to the Secretary, Ship Structure Committee.

Sincerely yours,

E. H. Thiele
Rear Admiral, U. S. Coast Guard
Chairman, Ship Structure Committee

Serial No. SSC-74

Final Report
of
Project SR-122

to the

SHIP STRUCTURE COMMITTEE

on

ELECTRON MICROSCOPE TECHNIQUES FOR STUDY OF FRACTURED SURFACES OF SHIP PLATE STEELS

by

A. Revere and R. Jaccodine

Stevens Institute of Technology
Hoboken, New Jersey

under

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Washington, D. C.
National Academy of Sciences-National Research Council
January 23, 1959

ABSTRACT

This project was directed toward developing and evaluating the use of replica techniques in the electron microscope study of fractured surfaces of ship plate steels. Its ultimate aim was to further the understanding of the micro-mechanism of fracture. Using 3/4-in. Navy tear test specimens, an iron single crystal specimen formed by the carbonyl process and a specimen of actual ship plate failure taken from a C-2 cargo ship, a satisfactory electron microscope replica technique for use directly on fractured metal surfaces has been demonstrated. This technique was found to be reproducible and capable of manifesting fine and coarse structures. It produces electron micrographs which give structures that can be measured, defined and classified.

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INTRODUCTION

The problem of fracture and the mechanism of fracture have been discussed and investigated for many years. As stated by Jonassen,[1] the brittle failure of ship plate has been of great concern to the nation. Accordingly, this project concerned itself with the development and use of electron microscope replica techniques on fractured steel specimens. A brief review of the published work on fracture in metals may be helpful in the consideration of the electron micrographs presented in this report.

Various theories have been proposed to account for the brittle behavior of normally ductile metals. Briefly, Griffith[2] proposes a theory that attributes weakness of bodies to indefinite flaws in the solids which are assimilated into submicroscopic cracks. The cracks propagate when energy conditions are favorable. Griffith also postulates a critical size and critical stress necessary for propagation, which he has substantiated and broadened by working with glass plates and rods. This theory has been well accepted and forms the basis for the work of others.

G. Irwin[3] has carried forth the fracture velocity studies of the David Taylor Model Basin and those suggested by N. F. Mott at the Strength of Solids Symposium, held at Bristol University, England.[4] He uses crackhead contours in the path of fracture as a means of indicating the different rates of fracture. Using the energy considerations given by Griffith and Mott, he set up criteria to separate the ductile from the cleavage portions of crack advance.

Various investigators have worked on the aspect of the exact origin of fracture in metals. Gensamer[5] showed that the fracture starts at the center of the specimen in cases where necking occurs prior to fracture. Zener,[6] in his proposed micromechanism of fracture, states that in metals, as in all crystalline material, deformation does not proceed in a homogeneous manner. He believes that microcracks originate because of the heterogeneous nature of plastic deformation. If, as shown by Kê,[7] grain boundaries behave in a viscous manner with no relative motion with respect to grain corners, then these corners act as keys, limit the displacement of adjacent grains, and become sites of intense concentration of stress. Considerable hydrostatic tension develops that cannot be relieved plastically, so it becomes more intense through shearing stress relaxation. A crack is initiated when the theoretical fracture stress is reached.

When plastic deformation occurs in the interior of the grains, it is confined to regions known as slip bands. These slip bands may be stopped either by grain boundaries or precipitate plate. Grain boundary stoppage may cause relaxation of shear stress behind the spearhead, which results in an increase of stress in front of the spearhead. That in turn leads to the initiation of new slip bands in the next grain. In the case where a slip band is stopped by a precipitate plate, local separation of atoms (a crack) may occur.

Zener also mentions the atomistic approach by introducing the mechanism of slip band nucleation in coalesced dislocations. He thus gives a plausible atomistic picture of how cracks can originate. The main point of this work is based on the premise that fracture is intimately associated with the discontinuous nature of deformation.

In a paper on the fracture of ferrite single crystals, C. F. Tipper and A. M. Sullivan[8] examined the actual surfaces of cleavage fracture to gain clues as to the behavior pattern. The surfaces themselves consisted of planes of several levels. They found markings of two kinds: coarse striae, which are associated with Neumann lamellae, running nearly parallel to the plane of the plate, and fine markings which assume a variety of patterns. The striations running from inclusions gave evidence of possible initiation of fracture.

Light micrographs on this work illustrate the possible position of crystal boundaries before recrystallization. A structure is found between lamellae, both in plastics and in glass, which leads to the belief that the velocity of propagation of fracture is greatest in the center of the bands (herringbone shape). This is considered direct evidence against the theory that fracture propagates along interfaces of Neumann lamellae and the unaffected metal. By examination of the ends of cracks, evidence was found supporting the fact that there is some plastic deformation even when fracture is chiefly "crystalline" in appearance.

Kies, Sullivan and Irwin[9] have interpreted the herringbone markings and have demonstrated that, on the basis of level differences, the fracture initiation point is close to the focus of the parabola-like figures and the streamers radiating from the focus trail in the direction of crack propagation. They have been able to simulate the chevron markings in ship plate by a tearing fracture in a cellulose acetate model. The initiation of fracture foci and the joining of levels to extend fracture were conveniently studied using these analogs. Sullivan and Kies[10] have also shown striking similarities, even exact duplications, of fracture surfaces of various materials.

That the various manifestations of fracture, as reflected by the surface markings, can yield valuable information is clearly illustrated by the conclusions given in the above mentioned papers.

Electron microscopy has been applied to the study of the development of fatigue failures by W. J. Craig.[11] The object of his investigation was to gain additional information about the process of crack formation and to develop some concept of the mechanism of fatigue by studying the deformation characteristics of simple substances. Materials under study included ingot iron, alpha brass, and aluminum. The work on alpha iron shows the irregular nature of the deformation process, resulting probably from simultaneous slip in three slip systems. There is evidence of fine striations within the deformed brass. Again, the conclusion points to progressive growth of fracture by the joining of microcracks. The exact cause of crack initiation still remains unknown, although there is evidence that cracks seem to grow from local deformed or fragmented paths. These streaks have the general direction of the slip bands, but they are of greater width and appear rippled and fragmented on the surface.

C. A. Zapffe[12] uses a new technique which he calls "fractography" (the descriptive treatment of fracture) to study fracture. From the appearances of the metal, the resistance of the steel to deformation and fracture can be inferred. Rough, highly inflected paths appearing after cleavage (called "oak leaf" or "coral" pattern) are indicative of tough materials, whereas flat, expansive and crystallographically marked facets are indicative of considerably lower resistance to fracture.

For several years, Zapffe and his co-workers[12] performed a thorough microscopic study on the metal grains of ship plate steel specimens that had cleaved at liquid nitrogen temperature. Fractographs contained in their report on these specimens illustrate the diversity of the patterns involved. Although these authors have employed the light microscope to the limit of its resolution, they observed that a vastly intricate substructure containing the secret of plastic deformation and phase precipitation remains to be explored.

ELECTRON MICROSCOPE

Because the wave length of the electron beam is one hundred thousand times shorter than the wave length of ordinary light, the electron microscope permits far greater resolution than can be obtained in the light microscope. It can therefore be used as a tool to explore such unknown substructure as that mentioned by Zapffe in his work on ship plate steels.

Since metal specimens cannot be placed directly in the electron microscope, replicas of the specimens' surfaces must be prepared. This follows from the fact that the electron microscope forms the image by transmission of the electron beams rather than by reflection as in the optical microscope. Although the surfaces of conventionally polished and etched metal specimens had been successfully replicated, little or no attempt had been made to prepare replicas on rough fracture surfaces. For this reason, the present project was undertaken to develop the use of replica techniques for the study of rough fracture surfaces of ship plate steels.

REPLICA TECHNIQUE

The use of polystyrene in making replicas of metal surfaces for examination in the electron microscope was reported by Heidenreich and Peck[13] and later reviewed by Gerrould.[14] Polystyrene is chemically inert and has high dimensional stability. The extreme mobility of silica condensing from vapor on the polystyrene captures accurately the shape and fine detail in the microstructure of the metal specimen.

In general, the technique is a two-step positive process (a detailed procedure is given in the appendix). The first step molds the polystyrene against the surface of the metal specimen, and the second evaporates, under vacuum, a thin film of silica on the top of the polystyrene replica--i.e., the side of the plastic in contact with the specimen surface. The polystyrene is then dissolved, leaving the thin silica replica for use in the electron microscope. This may be used directly or it may first be "shadowed" with metal, such as chromium, to increase contrast.

This technique is called a positive replica because the elevations in the original specimen are reproduced in the silica replica. Any doubt arising as to the irregular surface of the structure may readily be settled by stereoscopy.

SPECIMENS

The specimens, produced at different times during the project, were placed in desiccators and taken directly to Stevens Institute of Technology. Replicas were made on them immediately to keep oxidation or change in the fracture surfaces at a minimum.

The samples of A and C steels that had been polished and etched by the New York Naval Shipyard for standard light microscopy were replicated and studied under the electron microscope. In addition, the investigation included study of 1) an iron single crystal specimen formed by the carbonyl process and submitted by Dr. C. F. Tipper of Cambridge University and 2) a specimen of actual ship plate failure taken from a C-2 cargo ship together with tear test specimens of the ship plate.

Project steels "A" and "C" of the following compositions and transition temperatures were used for this investigation:

	Composition, per cent					Navy Tear Test Transition Temperature
	C	Mn	Si	Al	N	
Project Steel A	0.25	0.49	0.04	0.004	0.004	70 F
Project Steel C	0.25	0.51	0.05	0.015	0.009	135 F

These 3/4-in. Navy tear test[15, 16] specimens were broken at the New York Naval Shipyard at the following temperatures:

Project A Steel (degrees Fahrenheit)	Project C Steel (degrees Fahrenheit)
-100	-100
	50
10	10
	50
90	90
	120
150	150
	180
210	210

DISCUSSION

All replicas and micrographs were taken on the fractured surface of each specimen in an area approximately 3/4 in. x 3/4 in. located about 1/2 in. beyond the "thumb nail" region near the notch. An exception was made on the A steel specimen tested at 10 F: the actual thumb nail itself was micrographed and studied. Figure 1 shows a comparison of both faces of the broken specimen in the thumb nail area (shear area) tested at 10 F.

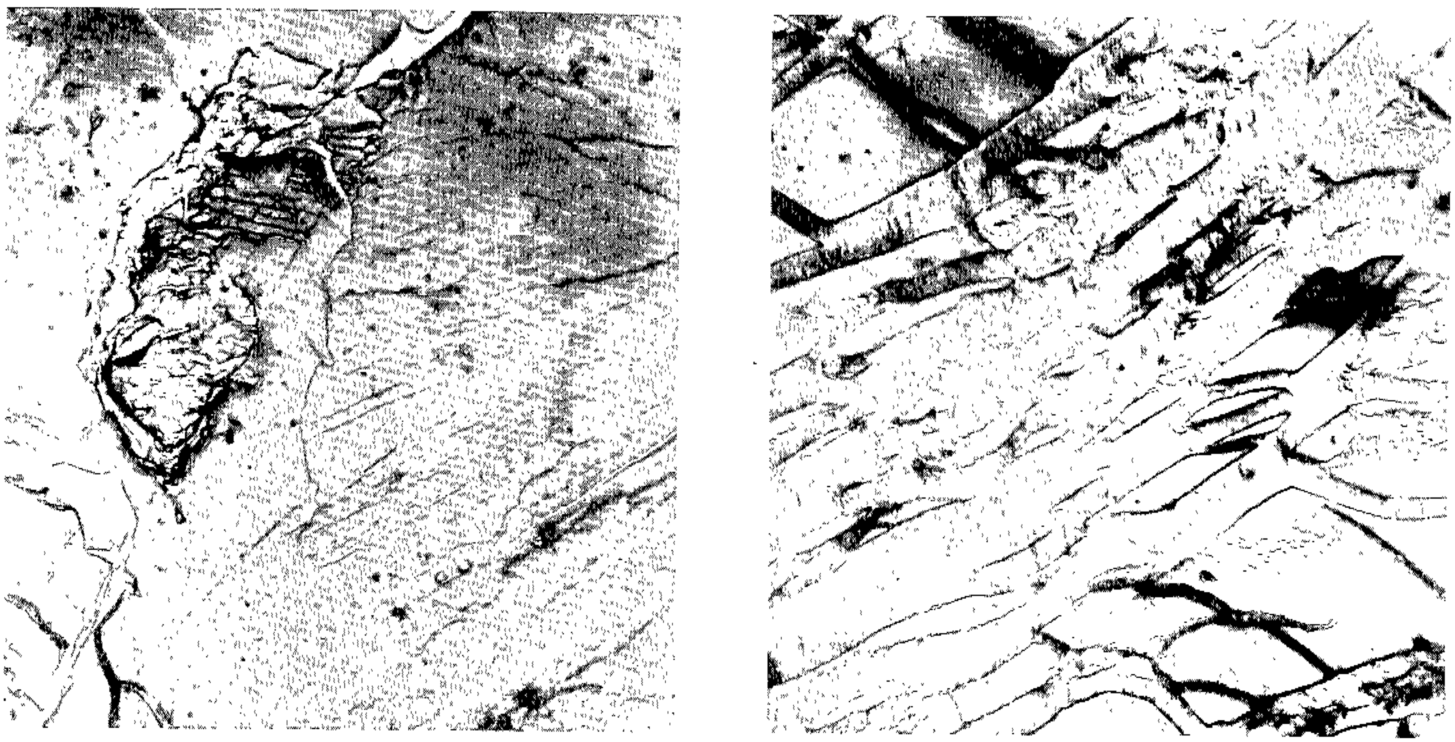

Figure 1. Electron Micrographs of the Thumbnail Area of Both Fractured Surfaces of a Tear Test Project A Steel Specimen Broken at 10 F. (X5000)

In order to determine whether any change might take place in the specimen surface, the second half of the A steel specimen tested at 10 F was examined in the brittle area at a later date. The microstructure of the latter half indicated no structure change in the fracture surface. A comparison of the micrographs for these tests can be seen in Fig. 2. The reproducibility of this specimen also served to indicate the reliability of the replica technique.

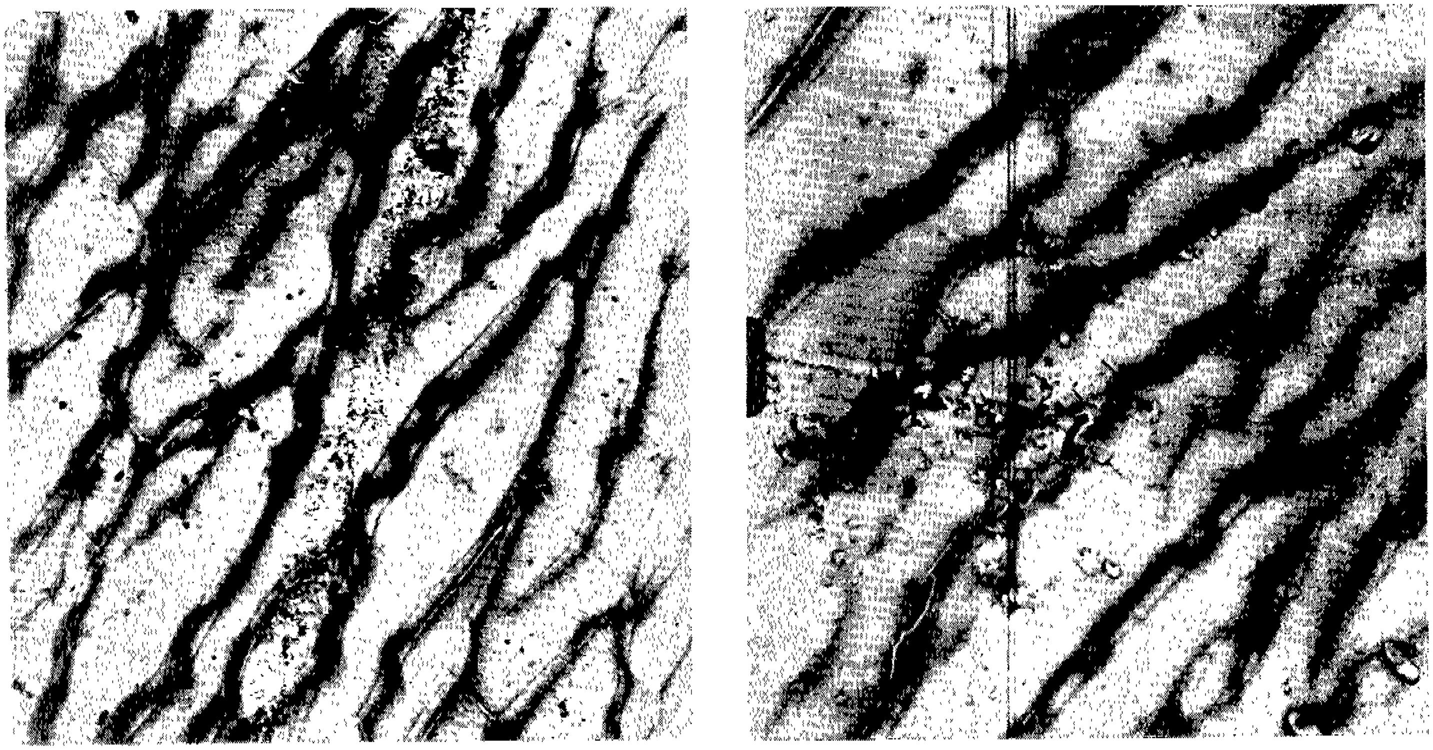

Figure 2. The Fractured Surfaces of a Tear Test A Steel Specimen Broken at 10 F Were Micrographed in the Brittle Region, the Left Micrograph First and the Right at a Later Date, to Indicate the Reliability of the Replica Technique. (X5000)

It should be noted that areas photographed in the electron microscope are representative and show structures that were found to be typical in at least seventy-five per cent of the surfaces surveyed.

Figure 3. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken at -100 F. (X5000)

Figures 3 through 11 are micrographs of C steel specimens that had been broken at each temperature noted in the preceding section. These micrographs

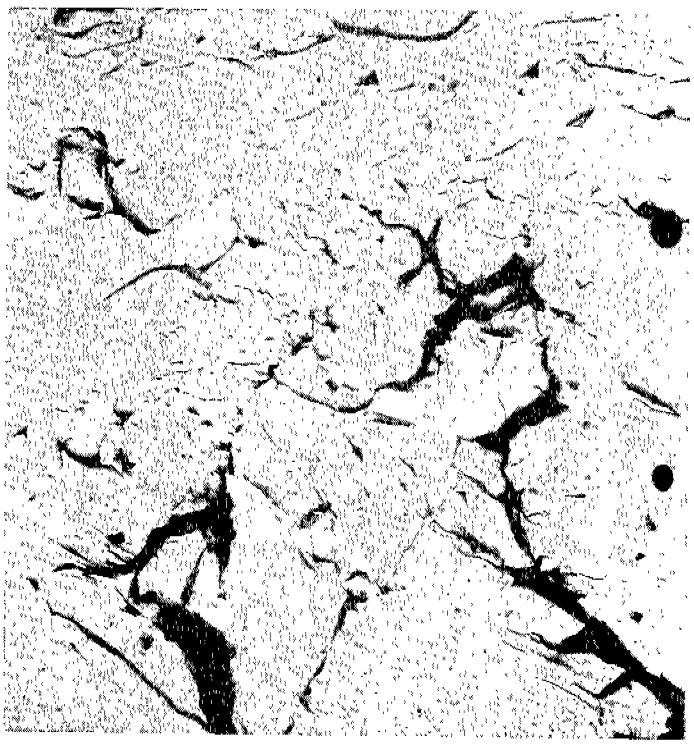

Figure 4. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken at -50 F. (X5000)

establish with greater certainty the reproducibility of the replica technique by demonstrating definite trends of structures as the test temperature is varied.

Figure 5. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken at 10 F (X5000)

Figure 6. Fracture Surface of a Project C Steel Navy Tear Test Specimen Broken at 50 F (X5000)

Figure 7. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken at 90 F (X5000)

Figure 8. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken at 120 F (X5000)

Figure 9. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken at 150 F (X5000)

Figure 10. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken at 180 F (X5000)

Figure 11. Fractured Surface of a Project C Steel Navy Tear Test Specimen Broken At 210 F (X5000)

The electron micrographs in Fig.12 were taken on replicas made on the rough fracture surface of the iron single crystal. Dr. Tipper compared

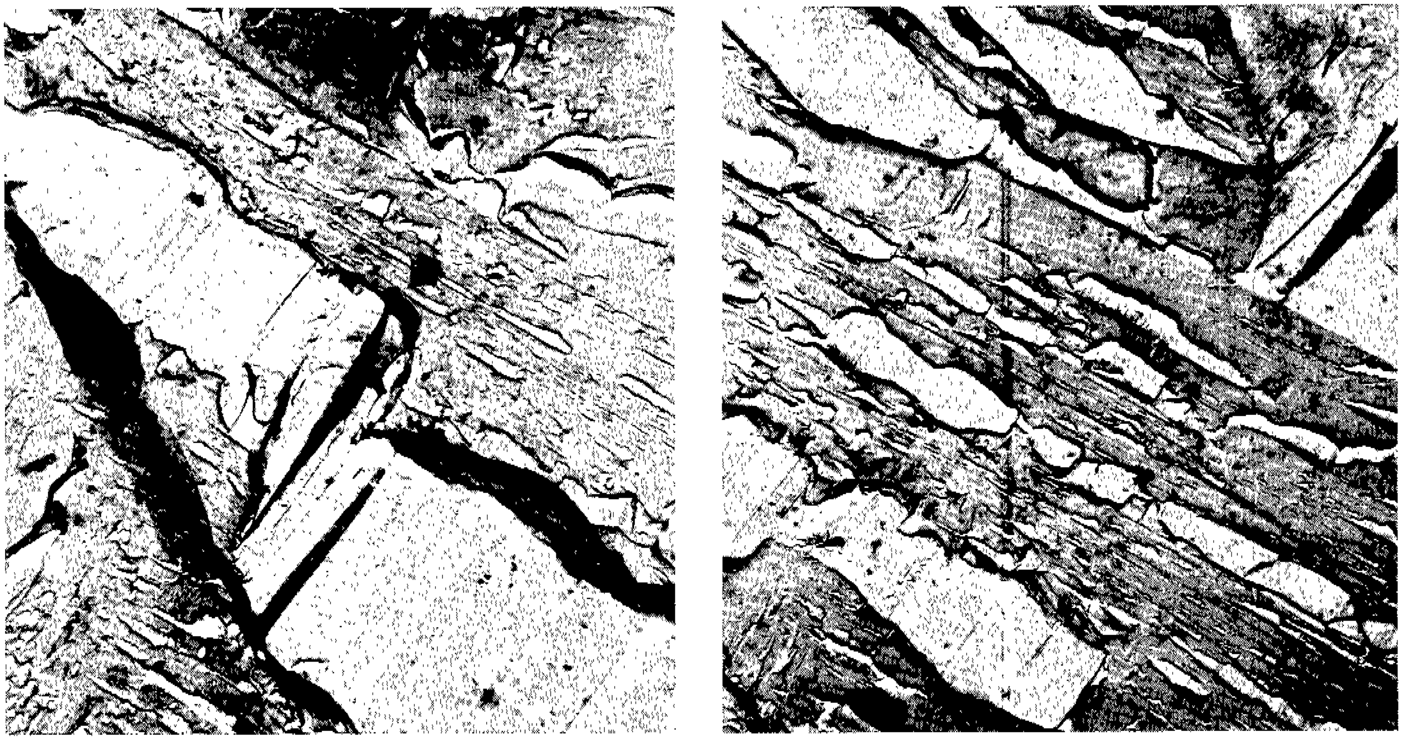

Figure 12. Two Electron Micrographs of an Iron Carbonyl Single Crystal Submitted Through the Courtesy of Dr. C. F. Tipper. (X5000)

these micrographs with similar test specimens viewed under an ordinary metallurgical microscope. She noted that the coarser marks shown on the micrographs appear to be the fine marks observed on specimens under the microscope. This illustrates the ability of the replica technique to capture the fine structure of fracture surface and the fact that such coarse microstructure as seen for the first time in the electron microscope can be interpreted and correlated with the fine structure that has been identified under the light microscope.

In order to compare specimens from a service failure with those of the tear test, micrographs were taken from specimens of an actual C-2 cargo ship plate failure (Fig. 13). The specimens were obtained within a few days after the failure. It is unfortunate that the temperature at the time of the failure was not recorded.

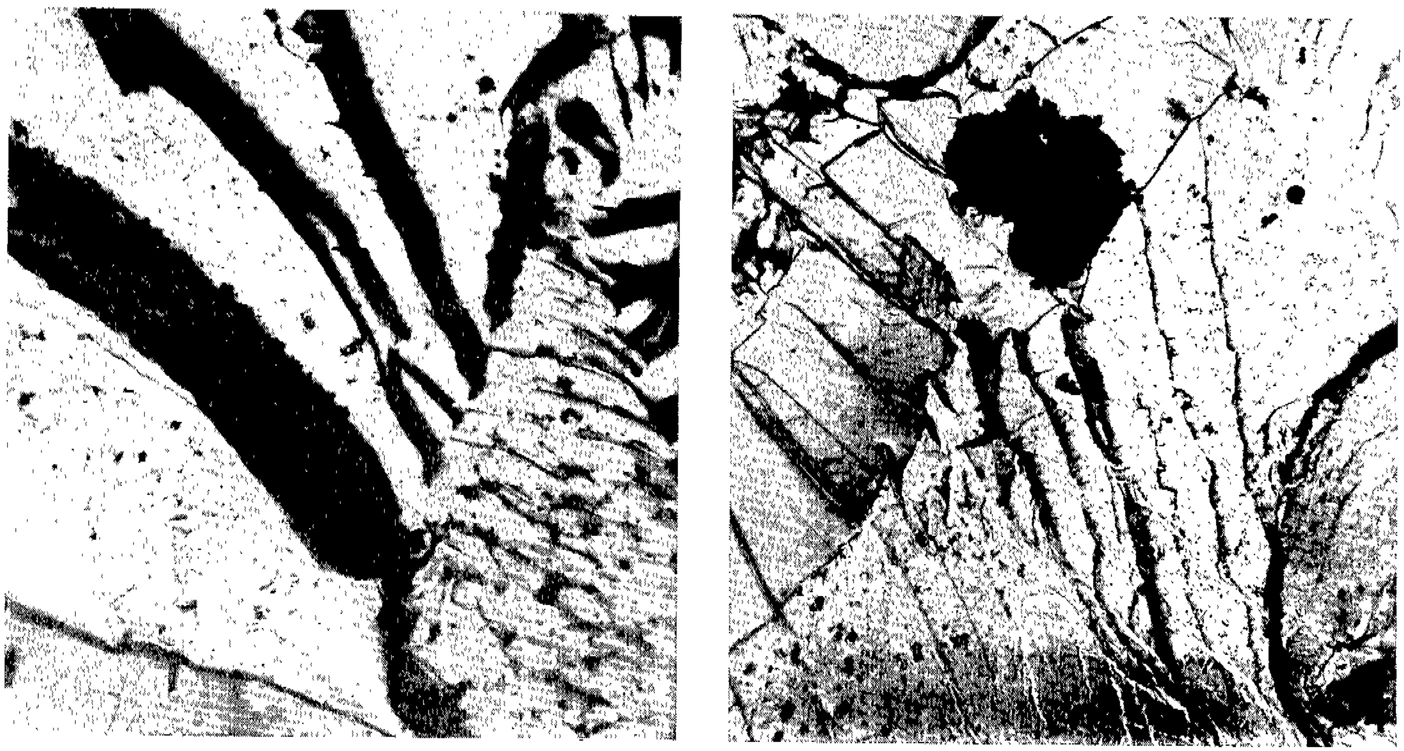

Figure 13. Micrographs of Fractured Surfaces of Ship Plate Steel Obtained Within a Few Days After the Failure of a Cargo Ship. (X5000)

Figure 14. The Polished and Etched Surface of a Project A Steel Specimen Micrographed to Show Fine Detail. (X5000)

Figures 14 and 15 are micrographs of polished and etched A and C steel specimens. These serve to indicate that a rough fractured surface is not necessary for an examination of detail; fine detail can be observed from a polished and etched specimen.

Figure 15. The Polished and Etched Surface of a Project C Steel Specimen Presents Fine Detail When Micrographed. (X5000)

CONCLUSIONS

The work under this project may be summarized as follows:

1. A replica technique can be applied successfully to rough fractured surfaces of ship plate steel.

2. The technique is reproducible. This is evidenced by studies of specimens broken at intermediate test temperatures, which show continuity of trends established in earlier tests. The later test of the mirror-image of the A steel specimen broken at 10 F also lends support.

3. This technique gives accurate reproduction of fine structure. Replicas made by this technique on polished and etched specimens show structures that are readily identified and correlated with those shown in conventional light micrographs made directly on the specimens.

4. The coarse and the very fine structures shown in replicas of both the rough fractured surfaces of ship plate steels and the iron single crystal specimen can be correlated with structures found on the specimens themselves under direct examination.

5. Electron micrographs on replicas give structures that can be measured, defined and classified.

Because fine structures--replicated and resolved for electron microscopy--are real, because they have some meaning, they must add new information that can be used for interpretation and eventual explanation in solving the problems of fatigue and brittle fracture in ship plate steel.

ACKNOWLEDGMENTS

The authors are indebted to Dr. C. F. Tipper, Dr. C. A. Zapffe, and the late Dr. Finn Jonassen for their aid and interest in this project.

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APPENDIX

POLYSTYRENE-SILICA REPLICATION METHOD
(Prepared by the New York Naval Shipyard)

1. Preparation of polystyrene negative-replica. The metal surface to be replicated is coated with a film deposited from a solution containing 1 per cent polystyrene dissolved in benzol. When the surface is dry, this operation is repeated twice and then a final coat of a 6 per cent polystyrene in benzol solution is applied.

2. Formation of polystyrene replica. A block of polystyrene, 5 to 10 mm thick and of an area sufficient to cover the replica, is cemented to the polystyrene film with methyl methacrylate monomer and heated at 65 C for one hour. The specimen and plastic are placed on dry-ice for 15 minutes and then pulled apart.

3. Formation of silica positive-replica. The plastic block is placed (replica side up) 7.5 cm below the center of a loop of 24 B & S gauge tungsten wire (15 cm in diameter) located in a vacuum-evaporation unit. After the wire is coated with a solution of Ludox,* the assembly is evacuated to a pressure below 10^{-3} mm of mercury, and a current of about 20-amperes is passed through the wire until evaporation of the silica is complete.

4. Shadowing of silica replica. The silica-coated plastic replica is placed 10 cm above a small graphite crucible containing 5 to 10 mg of germanium or chromium as the shadowing material. The plane of the replica is inclined 30° to the line connecting its center to the crucible. After evacuation to a pressure below 10^{-3} mm of mercury, the crucible is heated by a cone of 24 B & S gauge tungsten wire in which it is supported until an irridescent film of purple hue is formed on the silica surface. The time and temperature for the formation of this colored film must be found by experiment, but should be in the order of 15 seconds at 2700 C.

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*Ludox - Trade name for a colloidal silica solution supplied by E. I. duPont de Nemours and Company, Inc., Grasselli Chemicals Department, Wilmington, Delaware

5. Mounting of silica replica. The silica-coated plastic is immersed in a solution containing 85 parts ethyl bromide and 15 parts of benzol until the polystyrene dissolves sufficiently to allow the silica film to float free. A half-inch square of 200-mesh screen, held by a pair of forceps, is used to catch the silica film immediately after it is disengaged from the plastic. The film is washed by successive transfers to fresh ethyl bromide-benzol solutions. After washing, the film is examined under an optical microscope to locate unbroken areas, which are then punched out along with the supporting screen for mounting in the specimen holder of the electron microscope.