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A STUDY OF THE ROLE OF CARBON IN TEMPER-EMBRITTLEMENT AND THE EFFECT OF TEMPER-EMBRITTLEMENT ON THE FATIGUE PROPERTIES OF A 3140 STEEL

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PREFACE

Temper-embrittlement is one of the oldest, unsolved phenomenon in the metallurgical sphere of endeavor. Although its deleterious effects can be checked by proper alloying and heat treatment, a great deal of fundamental interest as to the cause and nature of the phenomenon still persists.

The author wishes to express his appreciation to the many people who have contributed to the success of this investigation.

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INTRODUCTION

The term temper-embrittlement has been used to describe the adverse effect on the impact properties of a tempered low-alloy steel when the material has been either slow cooled through a temperature region of 1250-700°F or isothermally held in this temperature range for an extended period of time.

Two methods have been used in the past for studying this phenomenon. The first was to compare the room temperature impact results obtained after different heat treatments. The liability of a particular steel to temper-embrittlement was expressed as the ratio of the notched bar impact properties after water quenching and after slow cooling, respectively, from the embrittling temperature. This ratio was called the "susceptibility ratio" of the steel and the larger the number the more susceptible was the steel to temper-embrittlement. The second method used, which has more validity, as has been demonstrated by Jolivet and Vidal,[93] is to carry out a number of impact tests over a range of temperatures for the tough and temper-embrittled conditions. This results in a transition temperature for the two conditions where fracture proceeds from a tough to a brittle manner. The difference in the transition temperatures for the two conditions is a measurement of the severity of temper-embrittlement.

Many criteria for establishing the transition temperature may be used. It may be defined as that testing temperature at which: 1) Brittle fracture first appears in the specimen, 2) the fracture shows a

given amount of brittle fracture, usually 50 percent, 3) the fracture energy is reduced to some arbitrary value, i.e., 15 or 25 foot-pound, and 4) the fracture energy is reduced to half the value required for completely ductile fracture. Whether one or the other is used for determining the transition temperature is immaterial, so long as the curves for the two conditions are compared on the same basis.

In spite of a large number of investigations and the accumulation of experimental data, there has evolved no consistent mechanism for explaining temper-embrittlement. Likewise, very little is known about the effect of temper-embrittlement on the fatigue properties of a susceptible steel. It was felt that a valuable contribution could be made to the first by limiting an investigation of temper-embrittlement to the role of carbon in the phenomenon, using carbon-14 as a tracer element in the steel. The fatigue aspect of susceptible steels was considered important in order to round out the picture on the effect of temper-embrittlement on the mechanical properties of steel.

STATEMENT OF THE PROBLEM

The problem that was studied in this investigation consisted of two aspects of temper-embrittlement. One aspect was concerned with the role of carbon in temper-embrittlement, while the other was concerned with the effect of temper-embrittlement on the fatigue properties of a susceptible steel.

In order to study both of these aspects, it was necessary to first establish a condition of severe temper-embrittlement so that a comparison between a ductile and temper-embrittled condition could be made.

The role of carbon in temper-embrittlement was studied from the point of view of how these atoms were distributed in the brittle condition as compared to the distribution in the tough material. For this purpose the technique of autoradiography was used, as well as the electron microscope, to more carefully study the size, shape, and distribution of the carbide particles for the two conditions of heat treatment.

The fatigue aspect of this investigation was performed with emphasis placed on determining the susceptibility of a temper-embrittled steel to damage due to a prestress at a level above the endurance limit for the material.

LITERATURE SURVEY

An excellent review of the literature on temper-embrittlement up to 1944 has been made by Hollomon.[67] Further work by Woodfine[146] has extended this compilation to 1952. Since that time there has been no survey on the subject published as a review.

Background

Apparently, the phenomenon of temper-embrittlement had been recognized, but not defined as such, as early as 1883. According to Howe[71] some blacksmiths used the practice of water quenching after tempering to avoid embrittlement experienced on slow cooling. This practice was then referred to as "water annealing". Various Krupp patents[102] established in 1900 also refer to a preferred procedure of handling nickel-chromium steels after tempering. With the coming of the First World War, when the demand for alloy steels for armor and ordnance was tremendously increased, the problem of temper-embrittlement began to receive serious attention. Among the very early experimenters on the subject were Greaves and his co-workers,[54,55,56,57] who accumulated considerable amounts of data on the effect of alloying elements on temper-embrittlement and the effect of temper-embrittlement on various mechanical and physical properties of susceptible steels. However, all of these data were obtained on the basis of a determination of a "susceptibility ratio" for different heat treatments. This method of study was later shown to be unsatisfactory by Jolivet and Vidal[93] when they proved that the notched-bar impact results are dependent on testing

temperature. These authors suggested the method of establishing the fracture energy-testing temperature curves for various conditions of heat treatment and measuring the degree of temper-embrittlement by the change in transition temperature between the resulting curves. This method seems to be the only reliable means of quantitatively measuring the degree of temper-embrittlement developed in a susceptible material and is the one currently used.

Means of Detecting Temper-Embrittlement

Although temper-embrittlement seems to produce substantial changes in the impact properties of a susceptible steel, it has little or no affect on other mechanical and physical properties. It has been reported that temper-embrittlement does not affect the hardness, yield point, tensile strength, or elongation as obtained in the tensile test.[55,56,57,68] Hollomon[68] has compared fatigue data obtained at a testing speed of 1750 rpm of a severely embrittled and non-embrittled SAE 3135 steel and found that the effect of the embrittlement was to shift the S-N curve toward smaller numbers of cycles. Further studies on the fatigue aspects of temper-embrittled steels were done by Kosting[101] at very slow cycling speeds. He found a considerable difference between fatigue properties of steels having different impact properties. However, in an investigation concerning the effect of temper-embrittlement on the endurance limit of an SAE 3140 steel, Jominy[94] found no effect.

In some severely embrittled steels the ductility as measured by the reduction in area in the tensile test may be reduced and some specimens exhibit a longitudinal "star" fracture instead of the usual cup and cone type.[67,104]

No evidence of precipitation or transformation has been observed by thermal analysis that could not be explained on the basis of relief of internal stresses in quenched specimens.[146] Specific gravity changes of specimens with and without temper-embrittlement have been reported by Greaves and Jones[56] to be less than one part in 20,000. Electrical and magnetic properties are also not affected by the embrittling process.[56,58]

Some X-ray evidence exists that correlates with the presence of temper-embrittlement. Maloof[109] has found that the lattice parameters for three types of steel specimens, furnace cooled, were less than those for the corresponding water quenched specimens. The maximum difference amounted to 0.0006 K X units. Residues from the tough and brittle samples were examined by X-ray diffraction. The residues were found to vary in composition, but a general decrease in chromium content was noted with increasing degrees of embrittlement. Bush and Siebert[22] have reported similar lattice parameter results in an isothermal study of temper-embrittlement of a 5140 steel in which a maximum difference of 0.0012 K X units decrease in lattice parameter was noted for a specimen embrittled 3,000 hours at 850°F. Further X-ray evidence has been reported which claims that the iron lattice lines obtained by back reflection techniques broaden in a severely embrittled structure.[115,140,141]

McLean and Northcott[116] have studied temper-embrittled materials with a dropping electrode using N/10 ferrous chloride and saturated calomel. Substantial differences were noted between tough and temper-embrittled samples. However, little correlation with other tests existed and so this method is not reliable for measuring the degree of temper-embrittlement.

Another method which has been used to differentiate tough and temper-embrittled specimens is the one suggested by Bückle, Jacquet, and Weill.[19,20,84] In this procedure a so-called Meyer's number is determined using micro-hardness measurements. If the Meyer's number exceeds a certain value, 2, then the material is brittle, if less, the material is tough. No theory exists as to the proper meaning of the Meyer's number; although, Jacquet and Weill[85] suggest that the higher the number, the higher is the internal strain in the specimen.

Observations with the electron microscope have been made on some temper-embrittled steels by Maloof[109] and Woodfine.[147] Their findings show a precipitate in the prior austenite grain boundaries for the brittle condition, but no continuous network seems to be present.

A unique method for distinguishing non-brittle from brittle specimens has been reported by Jaoul and Lacombe.[91] In this method specimens are etched in a radioactive dilute phosphoric acid solution. Surface counts of the specimens are made with a Geiger-Muller tube as a function of depth from the surface as successive layers are removed by mechanical polishing. The results show that the activated phosphoric acid penetrates the brittle samples to a larger extent than the ductile samples. The authors claim that this supports the hypothesis of the presence of a constituent in the boundaries and sub-boundaries in a temper-embrittled specimen.

Finally, several metallographic techniques for determining the presence of temper-embrittlement have been developed. These methods all hinge on the development of a suitable etching reagent which attacks a brittle material differently than a non-brittle material. As has been pointed out by many investigators,[25,56,58,116] no differences between

tough and brittle micro-structures are discernable when normal etching solutions for steels are used.

However, using several special etches developed recently the grain boundaries of an embrittled steel can be selectively etched; whereas, the boundaries in the tough state are unaffected. The boundaries that are attacked have been shown to be the prior austenite grain boundaries.[22,147] Among these etches is the one used by Cohen,[25] composed of picric acid, ether, and zephiran chloride. Another, proposed by Jacquet,[79,80,81,82,83] consists of a saturated picric acid solution in ether. Furthermore, it has been established that the type of brittle fracture in a temper-embrittled steel is intercrystalline compared to transcrystalline for the non-embrittled specimen.[6,7,8,22,80,121]

Of all the methods that have been proposed for determining temper-embrittlement, only one, the change in transition temperature for tough and brittle samples, has been used to a limited success for a quantitative determination of the degree of embrittlement. However, there is some question as to how quantitative even this method actually is. Vidal and Jolivet[93] suggested that the degree of embrittlement was the same whether the test was a slow bend or impact test at a constant temperature. On the other hand, Jaffe and Buffum[89] have shown that for a given degree of embrittlement, the difference in transition temperature varied with the method of testing, depending upon whether the test was a slow bend or impact fracture test. Furthermore, Hultgren and Chang[72] have shown that the difference in transition temperature produced using a V-notched Charpy specimen was different from that produced using a keyhole-notched impact specimen. It therefore seems that no true measure of temper-embrittlement can be established unless it is

invarient with respect to the test method. Brown[18] has recently proposed a quantative method of measuring temper-embrittlement which involves the determination of the differences in reciprocals of the transition temperatures. His data show that this difference is almost invarient with respect to the test methods that were investigated which included a standard V-notch Charpy specimen, a subsize V-notch specimen, a slow bend test, and a keyhole-notch thin specimen.

The Effect of Alloying Elements on Temper-Embrittlement of Steel

Various changes in the composition of a susceptible steel may either affect the amount of embrittlement that occurs or the kinetics of the embrittlement. Most of the investigations concerning the effect of alloy composition on temper-embrittlement have neglected the kinetic features of the problem, being more concerned with the amount of embrittlement that results from a particular heat treatment. In studying the effects of chemical composition on temper-embrittlement, complications result from the fact that the addition of an alloying element may produce changes either by itself or by its effect on the other alloying elements present. An alloying element may have some secondary effect on temper-embrittlement by changing grain size for instance. Furthermore, the addition of an alloying element may bring about a reduction in toughness which may not be related to temper-embrittlement at all. Therefore, interpretation of data on the effects of various alloying elements is not an easy task. Other factors are present which further complicate the picture. Such things as melting and deoxidation practices and existence of minor impurities are so interrelated that two steels made under the same conditions still vary in the degree to which they can be embrittled.

The one common element to all low alloy steels is carbon and its role in temper-embrittlement has been the source of much controversy in this field. Opinions as to the role of carbon have varied from a positive contribution to a completely negative contribution. Jaffe and Buffum[88] and Libsch and Bhat[107] have suggested that plain carbon steels are very susceptible to temper-embrittlement; so much so, that they become embrittled during the quench from the tempering temperature. However, Woodfine[146] has pointed out that other factors were present which accounted for the observed changes in impact strengths which were attributed to carbon and its effect on temper-embrittlement. From the available evidence it appears that plain carbon steels with manganese contents of less than 0.5 percent are not susceptible to temper-embrittlement.[146]

As yet, no systematic investigation has been done to determine the effect of varying the carbon content of a susceptible steel; although, Jolivet and Vidal[93] have shown that when the carbon content of a chromium steel was reduced from 0.22 to 0.073 percent, the temper-embrittlement was reduced but not removed. Jaffe[87] has reported that a vacuum melted nickel-chromium steel with 0.016 percent carbon was susceptible to temper-embrittlement. Buffum, Jaffe, and Clancy[21] claimed to have found no temper-embrittlement in an alloy containing 1.5 percent nickel, 0.6 percent chromium, and 0.003 percent carbon. Preece and Carter[125] also found no embrittlement in a series of synthetic chromium, phosphorus, iron and manganese, phosphorus, iron alloys where carbon was at a level of 0.003 percent.

On the other hand, work reported by Hum, Hultgren, McChesney, et. al.,[72,73,74,75,76,77,114] on a series of synthetic SAE 3300 steels

prepared by melting under helium, and free of carbon, temper-embrittlement, as measured by the transition temperature criterion, was found to exist under slow cooling conditions.

A large group of susceptible steels contain the alloying elements of nickel, chromium, and manganese. Any two or all three of these elements seem to contribute significantly to temper-embrittlement.[67,134] Plain nickel steels are not normally considered to be susceptible, but little reliable information really exists.[57] No impact energy changes or grain boundary attack comparable to that obtained in temper-embrittled steels was noted by McLean and Northcott[116] on a nickel steel containing 0.01 percent chromium after a typical embrittling treatment. However, Jones[95] did find a difference in impact properties of a nickel steel after isothermal treatments at 850°F up to 3,000 hours. These steels contained from 0.15 to 0.24 percent chromium and really were not pure nickel steels. Although it is doubtful that pure nickel steels are susceptible, the addition of nickel to a susceptible steel definitely increases the embrittlement for any given treatment.[134]

It is the general opinion that plain chromium steels are susceptible to temper-brittleness and that the temper-embrittlement increases with increasing chromium.[93] Results by several investigators on commercial steels verify this opinion.[15,26,56,113,119,134]

Similarly, plain manganese steels are also susceptible and the degree of embrittlement increases with increasing manganese content.[73,134] Furthermore, additions of manganese to steel increase the susceptibility to temper-embrittlement,[62,65,74,113,123] but the amount of manganese necessary has not been definitely established. Some workers[15,57,62,65] claim that with less than 1 percent manganese little

effect is noted; whereas, one[134] claims that more than 0.70 percent manganese is necessary to cause temper-embrittlement.

The effect of increasing the chromium content of a steel containing manganese, nickel, and chromium appears to be independent of the manganese and nickel.[146] However, the effect produced by manganese or nickel is increased by the presence of chromium.[134] Hollomon[67] has shown that if two steels have the same hardenability with respect to nickel, chromium, and manganese, they will have about the same susceptibility to temper-embrittlement.

Several elements are known to prevent embrittlement or at least decrease the tendency for a susceptible steel to become embrittled. These elements include molybdenum, tungsten, and niobium. Although there is some evidence[136] that a plain molybdenum steel is susceptible, Jolivet and Vidal[93] have shown that the addition of 0.26 percent molybdenum to a susceptible chromium steel prevented temper-embrittlement. Other evidence exists that the addition of molybdenum to chromium, nickel-chromium, and nickel-manganese steels considerably reduces temper-embrittlement resulting from a given heat treatment.[5,7,8,57,134] Tungsten has been reported as being beneficial[70,143] in retarding temper-embrittlement although some evidence to the contrary has been reported.[136] Some workers claim that niobium is as good as molybdenum in suppressing temper-embrittlement.[15,96] Maurer, et al,[113] have reported that 0.25 percent of niobium addition to a steel is even better than molybdenum for certain temper-embrittling treatments.

Vanadium is known to increase the susceptibility to temper-embrittlement;[93] although again, reports have been made in which substantial improvement was realized by vanadium additions to susceptible steels.[26,56,57,62,65,119]

Among the typical deoxidizers used in steel making, aluminum, titanium, zirconium, and boron, only boron has been reported to have a noticeable effect on increasing temper-embrittlement.[66] An addition of 0.5 percent aluminum to a susceptible chromium steel had no effect on the susceptibility according to Jolivet and Vidal.[93] Likewise, titanium and zirconium were found to have little or no effect on temper-embrittlement.[65]

Although these deoxidizers do not have a direct effect on temper-embrittlement, they may act indirectly to increase the susceptibility by their action of nitride formation. Nitrogen is known to contribute to the embrittlement of steels occurring around 750°F or below. This phenomenon is not temper-embrittlement, but it may have been confused as such in many of the early works in the field, especially in the experiments based on susceptibility ratios where experiments were conducted on furnace cooled specimens. This nitrogen embrittlement is thought to be caused by a critical dispersion of nitrides. As little as 0.008 percent nitrogen is sufficient to cause severe embrittlement.[22] The nitrogen effect can be completely eliminated by proper deoxidation procedures.

As to what effect nitrogen has on temper-embrittlement little is known. Griffiths[61] found that embrittlement had occurred when nitrogen was added to a plain carbon steel, but the basis for comparison was a susceptibility ratio, which as noted above, can lead to questionable results.

A general class of elements that find their way into a steel during the steel making operation includes the elements phosphorus, sulfur, oxygen, copper, antimony, silicon, arsenic, and tin. These may

be grouped into two categories, those that have been found to contribute to embrittlement and those that have not. In the first category are phosphorus, antimony, arsenic, and tin. Many contributors have noted substantial increases in susceptibility caused by phosphorus. Baeyertz, et al.,[7] showed that the temper-embrittlement of chromium steels increased when phosphorus was raised from 0.012 to 0.036 percent. Similar results were found by Herres and Elsea[65] in manganese and manganese-chromium steels.

Jolivet and Vidal[93] reported a substantial shift in transition temperature caused by antimony additions to susceptible steels. Investigations as to the effect of tin indicate transition temperature shifts as much as 200°F.[4,16] Only one study has been found which indicates a deleterious effect on impact properties caused by traces of arsenic in a susceptible steel.[75]

Among the elements in the second group that have little or no effect, sulfur[75,119] and oxygen[76] have been shown to decrease the overall impact properties. Copper[76,119] and silicon,[62] when present as impurities, have no effect on temper-embrittlement or overall impact properties.

A summary of the available data on the effects of chemical composition on temper-embrittlement is presented below:

1) Carbon apparently contributes to temper-embrittlement, but in what manner has not been established.

2) Combinations of alloying elements such as manganese, nickel, and chromium increase the susceptibility.

3) Plain nickel steels are not susceptible, but upon adding nickel to a susceptible steel, temper-embrittlement is increased.

4) Plain chromium steels and plain manganese steels are susceptible. Additions of chromium or manganese to a susceptible steel embrittles the material still further.

5) Molybdenum, tungsten, and niobium on the whole decrease the tendency for a steel to develop temper-embrittlement.

6) Boron and vanadium increase the susceptibility to temper-embrittlement.

7) Nitrogen is known to contribute to embrittlement below 750°F but not to temper-embrittlement.

8) Deoxidizers such as aluminum, titanium, and zirconium have no effect on temper-embrittlement.

9) Minor traces of phosphorus, arsenic, antimony, and tin apparently contribute significantly in bringing about temper-embrittlement.

10) Minute quantities of sulfur, oxygen, copper, and silicon have no effect on temper-embrittlement.

It appears likely that no one element is solely responsible for temper-embrittlement. A large group of elements, all of them very common in steels, may act as a group in various combinations to cause the phenomenon. These elements include: carbon, chromium, nickel, manganese, phosphorus, and nitrogen.

Theories on Temper-Embrittlement

A large number of theories have been formulated as to the mechanism of temper-embrittlement. Many of them are very similar in concept while some of them have been contrived to explain just one facet of a very complicated phenomenon. Others have been shown to be in error by new data which are continually being compiled on the subject. Any theory which is to be of value must explain, at least, the following observations:

1) Loss in impact properties while other mechanical properties are virtually unaffected.

2) Grain boundary attack found with several special etches.

3) Intergranular brittle failure of an embrittled impact bar along prior austenite grain boundaries.

4) Why so many alloying elements affect the susceptibility of steels to temper-embrittlement and in what manner they do so.

5) The reversibility of the embrittling reaction as a function of time as well as temperature.

6) Why the shift in transition temperature progressively increases to a maximum value with time.

As yet no theory has been advanced which has been substantiated by experimental data on all of these points.

Two hypotheses, which suggested an allotropic modification in the steel below 1300°F, proposed by LeChatelier[106] and Jefferies[92] must be rejected since no evidence of specific heat or changes in enthalpy have been observed.

Several people[38,133] have proposed the novel suggestion that as a result of carbon segregation to the grain boundaries, austenite was retained there and subsequently decomposed to form films of martensite enveloping the grains. This proposal is not logical since it cannot explain how temper-embrittlement can be produced in susceptible steels by slow cooling from the embrittling temperature range after tempering sufficiently to decompose any retained austenite; nor is it consistent with the fact that temper-embrittlement is known to be reversible, i.e., it appears, disappears, and reappears as a function of time at a given temperature.

A large number of hypotheses, which can be grouped under a common name of precipitation theories, have been proposed to explain temper-embrittlement. In these theories it is postulated that the precipitation of a compound takes place from solution in the ferrite

primarily at the ferrite and prior austenite grain boundaries. The suggested compounds include cementite or another carbide, nitrides, phosphides, and various oxides of either iron or some alloying element. The suggestion that the precipitate is a carbide seems to be the most popular. Rogers,[129,130] Grenet,[59] Andrew and Dickie,[3] Honda and Yameda,[69] Nagasawa,[119] Jaffe and Buffum,[90] and Kishkin[96] all are of this school of thought. A nitride precipitate has been suggested by Griffiths,[61] Hollomon,[67] Imai and Ishizaki.[78] Phosphides are considered to be the major precipitate by Feszenko and Czopiwski[41] and Bennek.[11] The difficulty with the precipitation theories is that, as yet, no precipitate has been detected in temper-embrittled steels, since micrographs showing the effects of special etches like ethereal picric and zephiran chloride reagents on temper-embrittled specimens do not reveal a boundary phase, but merely a grain boundary attack. This discrepancy has been circumvented in the suggestion that embrittlement might arise from a segregation of various elements, particularly carbon, to the prior austenite grain boundaries[22,116] on a submicroscopic scale.

Another group of similar hypotheses fall under the classification of carbide modification theories. Advocates of this type of theory are Maurer and Hohage,[112] Bischof,[14] Maurer, Wilms, and Kiessler.[113] These theories are not supported by the fact that significant shifts in transition temperature have been observed in synthetic steels, melted under helium, and essentially free of carbon.[72,73,74,75,76,77,114]

Another theory proposed by Jaffe[86] suggests that small particles of another phase segregate to the ferrite grain boundaries where the interfacial energies are favorable. However, it has been shown that the brittle fracture of temper-embrittled specimens follows prior

austenite grain boundaries[22,147] and it is not clear how the ferrite boundary concept can be consistent with this observation. Maloof[109] considers the size and distribution of the carbides as being the principle factor in temper-embrittlement. He states that at some given time and temperature the carbides will be arranged to give maximum embrittlement. Further isothermal treatment at this temperature will cause a coagulation or overaged condition with some return of impact strength.

Greaves and Jones[56] have suggested a critical dispersion of chromium oxide as being responsible for temper-embrittlement. However, the presence of embrittlement in the absence of chromium, and in the absence of oxygen, refutes this theory.

Several other theories have been proposed which are of little value on the basis of consistency with observed facts.[33,119,124,133]

Thus, it appears that none of the proposed theories have been able to fully explain all of the criteria listed in the beginning of this section. No one constituent is likely to be the sole cause of temper-embrittlement. The more elements present in a steel, the greater seems to be the possibility for temper-embrittlement to occur.

Fatigue Phenomenon

Fatigue phenomenon has developed into one of the most complex and intriguing problems in the metallurgical sphere of endeavor. The crux of the fatigue problem can perhaps be best presented by asking the question: Why does a metal fail under repeated applications of a load which it could support indefinitely under static loading conditions? According to Gough[52] fatigue has much in common with plastic flow and fracture of metals under static conditions of stress. Under repeated

stresses the metal deforms by slip on the same planes and directions that are active in static slip. The slip system is given by the same resolved shear stress law in either case. No macro changes in the specimen can be observed during fatigue testing since alternate half cycles reverse the deformation. However, on a microscopic scale slip occurs with reverse deformation taking place on planes parallel to those that have initially slipped. Cracks are seen to develop in regions of heavy slip and tend to propagate along these active slip planes to eventual failure. These observations have lead to the classification of fatigue failure into three stages of development: the primary, intermediate, and final stages of fatigue.[63]

The primary stage is most noticeable in the softer metals where the maximum applied stress is above the initial yield point of the material. It may be absent when the stress is below this maximum value. In this period bulk plastic deformation occurs until the metal is work hardened to the level of the applied stress. Tensile tests show that the yield point has been raised to about the maximum applied alternating stress.[145]

The intermediate stage is believed to form the major part of the life of the specimen; although, there is some evidence to the contrary which will be discussed presently. It extends from the widespread settling down of the initial period, which is absent in high strength materials, to the formation of a visible crack. A gradual thickening of a number of slip bands formed in the primary stage occurs.[17,45,145] Eventually, a crack develops in these heavily deformed regions and the propagation of the crack constitutes the final stage of fatigue.

Rates of propagation[12,13,30] of the initial crack show that the length increases exponentially until the cross section is sufficiently reduced to lead to simple tensile failure. At high stresses the number of cracks formed are considerably greater than at low stresses, and at high stresses fracture will be more rapid because of the more rapid growth of individual cracks and the coalescence of several cracks to form larger ones. At lower stresses usually only one crack is observed and its rate of growth is slower. When the crack is first observed it is already large atomicwise. It is more pertinent to inquire how and when these cracks begin rather than when they are observed. This question has led to a number of interesting observations which tend to support the idea of the presence of a crack in a metal at a very early stage of its fatigue life. Ferguson[40] has observed cracks in a material given a 5 percent static extension after only 10 percent of the number of cycles required to fracture a similar virgin specimen. Sinclair and Dolan,[132] on the assumption that the formation of a fatigue crack was preceded by local work hardening, periodically interrupted a fatigue test and recrystallized the specimen to see if its life could be extended by removing the work hardening. Ten such recrystallizations, equally spaced during the life of the test, resulted in no increase in fatigue life. The authors concluded from this that the submicroscopic fatigue crack had already been formed in the initial 10 percent of the life of the specimen. Further evidence of early crack formation has been presented by Fenner, Owen, and Phillips.[39] These experiments indicate that fatigue cracks may actually form early in the life of the specimen and propagate rather slowly.

It appears, then, that substantial improvement could be made in explaining the fatigue phenomenon if more were known of the intermediate stage, because in this stage deformation occurs in limited regions and it is there that the crack is ultimately seen. Usually the crack is assumed to be formed as a result of severe distortion in these regions. However, the observations cited earlier, that cracks are sometimes detected very early in the life of a specimen, suggest that the deformation in localized regions is the result of the stress concentrations around a submicroscopic crack.

Microscopic observations of slip bands during the intermediate stage of fatigue suggest that individual slip lines of the band are formed at irregular time intervals. Now, if fatigue deformation is an intermittent process, then it is clear that an element of randomness in fatigue is present, and this is supplemented by the large scatter found in experimental S-N curves. This element of randomness in fatigue data cannot be explained on the basis of random thermal fluctuations occasionally aiding the applied stress to initiate slip since there exists substantial experimental evidence to show a small temperature dependence of fatigue.[63,144] There is, therefore, a statistical element involved in fatigue which needs considerable elaboration.

A number of theories on fatigue have been suggested in an attempt to answer the disarming question posed at the beginning of this discussion. The remainder of this section will be devoted to presenting these attempts.

Theories on the Fatigue of Metals

A number of theories have been proposed to explain fatigue in terms of static elastic properties of metals.[23] These have not met

with success because the theory of elasticity presupposes matter to be homogeneous and isotropic, conforming to Hooke's Law. A metal does not conform to any of these suppositions, nor is the distribution of stress imposed on a test specimen uniform which the elasticity theory further assumes.

Theories have been formulated which attempt to consider these three principles of elasticity theory, i.e., 1) homogeneity of material, 2) uniformity of stress distribution, and 3) soundness of Hooke's Law. The first two principles are involved in theories which can be conveniently classified in Griffith's[60] words as "theories of secondary stresses."

Orowan[122] considered a minute localized inhomogeniety in the form of a small plastic region embedded in an elastic matrix. Under an applied stress the plastic region yields, passing on part of the stress to the neighboring elastic medium. This process work hardens the plastic region and, with repeated loadings, the extent of work hardening per cycle diminishes in a geometric progression so that it tends to a finite limit which depends on the applied stress. If this limit is below the fracture stress, then the metal can withstand an infinite number of cycles. If the stress is higher, the fracture strength will be reached in a finite number of cycles and rupture occurs.

The third principle, the soundness of Hooke's Law, forms the basis of a number of fatigue theories which have to do with the elastic hysteresis properties of a metal. It is well known that when a metal is elastically strained it will not return along the same stress-strain line after the load has been removed. The difference in the two stress-strain curves, one on loading and the other on unloading, is called hysteresis. LeChatelier[105] believed that in the case of alternating

stresses the addition of these hysteresis effects assumes significant importance and brings about the eventual deterioration of the metal. Bairstow[9] and Gough[53] have shown that an upper limit of alternating stress exists and the stress-strain curve will remain a straight line for an infinite number of cycles if the stress is below this value. It has been suggested by these authors that this limiting stress corresponds to the endurance limit of the material. Bauschinger[10] had this in mind when he called the endurance limit the "natural elastic limit."

In certain metals the endurance limit does correspond to the stress that will just produce hysteresis, and in certain cases the endurance limit has been determined based on these deformations and the resulting absorbed energy that accompanies hysteresis.[23,53] However, the principles are not generally applicable for it is often observed that heating occurs during a fatigue test without fracture necessarily occurring. If the hysteresis loop is closed, the emission of heat should not be detrimental since the initial and final conditions of each cycle are the same. When first starting a fatigue test, the hysteresis loop increases with successive cycles. Up to a certain value of stress the loop tends toward a stable form and the material is said to have reached the state of accommodation. Upon exceeding this stress the area of the hysteresis loop increases and fracture occurs. In these terms the mechanism of fatigue is regarded as a deleterious action leading to progressive cracking of the crystals being opposed by a strengthening action arising from the phenomenon of accommodation. The endurance limit represents a balance of these two actions. This concept forms the main argument in Dehlinger's[31,32] proposed mechanism of fatigue. Because of widely differing orientations of the grains and

inhomogeneities in a metal, there exists a variation in elastic and plastic properties from point to point. Under repeated loadings locked-up internal stresses develop due to the inhomogeneous deformation and, when they exceed the fracture strength of the metal, cause a crack to form.

Machlin[108] has approached the subject with a somewhat different concept. In his theory he presupposes the existence of microscopic cracks in the material. These cracks are presumed to propagate by the generation of dislocations in the stress field at the tip of the crack with the aid of thermal fluctuations. However, this theory appears to be too presumptuous because it predicts a large temperature dependence of fatigue which has not been observed.[135] Furthermore, it does not seem likely that cracks with sufficiently large stress concentrations are generally present in all metals.[46]

A number of theories have been developed on the basis of the presence of an element of randomness in fatigue. Weibull[137] developed a theory for Griffith solids in which he chose a random distribution of flaws in a material and assumed that the probability of fracture in any one volume is independent of the probability of fracture in any other element at an equal stress. The theory works well with brittle bodies but not with metals.

Fisher and Hollomon[43] have developed a theory similar to Weibull's but applied to metals. However, the assumption that the probabilities of fracture in given volumes at the same stress are independent does not hold for metals as was pointed out by Zener.[149]

Another statistical theory of fatigue has been proposed by Freudenthal.[48] He assumes that fatigue is associated with the destruction

of atomic cohesive bonds and that plastic slip and strain-hardening are important only insofar as they modify the intensity and rate of bond destruction. By applying probability theory he deduced relationships for the establishment of the typical S-N curve. However, the probability of failure is not the same after the application of a number of cycles of stress as it is at the outset. Therefore, the theory cannot be applied to plastic materials.

Finally, Afanasiev[1] has proposed a statistical fatigue theory based on the concept of an average stress-strain relationship within individual grains of a polycrystalline metal. He assumes equal yield strengths in all grains in the direction of the acting load, but a non-uniform stress distribution due to crystal inhomogeneities and anisotropy. Further, assuming a particular frequency distribution of the stresses in the crystals and a linear relationship between strain-hardening and plastic deformation, the increase in flow stress as a function of the number of cycles is computed. Cracks occur when the flow stress reaches a value of the strength of the material. The probability of occurrence of the formation of a number of cracks in adjacent grains is calculated and gives expressions for the fatigue strength as a function of the number of stress cycles. The number of assumptions made in this theory cast dispersions of doubt as to the validity of this approach in its present form.

Thus, a purely statistical approach has not led to a satisfactory explanation for the phenomenon of fatigue of metals. Probably the basic mechanism of fatigue failure is connected with the laws of plastic deformation, work hardening, and fracture, but exactly how these are applied to fracture by fatigue is not clear at the present stage of development.

Overstressing and Understressing

A number of investigators have studied the fatigue phenomenon in terms of the effect of varying the amplitude of the stress cycles on the fatigue life. These studies have been called overstressing or understressing experiments on fatigue. The term overstressing usually applies to a testing condition in which the stress is above the endurance limit of the material for a limited number of cycles. Understressing is defined as stressing the fatigue specimen below the endurance limit a limited number of cycles. The effect of overstressing or understressing is then evaluated by testing the specimen at some different stress. If the fatigue life is shortened by the prestress cycles, the material is said to be damaged. If the fatigue life is increased, then the effect of prestress is beneficial to the fatigue properties.

A number of different methods for evaluating fatigue damage have been used in the past. Kommers[98] used a method whereby he determined a new endurance limit as compared to the virgin endurance limit. The method proposed by French[47] and used by Wishart and Lyon[144] in their work was similar except that damage by overstress was evaluated by running the overstressed specimens at the stress of the original endurance limit. If the specimen failed, then damage had been done. If the specimen ran indefinitely, then no damage had been done.

A very severe difficulty in the above methods of evaluating the effects of overstressing is apparent if it is remembered that the fatigue phenomenon is subject to statistical variations. An accurate establishment of the endurance limit of a material is extremely difficult. Methods have been developed lately which can successfully cope with the statistical nature of the endurance limit;[34,42,126] however,

these methods require either a large number of test data or, in the case of the Prot method, a special type of loading mechanism. For this reason much of the early work on fatigue damage by overstressing is questionable since the endurance limit was not established in a statistical manner.

A method for evaluating damage by overstressing used by Muller-Stock[118] circumvents the difficulty of establishing the endurance limit accurately. In this method a specimen was first run at an overstress for a given number of cycles. The percentage of damage caused by this overstress was then obtained by running the specimen at a still higher stress and comparing the decrease in the number of cycles for failure at this higher stress with the virgin life of the material at that stress.

In general, the effect of overstressing on the fatigue life is to weaken the material. However, slight degrees of overstress and a short number of cycles may actually improve the endurance life.[98]

Understressing experiments, on the other hand, have shown substantial increases in fatigue strength.[12,98] In general, understressing is most effective in strengthening the material at stresses just below the endurance limit. Apparently, such stresses induce a favorable residual stress pattern which results in better fatigue properties. Bennett[12] and many other investigators have shown that if several million cycles of a stress just below the endurance limit are applied to a fatigue specimen, and then the stress is raised slightly and again applied for a few million cycles, this process can be repeated before the specimen fails. Some materials can be strengthened to as much as

50 percent above their original value. This method of improving fatigue properties has been termed coaxing.

Two explanations for the coaxing effect have been advanced in recent years. It is believed that, in general, any process which inhibits the continuation of slip in a metal will increase its fatigue strength. Therefore, in attempting to explain the coaxing phenomenon it is assumed that the process of understressing must in some manner increase the elastic limit of slip planes where fatigue damage begins. One explanation involves the beneficial effects of work hardening of the metal during the short periods of understressing.[100] However, this explanation is not consistent with the fact that cracks form in regions of maximum slip where work hardening is the greatest. Reports[35,97,98,99,100] of substantial fatigue strength improvements by coaxing have been made only on ferrous materials subject to strain aging. These and some of Sinclair's[131] own work have led him to propose a strain aging mechanism for the second explanation of the coaxing phenomenon in fatigue.

In determining the amount of damage inflicted by some prestress, early workers[103,117] have assumed a linear relationship between the amount of damage done and the cycle ratio at the testing stress. This assumes that the cumulative cycle ratio at failure for a given specimen is 100 percent. Experiments have shown that such is not the case.[12,35,98,111,128] These show that the damage-cycle ratio curves vary with stress, but that the order in which the stresses are applied is not critical.[120]

Mathematical Treatment of Fatigue Data

The wide scatter of results of fatigue tests is well recognized today. However, many investigations are still carried out on the

assumption that the fatigue life of a material is a well controlled physical property and can be defined with reasonable accuracy by a small number of tests performed at many stress levels. This lackadaisical attitude towards the statistical nature of fatigue is brought out in an ASTM Manual on Fatigue Testing in which a preferred procedure for determining an S-N curve is described.[110] In it is recommended a procedure in which at least ten specimens be run, selecting stresses so as to yield a uniform distribution of test points over the stress range. S-N curves established in this manner may be at best an estimate of the mean fatigue life, but they give no information as to the expected range of values that exists about this mean.

In an attempt to establish in which type of universe fatigue tests fall, Muller-Stock[118] conducted 200 fatigue tests on steel at one stress level. The frequency distribution of the cycles to failure, N, was found to be skewed toward the high cycles to failure end. However, when the frequency distribution was plotted with respect to log N, the distribution approached a normal, or Gaussian distribution. This type of distribution has been used very extensively in the field of fatigue as a basis for statistical analysis of test data.

Some criticisms have been made against the use of a normal distribution to describe fatigue data. These have been made on the basis that if the normal distribution represents the true statistical nature of fatigue, then there exists a small, but finite probability of failure occurring at very low or very high cycles of stress with respect to the mean life in the finite life region of the S-N curve. Before the logarithmic normal distribution for fatigue life can be entirely accepted, the occurrence of these failures must be observed

experimentally. Weibull[139] has proposed an alternative distribution which circumvents the objection raised against the logarithmic normal distribution. His exponential distribution function provides finite limits to the distribution of the number of cycles to failure at a constant stress. Freudenthal and Gumbel[50] have proposed an extreme value distribution which also predicts a finite life for a fatigue specimen in the upper regions of the S-N curve. However, there is little practical difference between these three distributions. Over the central range of values, extending from about a 5 percent probability of failure to 95 percent probability of failure, the three methods yield almost identical results.[49] In fact they are so close that in order to distinguish which one is more effective would require the testing of about 10,000 specimens at one stress level.[138] The logarithmic normal distribution has been used extensively because standard statistical methods can be applied to the interpretation of tests of small numbers of specimens with log N as the variable.

A theoretical justification for the use of the logarithmic normal distribution in describing fatigue data, which follows the statistical reasoning of Cramer,[29] has been given by Freudenthal.[49] Making a few simplifying assumptions he has shown that the function P(N) will be logarithmic-normal as a first approximation.

The decision as to how many tests should be performed to establish an accurate S-N curve is a difficult one to make. Usually an economic compromise must be made between the uncertainty that one wishes to tolerate and the number of available specimens. The number of tests that should be run at any one stress in order to give a reasonable estimate of the mean life has been estimated by Freudenthal[49] to be at least

six, preferably eight or ten. In order to estimate the range of values of fatigue life about a mean value at a given stress level, at least 12 specimens should be tested. In planning a fatigue program it is preferable to run a number of specimens at two or three stresses rather than only a few specimens at many stresses.

Thus far consideration has been given to the finite region of the S-N curve. When the establishment of the endurance limit is desired, a more complicated problem is encountered for in this case the situation is one of go or no go. Either the specimen survives or it fails. Three methods have been used recently to establish the endurance limit in a statistical manner. Ransom and Mehl[127] used a method referred to as staircase testing. This procedure consisted of running a series of tests, each run at a slightly lower or higher stress than the preceding test, depending upon whether the preceding test failed or ran out, respectively. In their work they found a 2σ band of $\pm$ 6,000 psi ranging about the endurance limit of 46,000 psi.

A different technique was employed by Epremian and Mehl[37] in their work in a statistical study of the endurance limit of several steels. The approach was similar to the statistical methods used in the biological fields in determining various lethal dosages of drugs. In their procedure a mortality curve of percent failure versus stress level was established using the technique of probit analysis.[42] The results showed that the scatter in the endurance limit was more sensitive to metallurgical variables than was the scatter in fatigue life.

A third method for establishing the endurance limit has been proposed by Prot[126] and is now being used quite extensively in recent fatigue experiments. This method requires the testing of fatigue

specimens at various rates of increasing stress. A plot is then made of stress versus the square root of the rate of stress increase which results in a straight line. The intersection of this straight line with the stress axis is the endurance limit for the material. The results obtained by the Prot method correlate well with endurance limits established by procedures using constant stress amplitudes and the method has the advantage of requiring testing on a less extensive scale than either the stepwise or probit methods.[27] However, the method is only applicable to materials with a well defined endurance limit and cannot be applied to materials where no endurance limit seems to exist. Furthermore, materials that are subject to coaxing exhibit higher endurance limits by the Prot method than by the conventional methods.

EXPERIMENTAL PROCEDURES

The material used in this investigation was an SAE 3140 steel obtained in 0.75 inch round bars from a single heat having the following composition:

C	Mn	P	S	Si	Cr	Ni	Mo	Grain Size
0.39	0.78	0.010	0.019	0.28	0.61	1.26	0.04	No. 8

Prior to the heat treatment of this material for impact and fatigue specimens, the bars were rough machined oversize, then after heat treatment the specimens were finish machined as required. For the impact bars, specimens were rough machined to 0.44 x 0.44 x 2.19 inches. Fatigue bars were initially machined to 0.56 x 3.56 inches. Bars that were used for tensile specimens were not machined prior to heat treatment.

Heat Treatment

All of the heat treatments prior to tempering were carried out in a Hoskins electric furnace. Tempering to the tough condition was performed in a salt pot and embrittling treatments were accomplished in a vertical pot type resistance wound furnace. The bars, as received, were first normalized one and one-half hours at 1650°F, then austenitized one hour at 1600°F and quenched into an agitated oil bath. The bars were cleaned in an organic solvent to remove the residual oil and then tempered one hour at 1250°F in salt. Six specimens, evenly spaced in a stainless steel basket, were heat treated as a unit. To each basket was attached a chromel-alumel thermocouple which was connected to a

chart recorder. All the times given for any heat treatment are times at temperature as was indicated by the recorder.

Embrittling treatments on impact specimens were carried out at 850, 875, 900, 925, and 950°F for times of 100 and 500 hours. Fatigue bars were embrittled only at 850°F for 500 hours.

Some samples that were used for extraction carbide studies were given, after an initial 500 hour embrittling treatment, a reheat of one hour at 1250°F. This was done to produce a recovery in the structure from the effects of temper-embrittlement.

Tensile bars were heat treated in a similar manner in the same facilities. Embrittling treatments for these specimens consisted of 100, 250, and 500 hours at 850°F. Specimens, following all tempering and embrittling treatments, were water quenched.

Samples for autoradiographic studies were prepared by cutting thin slugs of 0.06 inches thickness from the as received bar stock. These slugs were then decarburized in wet hydrogen. Best results were found using a 30 percent by volume water mixture at 1550°F. In 100 hours these slugs were completely decarburized as far as was determinable microscopically. After decarburization each slug was sectioned into three nearly equal pieces and carburized in a capsule containing a mixture of charcoal and barium carbonate. Some of the barium carbonate contained radioactive carbon-14. A loaded capsule is illustrated in Figure 1. After loading the capsule was sealed by welding and heated for 72 hours at 1700°F, followed by a water quench. Following carburization the capsules were carefully opened by first drilling into the cavity to allow any gas build-up to escape. None was noted in any of the capsules, however. The specimens. three to a capsule, were removed

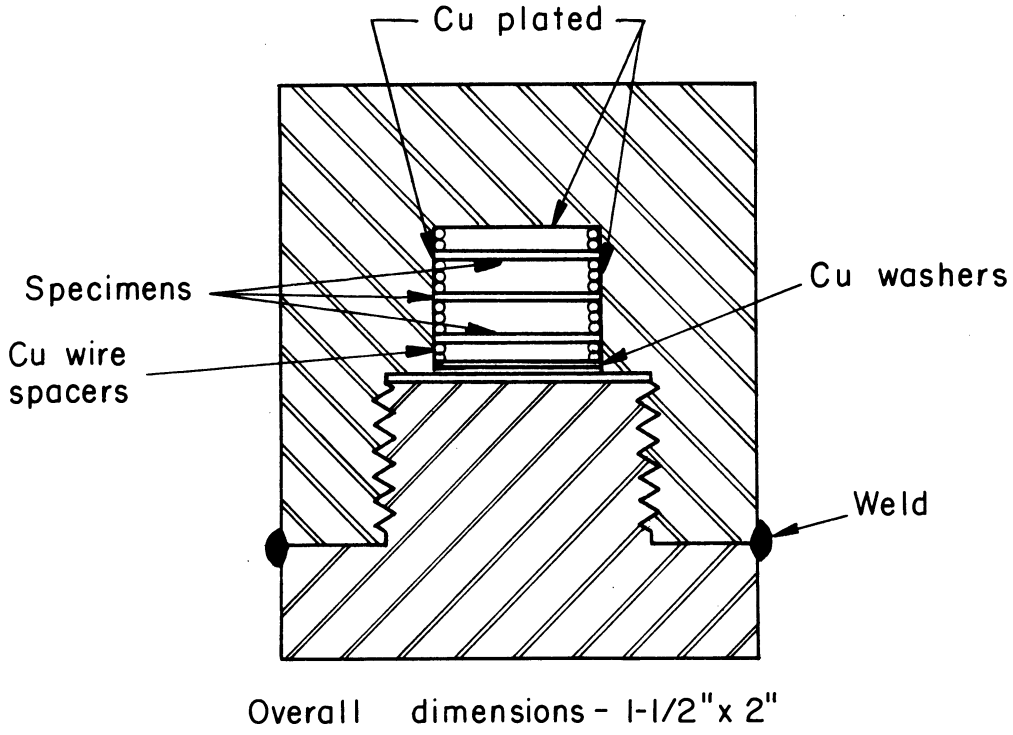


Figure 1. Cross-Section of Steel Carburizing Capsule in Loaded Condition.

under a hood and cleaned in alcohol. Then they were examined metallographically for homogeneity and carburization completeness.

After carburizing the radioactive samples were sealed in vycor tubing under vacuum and run through the normal heat treating cycles to produce the tough and temper-embrittled conditions. These heat treatments were similar to the heat treatments for the fatigue and impact specimens except that instead of austenitizing at 1600°F followed by an oil quench, the radioactive samples were austenitized at 2000°F for two hours and brine quenched. The higher austenitizing temperature for the radioactive samples was employed to increase the grain size of the material to facilitate an easier autoradiographic examination.

Extraction and Analysis of Carbides

Extractions on samples in the tough, 500 hour temper-embrittled, and temper-embrittled plus one hour at 1250°F conditions were obtained electrolytically in a 5 percent hydrochloric acid solution using a platinum cathode and a potential of 2 volts. A sufficient amount of extracts were obtained in two and one-half hours. These extracts were examined by X-ray diffraction and spectroscopic techniques.

X-ray analysis on the extracts was done using unfiltered cobalt radiation in a Debye-Scherrer camera. In addition, back reflection photographs of the tough, embrittled, and reheated one hour at 1250°F structures were also obtained on solid samples using unfiltered cobalt radiation. Spectroscopic analysis on the extracted powders was performed on a Cornu spectrograph using graphite cups as receptacles for the powder specimens. The intensities of the 2926.59 Å iron line and the 2855.68 Å chromium line were used to compare the relative amounts of iron and chromium in the extracts.

Testing Procedures

It was first necessary to establish a suitable heat treatment for temper-embrittlement which could be used in subsequent experiments involving fatigue testing and carbon-14. This was done using the transition temperature criterion of temper-embrittlement established using V-notched Charpy impact specimens heat treated to various degrees of embrittlement.

Impact Tests

A standard V-notch Charpy specimen was used for all impact tests. These specimens had a final dimension of 0.394 x 0.394 x 2.165 inches in length. All specimens were machined by the same machinist and the same cutter was used to machine the V-notch.

A Sonntag Universal Charpy Impact Machine of 240 foot-pound capacity was used for all tests. Precautions were taken to insure proper anvil knife edges and contact of the tup with the specimen surface opposite the notch. In each test the anvil swung cleanly through the specimen without jamming the specimen to the sides of the knife edge supports.

Specimens were tested over a range of temperatures from room to -314°F. Impact bar temperatures between -314°F and -116°F were obtained by suspending specimens above liquid nitrogen contained in a Dewar flask. Intermediate temperatures between -116°F and 32°F were achieved in an insulated cold box containing dry ice. Samples were immersed in acetone until an equilibrium condition was obtained. Temperatures above the melting point of ice were attained by suspending samples above an ice-water solution in a Dewar flask. In all cases the specimens were soaked at least thirty minutes at the specific temperature in question.

Thermometers were used to measure temperatures in all cases. For the range of temperatures from -314°F to -116°F a pentane thermometer was used, while from -116°F to 32°F a toluene thermometer was used. Above 32°F temperatures were measured using a mercury thermometer. The pentane and toluene thermometers were checked at the fixed points of liquid nitrogen and dry ice, respectively. The mercury thermometer was checked at the freezing and boiling points of water. The thermometers checked within one degree at each check point which was believed sufficient for this type of testing.

After the allowed time had expired, thirty minutes, the specimen was removed from the cold box with stainless steel tongs, which were also cooled to the temperature of the specimen, and placed in the Charpy unit and broken. The elapsed time in removing the specimen to fracture was less than five seconds. Baeyertz, et al.,[8] have shown that this short interval of time has no effect on the resulting impact properties.

Specimens were placed in acetone after fracture and allowed to reach room temperature. Then, Rockwell C hardness values were determined on the two halves of the specimen.

Transition temperatures were determined using a 50 foot-pound criterion. At this level the resulting energy-temperature curves seemed to fall off rather sharply from a high to a low impact energy value.

Fatigue Tests

Fatigue specimens were finish machined after the completion of all heat treatments on the fatigue bars. The radius of the fatigue specimens was made by successive cuts of about 0.030 inches until 0.020 inches remained on the reduced section of the bar. Then, a final cut

of 0.015 inches was made leaving 0.005 inches to be removed with emery papers of 120 and 300 grades. The final 0.001 inch was removed by polishing with a 3/0 metallographic paper while the specimen turned in the lathe, moving the paper parallel to the axis of the specimen. The type of fatigue specimen used in this investigation is shown in Figure 2.

All of the fatigue testing in this program was done on a single R. R. Moore type rotating beam machine. This machine was of a variable speed type covering the range from 500 to about 12,000 rpm. A single speed of 7,000 rpm was used for all of the tests. Tests were conducted in a cold room maintained at 0°F.

The specimens were measured to the nearest 0.0005 inch using a Browne and Sharpe micrometer with rounded tips. Care was taken in measuring the diameters so as to prevent injury to the surface of the specimen. All of the weights used in these tests were calibrated and the maximum probable error in the stress placed on any given specimen was ± 250 psi.

For the determination of the S-N curve for the tough and brittle samples, the specimens were merely loaded and run to failure. In determining the damage inflicted by a prestress, the specimens were loaded to the required prestress, run to a predetermined number of cycles, and then without stopping the test a sufficient amount of weight was removed from the pan to lower the stress to the run out stress of 75,000 psi. The S-N curves were determined by running either three or four specimens of each type, tough or brittle, at three stress levels, 90,000, 85,000, and 75,000 psi. Damage determinations were run at three prestress levels of 90,000, 85,000, and 80,000 psi for various

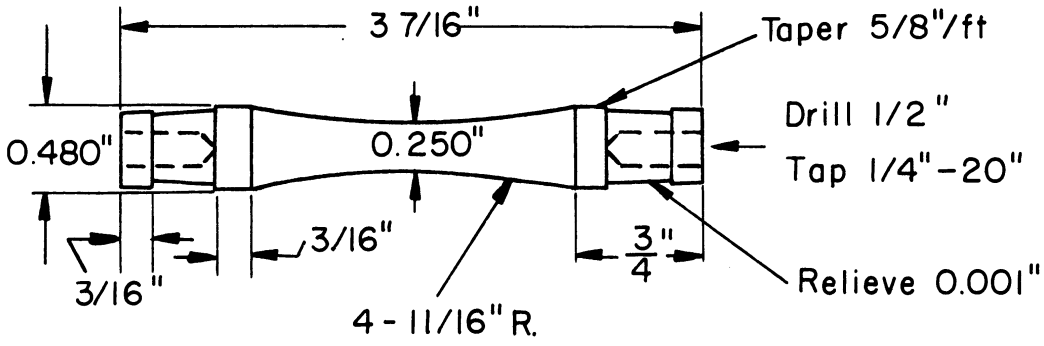


Figure 2. Fatigue Test Specimen.

numbers of cycles. For the most part, four specimens at any given set of prestress conditions were run.

In all of these tests no heating of the specimen was detected either by feel or by a thermocouple placed as close to the reduced section of the specimen as possible during the course of the test. However, just before fracture occurred the specimen did increase in temperature. This heating was due to actual abrasion of fractured surfaces in the reduced section and in no way alters the fact that the specimens throughout a major portion of their lives, at least 99 percent, were run at 0°F.

After fatigue testing the specimens were immersed in acetone and allowed to reach room temperature. Rockwell C hardness readings were then taken on both halves on the curved surface, at each end, between the bearing surfaces of the specimen.

Tensile Tests

The mechanical properties of the tough and temper-embrittled conditions were established on a Baldwin and Southwark tensile testing machine using a variable transformer type strain gage on standard 0.505 inch tensile specimens. Three specimens of each condition were tested. From these tests it was noted that a substantial difference in the drop in the beam existed for the tough and brittle specimens. To investigate this more thoroughly a Tinius-Olsen tensile machine was used with a Tinius-Olsen type S3 strain gage. Specimens were tested in the tough and 100, 250, and 500 hours at 850°F temper-embrittled conditions at a strain rate of 0.003 inches per minute. In these tests the specimens were pulled through the yield point to 0.4 percent elongation. The load was then released and the specimens artificially aged 24 hours at

400°F in an oil bath, after which the yield point was again determined and compared to the initial yield point properties.

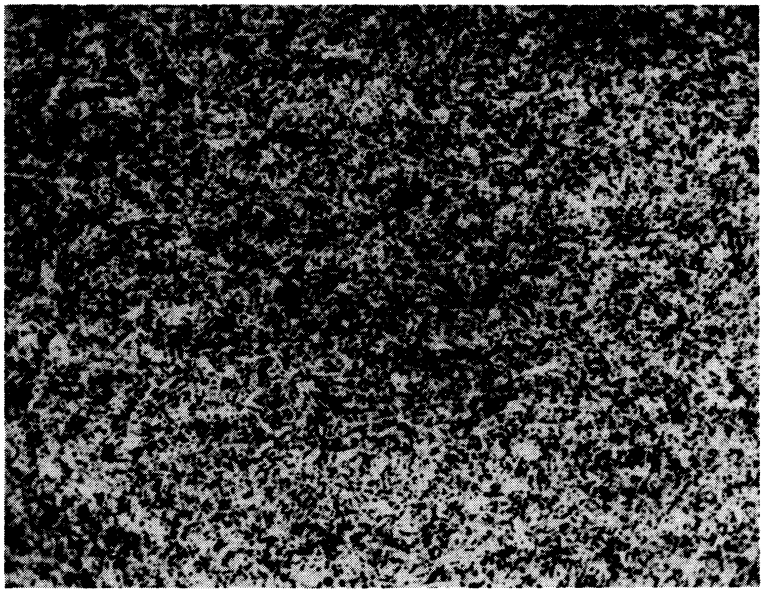
Two tensile bars were pulled through the yield point which were put through the reversible heat treating cycle, i.e., one hour at 1250°F plus 500 hours at 850°F plus one hour at 1250°F. These bars were run in a Baldwin and Southwark tensile machine using an O. S. Peters microformer strain follower.

Metallography

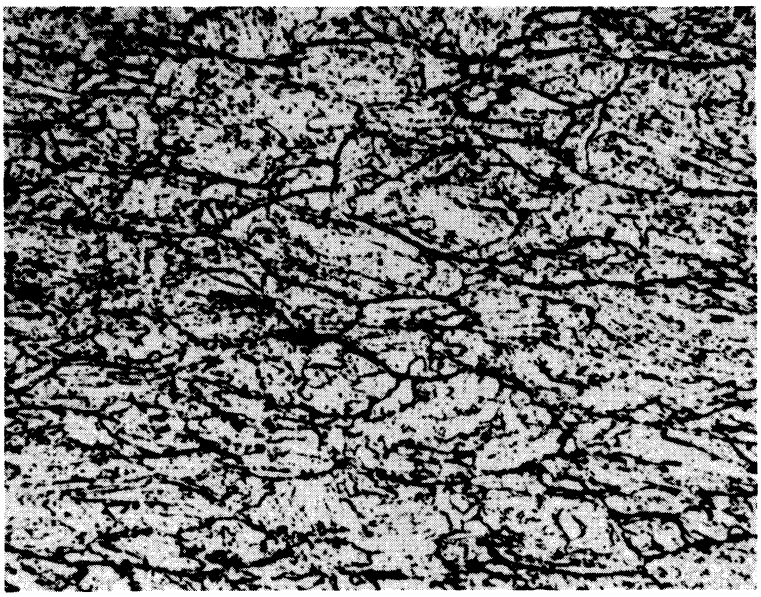
A metallographic examination was made on all of the structures produced by the various heat treatments used in this investigation. In the case of the experiments to determine a condition of severe temper-embrittlement an examination was made on impact specimens that had been tested at room temperature.

The preparation of samples for metallographic examination consisted of an initial polish through the 240, 400, and 600 girt silicon-carbide wet papers, followed by 0-2 micron diamond wheel, and then an electrolytic polish with a Buehler-Waisman electro-polisher. The electrolyte consisted of a 20 percent perchloric acid (d = 1.21) solution in ethanol (95 percent). Optimum results were obtained using 20 volts and a current density of 5 amp/in.^{2} for 20 seconds.

Electrolytic polishing was used initially rather than mechanical polishing because it has been reported by Jacquet[82,83] that the cold work left on the surface of a mechanically polished specimen interferes with the selective etching of temper-embrittled materials. However, in subsequent work with radioactive samples where electrolytic polishing was not feasible, no trouble was encountered in this respect by the use of mechanical polishing methods. In Figure 3 is presented the



a) 1 Hour, 1250°F, W.Q., Tough Condition



b) Tough Condition + 500 Hours, 850°F, W.Q.
Temper-Embrittled Condition

Figure 3. Microstructures of Tough and Severely Temper-Embrittled Conditions Taken in the Fractured Region of Broken Tensile Bars. Electrolytic Polish. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x

microstructures of tough and temper-embrittled fractured tensile bars. The areas observed are the "necked-down" regions of the fractured tensile specimens. Even in the severely distorted structure of the embrittled bar, the ethereal picric etchant selectively attacked the prior austenite grain boundaries.

The etchant used to reveal the temper-embrittled structures was the one proposed by Jacquet[82] and consisted of a saturated solution of picric acid in ether. The etchant was prepared by dissolving 50 grams of picric acid in 250 milliliters of water to which was added 250 milliliters of ether. This mixture was vigorously shaken and upon settling formed two distinct layers. The top layer containing picric acid in ether was decanted as needed for etching. Etching was done by immersion for times from 10 seconds to 2-1/2 minutes, depending upon whether the specimen was to examined with the electron microscope or optically. After etching the samples were rinsed in alcohol, water, and then alcohol again and dried in an air blast. Microscopic observations and micrographs were made up to a magnification of 2,000 diameters using a Bausch and Lomb Research Metallograph. Direct bright field illumination was used for all of the micrographs.

Electron Micrographs

Since optical microscopy is limited to magnifications of the order of 3,000 diameters because of poor resolution, it was necessary to resort to electron microscopy to examine the grain boundaries of these structures to a greater extent.

In preparing specimens for this work, extreme care was necessary in the polishing and etching procedures. The technique finally established differed somewhat from that employed in preparing specimens

for optical examination. Notably, the etching time was reduced from 2-1/2 minutes to 10 seconds. Also, after etching, the samples were cleaned in a jet stream of alcohol, followed by a jet stream of distilled water, followed by a jet stream of alcohol, and finally, rinsed in pure acetone. Between each wash the specimen was dried using a rubber syringe. In none of the operations was cotton employed to swab the specimen. Otherwise, the polishing and etching conditions were the same as was used for optical micrography.

After etching, replicas of collodion were prepared. A collodion film was stripped from the specimen with scotch tape and discarded prior to the actual replica preparation. At least two replicas on each sample were prepared for examination. Polystyrene-latex particles of known size were placed on the replicas prior to shadowing with palladium. Replicas were examined at magnifications up to 20,000 diameters.

Autoradiography

In conjunction with the light and electron microscopes an autoradiographic technique was employed in an attempt to further study the grain boundary structure of a temper-embrittled steel. Essentially, the method consisted of using a radioactive isotope as a tracer in the form of carbon-14 in the steel, heat treating the steel, and then locating the carbon-14 atoms by their resulting effect on a photographic emulsion placed in intimate contact with the specimen.

In this investigation the stripping film technique of high resolution autoradiography was used exclusively.[51,148] The stripping film used was obtained from Eastman Kodak, known as Experimental Permeable Base Autoradiographic Stripping Film. The film consisted of

an emulsion layer of approximately five microns thick supported by a pure gelatin layer of equal thickness. These layers were further supported on a cellulose acetate base. The film was applied to the specimen after the gelatin emulsion base was stripped from the cellulose backing with the emulsion side towards the specimen.

It was necessary to mechanically polish the specimens for the autoradiographic studies since electrolytic polishing did not produce a satisfactory surface for film stripping because of the size and shape of the specimens. The procedure consisted of polishing through the 600 silicon-carbide wet paper, followed by a 0-2 micron diamond polish. After several successive polishing and etching cycles with nital, the samples were then polished using Linde B. Several polishing and etching cycles were also performed with this compound. After polishing the specimens were etched in ethereal picric solution prior to the actual autoradiographic step. Upon comparing microstructures of samples prepared by electrolytic and mechanical polishing methods no difference was noted between the two. In every case one could unequivocally determine which specimen had been embrittled and which one had not. On this basis a standardized mechanical polishing technique was adopted for subsequent work on samples that were to be examined autoradiographically.

The procedure for preparing the etched specimen and the actual placement of the photographic emulsion on the radioactive specimen has been very adequately described by Yukawa[148] and the description of such need not be elaborated upon here. However, in the actual examination of the emulsion and microstructure after development, a somewhat modified procedure had to be adopted. In examining the microstructure of an autoradiographic sample one actually looks through the developed

photographic emulsion which is in intimate contact with the specimen. Since the microstructure of tempered martensite appears as a distribution of carbide particles, which appear as black spots in a white ferrite matrix, difficulty was encountered in distinguishing these carbide particles from the black, developed silver grains of the photographic emulsion. By focusing on the metal structure and then focusing on the emulsion this difficulty was circumvented but required two photomicrographs of the same area. By this means one could trace the distribution of the active carbon atoms in the material without confusing these activity centers with normal carbon carbides. At a magnification of 1000 diameters this method could be applied successfully because of the small depth of field characteristic of the lens.

Autoradiographic studies were performed on samples in the tough condition and the 100 hour and 500 hour temper-embrittled conditions. Various etching times, exposure times, and specific activity levels were used in an attempt to maximize the conditions for observing any segregation of the carbide particles that might take place.

Statistical Treatment of Data

Since the phenomenon of fatigue failure in metals is statistical in nature one must study the behavior in a statistical manner to obtain reliable results. However, this procedure may sometimes necessitate the accumulation of enormous amounts of data. Many times in determining the number of tests to be run in an experiment one has to strike a compromise between a low degree of certainty in the results on the one hand and the economic feasibility of running a larger number of tests on the other hand.

The underlying assumption that fatigue data obeys the Gaussian distribution when the cycles to failure are expressed as logarithms has been used by many investigators[37,49,118,127] as a basis for evaluating statistically the phenomenon of fatigue. This assumption has been made in the present investigation.

Standardized statistical methods have been employed in this investigation. All methods applied to these data are adequately described in the books on statistics by Duncan[36] and Fisher.[44]

Tests on homogeneity of variances were done using the F-test. Since these tests indicated a lack of homogeneity in variance, no analysis of variance as such was made, because an analysis of variance presupposes a uniform variance.

The means and standard deviations were computed for all sets of data and compared for real differences. Confidence intervals for the means of the S-N curves were also calculated.

RESULTS AND DISCUSSION

Impact Properties

A comparison of the transition curves obtained for the tough and temper-embrittled conditions is shown in Figures 4 and 5. It must be remembered that all of the embrittling cycles were preceded by the heat treatment given the tough samples, i.e., 1 hour at 1250°F, W. Q. In Figure 4 the tough condition is compared to the 100-hour embrittling treatments at various temperatures. Figure 5 presents the tough and 500-hour embrittling treatments at various temperatures.

The shifts in transition temperature, caused by various temper-embrittling heat treatments, are tabulated in Table I. These shifts in transition temperature have been established using a criterion of 50 foot-pounds impact energy.

It can be seen from the graphs and Table I that, among all of the heat treatments used, the 500 hours at 850°F treatment resulted in the greatest shift in transition temperature, amounting to about 181°F. Furthermore, this embrittling treatment yielded a large difference in impact energy absorption at 0°F, the temperature at which the fatigue program was carried out. In Figure 5 it can be seen that at 0°F the tough condition exhibited an impact energy value of 115 foot-pounds; whereas, for the case of the 500 hours at 850°F embrittling treatment, the resulting impact energy value was only 35 foot-pounds. Therefore, approximately four times as much toughness, as exhibited in the impact test, was present in specimens in the tough condition compared with the temper-embrittled condition at 0°F.

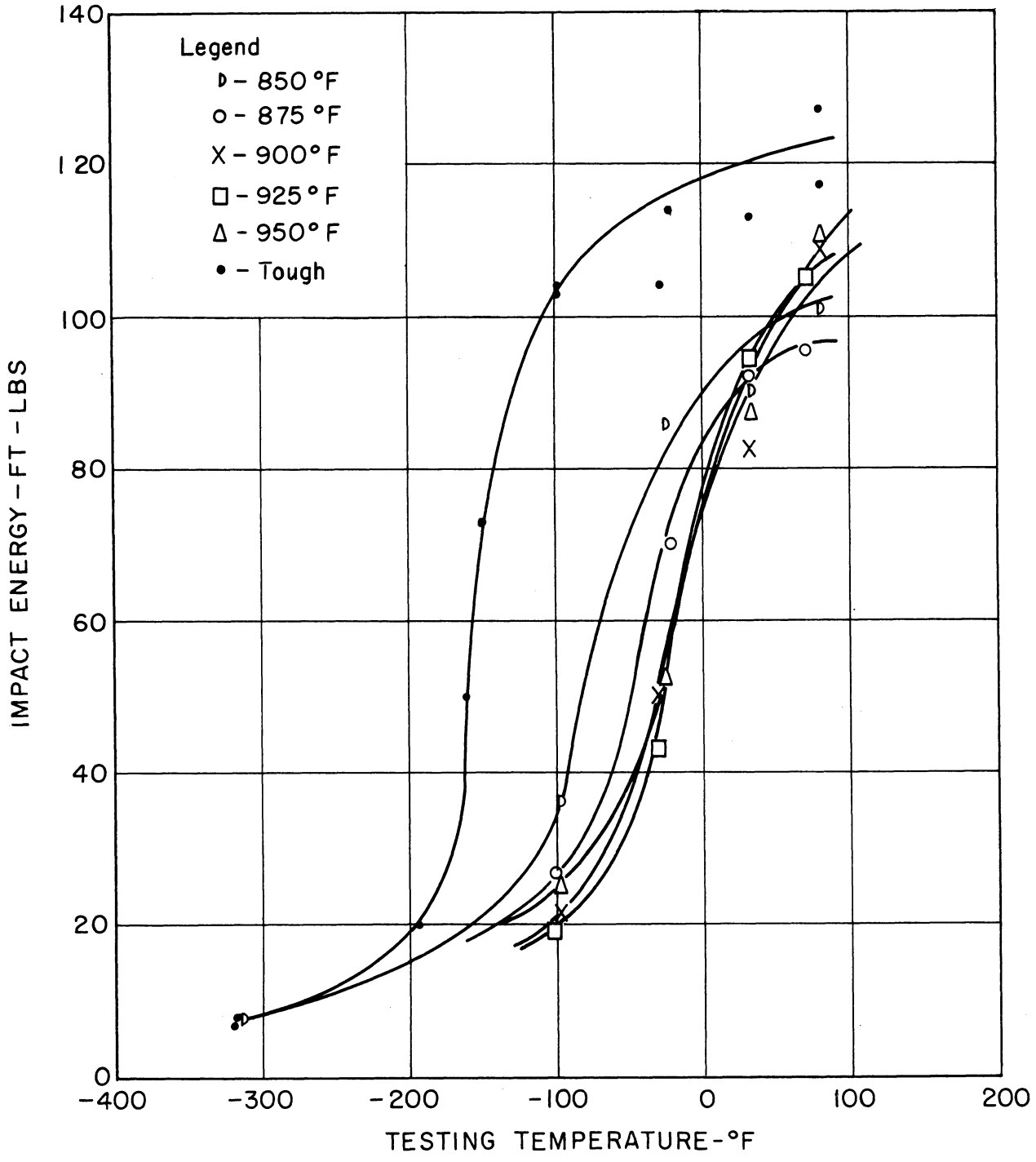


Figure 4. Comparison of Impact Energy - Testing Temperature Curves for Specimens Temper-Embrittled 100 Hours at Indicated Temperatures and Tempered 1 Hour at 1250°F, Tough Condition. SAE 3140 Steel.

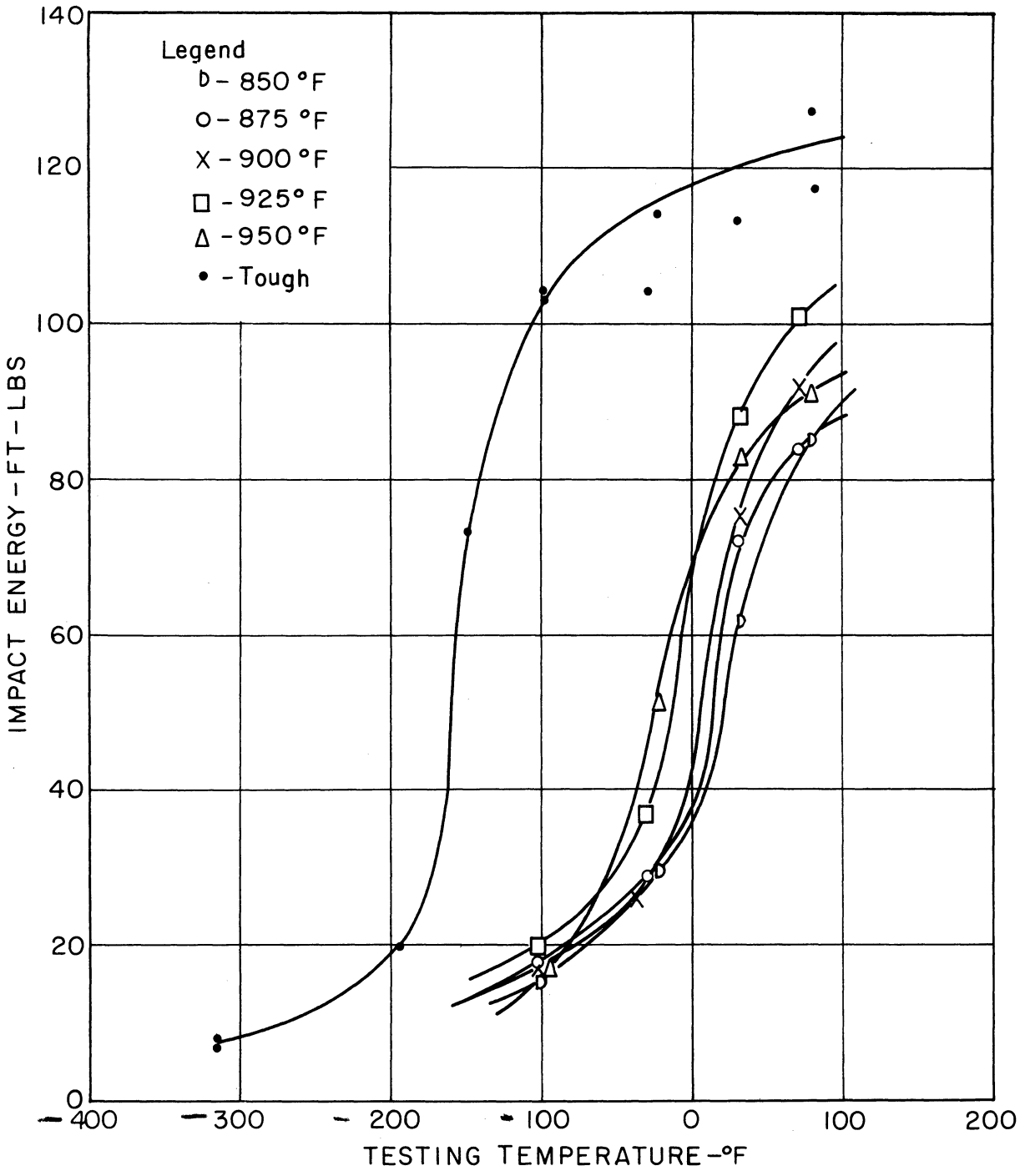


Figure 5. Comparison of Impact Energy - Testing Temperature Curves for Specimens Temper-Embrittled 500 Hours at Indicated Temperatures and Tempered 1 Hour at 1250°F, Tough Condition. SAE 3140 Steel.

TABLE I. TRANSITION TEMPERATURES FOR FIFTY FOOT-POUND ENERGY CRITERION ON SAE 3140 STEEL

Heat Treatment	°F	ΔT^{α}
Normalized 1-1/2 hour at 1650°F + Austenitized 1 hour at 1600°F, O. Q. + Tempered 1 hour at 1250°F, W. Q., Base, Tough Condition	-162	-
850°F - 100 hours, W.Q.[1]	- 81	81
850°F - 500 hours, W.Q.	19	181
875°F - 100 hours, W.Q.	- 49	113
875°F - 500 hours, W.Q.	14	176
900°F - 100 hours, W.Q.	- 31	131
900°F - 500 hours, W.Q.	5	167
925°F - 100 hours, W.Q.	- 27	135
925°F - 500 hours,,W.Q.	- 13	149
950°F - 100 hours, W.Q.	- 31	131
950°F - 500 hours, W.Q.	- 26	136

[1] All embrittling treatments were preceded by heat treatment given for the base, tough condition.

α Shift in transition temperature between the tough and temper-embrittled conditions using a 50 ft.lb. impact energy criterion

A compilation of the average Rockwell C hardnesses and ranges obtained on all of the impact bars tested is shown in Table II. It is apparent that no significant change in hardness resulted from the prolonged times at the various embrittling temperatures. Thus, the change in impact energy as a result of heat treatment is due only to temper-embrittlement and not the result of a lower hardness level.

Tensile Properties

Typical tensile properties for the tough and 500 hours at 850°F temper-embrittled conditions were determined on three tensile bars for each condition. These data are tabulated in Table III. No significant difference in tensile strength, proportional limit, or elongation was observed. A slight change in reduction in area was noted, however. The embrittled bars exhibited an average reduction in area of 37.9 percent; whereas, the tough bars showed an average reduction in area of 42.2 percent.

A difference in the appearance of the fracture was also observed for the two types of bars. This is shown in Figure 6. The bars labeled 1 through 3 are in the tough condition and exhibit a typical ductile cup-cone type of tensile fracture. The bars labeled 4 through 6 in Figure 3 are the temper-embrittled tensile bars. These bars had a more jagged appearing fracture and, in the case of the bars marked 4 and 5 in Figure 6, actually exhibited a crack running from 0.2 to 0.5 inches away from the fractured surface.

In determining the tensile properties for the tough and temper-embrittled conditions, it was observed that the drop in the beam at the yield point was almost twice as large for the embrittled bars as it was for the tough bars.

TABLE II. ROCKWELL C HARDNESS OF IMPACT SPECIMENS

Heat Treatment	Average Hardness	Range		No. of Specimens Tested[2]
		Maximum	Minimum	
Normalized 1-1/2 hour at 1650°F + Austenitized 1 hour at 1600°F, O.Q. + Tempered 1 hour at 1250°F, W.Q., Base, Tough Condition	22.4	23	22	11
850°F - 100 hours[1]	22.0	22	22	5
850°F - 500 hours	23.8	24	23	4
875°F - 100 hours	22.5	23	22	4
875°F - 500 hours	23.0	23	23	4
900°F - 100 hours	22.0	22	22	5
900°F - 500 hours	22.0	22	22	4
925°F - 100 hours	22.0	22	22	4
925°F - 500 hours	23.0	23	23	4
950°F - 100 hours	22.0	23	21	5
950°F - 500 hours	22.8	24	22	4

[1] All embrittling treatments were preceded by the heat treatment given for the base, tough condition.

[2] Eight readings were taken on each specimen.

TABLE III. TENSILE PROPERTIES OF TOUGH AND TEMPER-EMBRITTLED SAE 3140 STEEL

Condition	Tensile Strength psi	Proportional Limit, psi	Reduction in Area-%	Elongation in./in.- %
Tough[1]	116,900	102,900	41.7	25.0
Tough	118,200	104,600	42.8	23.5
Tough	119,000	105,700	42.0	24.0
Brittle[2]	114,500	106,200	38.2	24.5
Brittle	115,900	108,800	36.8	25.5
Brittle	116,300	109,300	38.9	24.0

[1] Normalized 1-1/2 hour at 1650°F + 1 hour at 1600°F, O.Q. + 1 hour at 1250°F, W.Q.

[2] Same as tough condition + 500 hours at 850°F, W.Q.

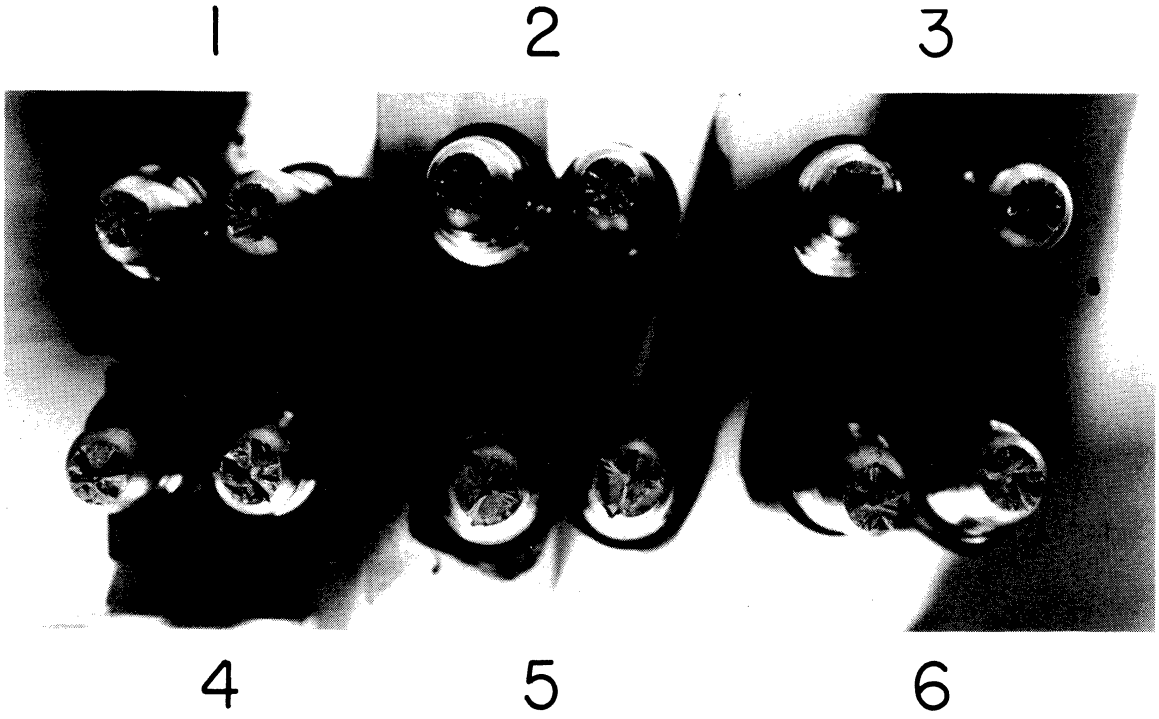


Figure 6. Appearance of Tensile Fractures for Tough and Temper-Embrittled Conditions. Bars 1-3 are in Tough Condition, 1 Hour at 1250°F, W.Q. Bars 4-6 are Temper-Embrittled 500 Hours at 850°F, W.Q. in Addition to the Tough Treatment.

This phenomenon was more carefully investigated with eight more tensile bars. These were initially strained 0.4 percent and then aged 24 hours at 400°F and again strained 0.4 percent. The results for the tough and various degrees of temper-embrittlement conditions are presented in Table IV. A clearer picture of the difference in yield point phenomenon for the tough and embrittled materials is shown in Figure 7. Prior to aging, a considerable difference in drop in the beam at the yield point results between the tough and temper-embrittled conditions. This difference amounts to 2,500 psi for a condition of embrittlement caused by 100 hours at 850°F. Further embrittlement does not seem to affect the yield point in any substantial manner. After aging the yield behavior for both the tough and the 100 hour and 250 hour embrittling treatments returns to about the original value obtained for the unaged tough condition. However, for the 500 hour embrittling treatment, a significant difference in the drop at the yield point occurs amounting to 2,500 psi below that observed for the tough, unaged condition.

From these data it appears that the presence of temper-embrittlement in this steel affects the yield point phenomenon in a subtle way. If this observation is correct, then the difference in yielding characteristics that is exhibited by the temper-embrittled structure should disappear when an embrittled structure is reheat treated to a tough condition. To test this premise, two tensile bars that were embrittled 100 hours at 850°F were reheated 1 hour at 1250°F. Although no impact specimens were run on this condition of heat treatment, it is known[22,147] that a heat treatment of this type will remove the effects of temper-embrittlement. Metallographic observation on this

TABLE IV. COMPARISON OF DROP IN THE BEAM AT THE YIELD POINT[1] FOR TOUGH AND TEMPER-EMBRITTLED SAE 3140 STEEL BEFORE AND AFTER AGING

Condition of Specimen	Upper Yield Point, psi	Lower Yield Point, psi	Δ Yield Point, psi	Modulus, psi x 10^6
Tough[α]	101,000	95,500	5,500	31.4
Tough*	101,000	95,000	6,000	31.3
Tough	102,500	96,875	5,625	31.0
Tough*	102,500	97,500	5,000	31.1
B-1	98,500	91,100	7,400	31.1
B-1*	97,000	94,500	2,500	30.8
B-1	104,100	95,700	8,400	30.6
B-1*	103,100	97,500	5,500	30.7
B-2	101,000	93,800	7,200	31.4
B-2*	101,100	95,700	5,400	31.4
B-2	105,000	97,500	7,500	31.3
B-2*	102,000	96,700	5,300	31.1
B-3	100,900	93,900	9,000	30.8
B-3*	98,700	96,200	2,500	31.1
B-3	98,100	96,200	5,900	31.2
B-3*	96,500	93,000	3,500	31.8

[1] Strain rate 0.003 in./min.

[α] Tough condition - Normalized 1-1/2 hours at 1650°F + 1 hour at 1600°F, O.Q. + 1 hour at 1250°F, W.Q.

* Same as preceding specimen but aged 24 hours at 400°F after an initial strain of 0.4% in./in.

B-1 - Tough condition + 100 hours at 850°F, W.Q.

B-2 - Tough condition + 250 hours at 850°F, W.Q.

B-3 - Tough condition + 500 hours at 850°F, W.Q.

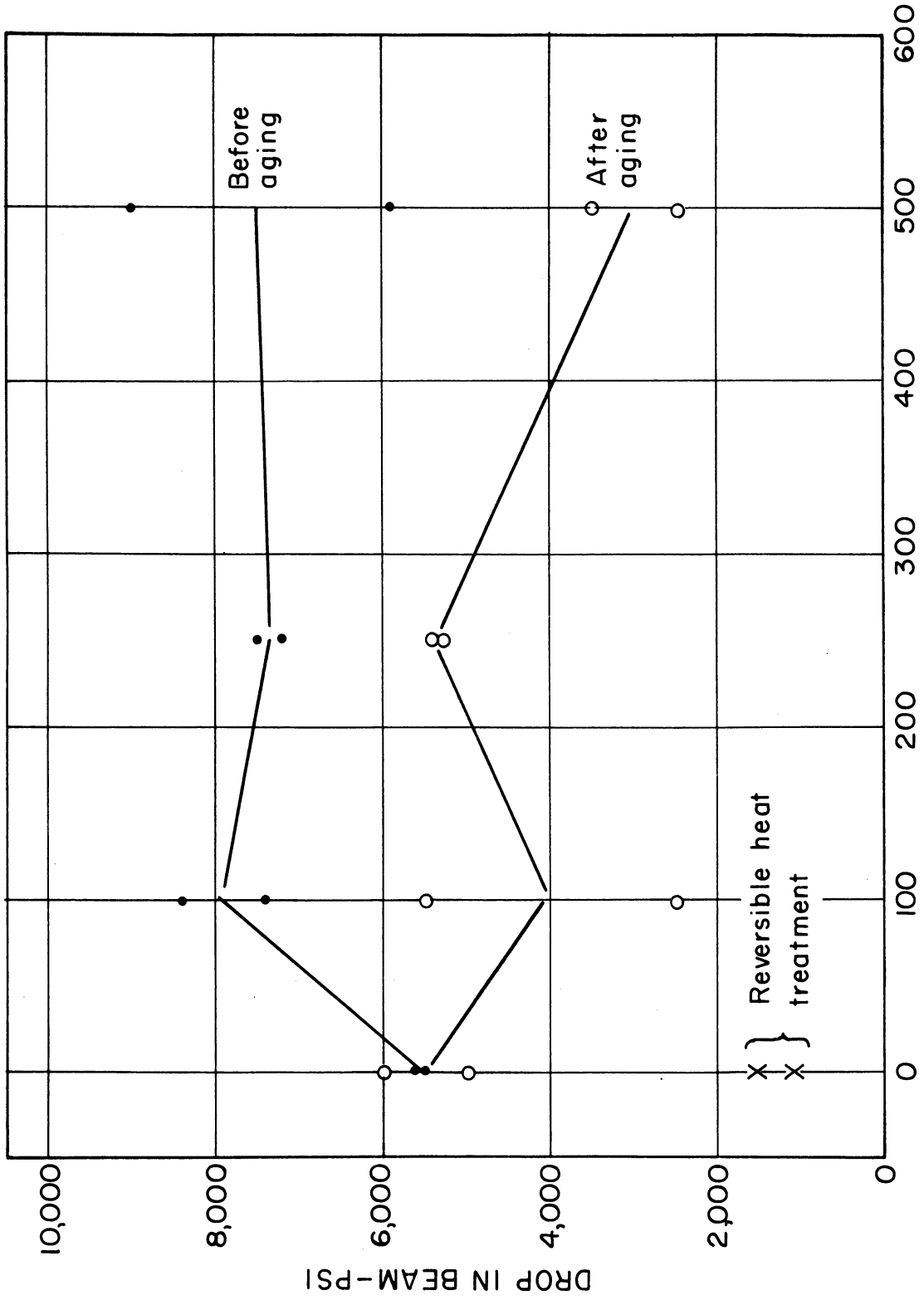


Figure 7. Comparison of Drop in the Beam in the Tensile Test Before and After Aging 24 Hours at 400°F as Affected by Time at an Embrittling Temperature of 850°F for an SAE 3140 Steel.

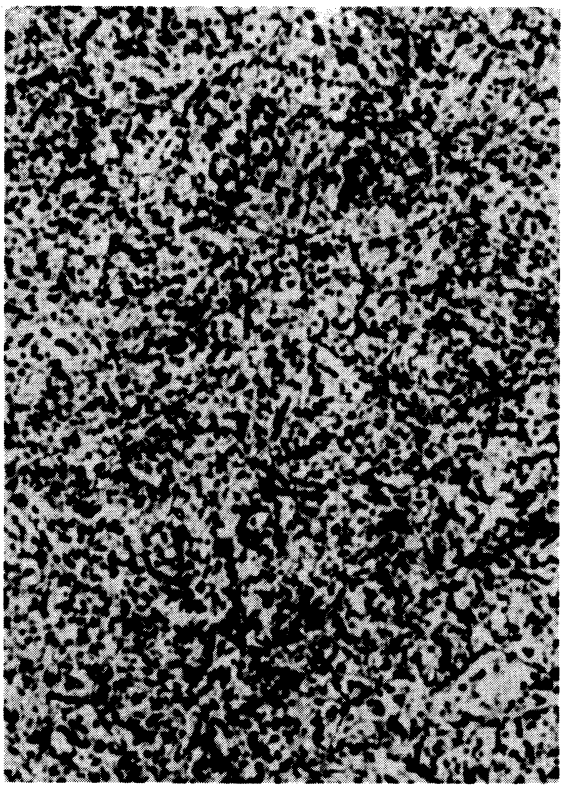
structure showed no prior austenite grain boundary attack such as is characteristic for a temper-embrittled structure etched with ethereal picric acid solution. Figure 8 presents a comparison of the structures obtained in a tough condition, 500 hours at 850°F temper-embrittled condition, and the reheated condition of 1 hour at 1250°F after the 500 hour embrittling heat treatment.

The drop at the yield point for the two reheat treated tensile bars amounted to 1,100 and 1,500 psi. These points are included in Figure 7. It appears from these data that the different yielding characteristics observed between tough and temper-embrittled structures are affected by the heat treatment which results in temper-embrittlement. These two phenomena may or may not be related.

Metallography

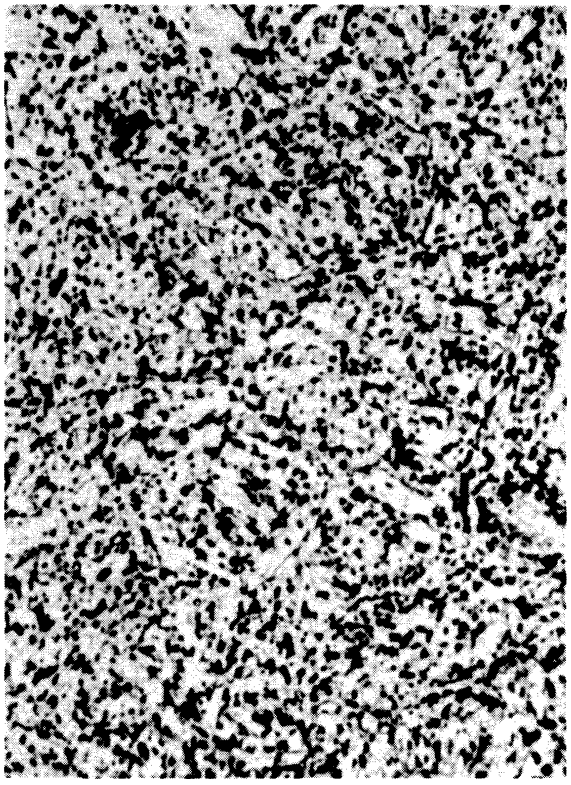
The specimens, whose structures appear in this section, were prepared by an electrolytic polish and etched by immersion for two and one-half minutes in an ethereal picric solution. Photomicrographs are presented at a magnification of 2000 diameters.

It has been shown by several investigators[22,25,79,147] that temper-embrittled steels are selectively etched at prior austenite grain boundaries; whereas, the same steel in the tough condition is not susceptible to this attack. The photomicrographs of Figures 9 and 10 verify this observation on this heat of steel. In Figure 9 is presented the microstructures resulting from the various heat treatments prior to any embrittling procedure. No evidence exists of a selective prior austenite grain boundary attack in any of these conditions. In Figure 10 the structures of three temper-embrittled conditions are presented. As the severity of temper-embrittlement increases by virtue



a) 1 Hour, 1250°F, W.Q.

b) "a" + 500 Hours, 850°F, W.Q.



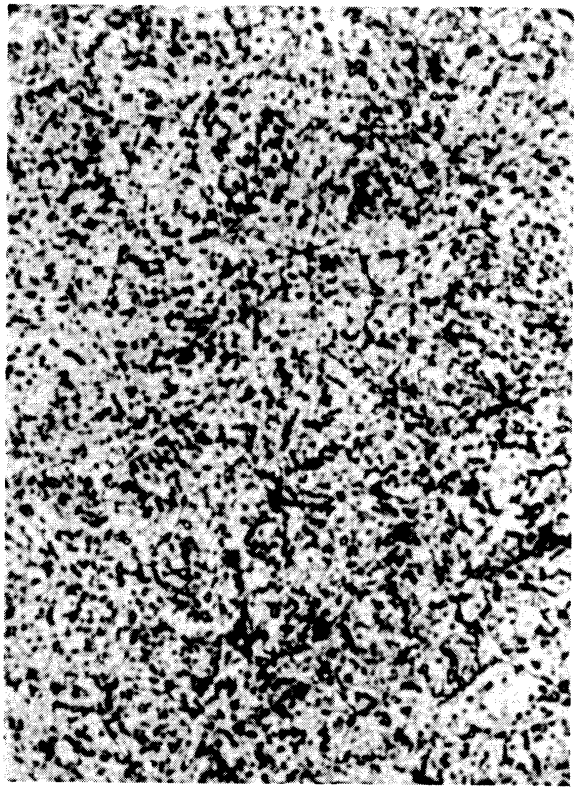
c) "a" + "b" + 1 Hour, 1250°F,W.Q.

Figure 8. Photomicrographs Showing the Reversal of Temper-Embrittlement by Heating to a Higher Temperature. Electrolytic Polish. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 2000 x

a) As Received Annealed

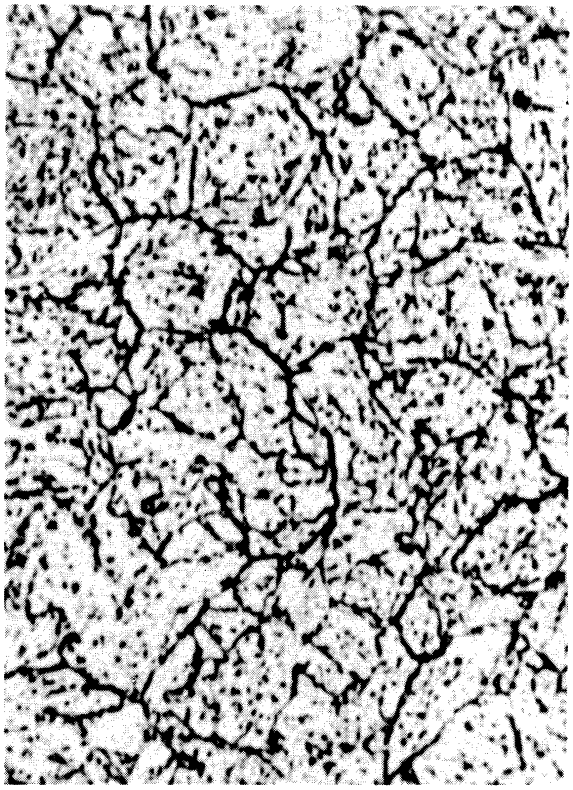
b) Normalized 1650°F - 1.5 Hours

c) As Quenched. Austenitize 1600°F
1 Hour, O.Q.

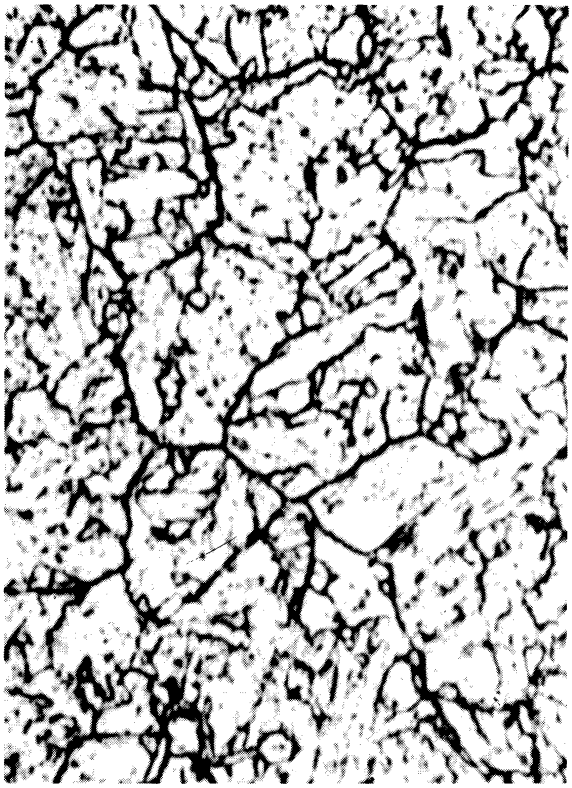


d) Tempered. 1250°F
1 Hour, W. Q.

Figure 9. Typical Microstructures Obtained in Heating Cycles Prior to Embrittlement. Electrolytic Polish. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 2000 x



a) 100 Hours



b) 250 Hours

c) 500 Hours

Figure 10. The Effect of Time at Embrittling Temperature of 850°F on the Microstructure. Normalized, Austenitized 1 Hour-1600°F, Tempered 1 Hour-1250°F, W.Q. Electrolytic Polish. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 2000 x

of heat treatments at 850°F from 100 hours to 500 hours, the etching attack becomes more continuous and much stronger. By simply comparing the degree of attack for the 100 hour and 500 hour embrittling treatments, it appears that the latter has been more severely embrittled. However, the extent to which embrittlement has occurred cannot be accurately established from microstructural examination alone.

From these photomicrographs it is not clear whether or not any precipitate is present in the grain boundaries of a temper-embrittled structure. The etching attack appears continuous and becomes broader as the severity of embrittlement increases. Further observations at higher magnification were necessary to determine the nature of this attack. These were done with the electron microscope and are described in the next section.

Electron Microscopy

Many investigators[11,67,90] have proposed that a precipitate of some kind is responsible for the phenomenon of temper-embrittlement. Since brittle fracture in temper-embrittled steels is known to take place along the prior austenite grain boundaries, the precipitation theories have revolved about the concept of a precipitation reaction occurring at these grain boundaries during heating in the susceptible temperature region.

To more carefully examine the grain boundary regions of a tough and temper-embrittled steel, electron microscopy was employed. The purpose of this phase of the investigation was to establish:

1) The presence or absence of a precipitate or a continuous film in the prior austenite grain boundary of an embrittled steel.

2) Whether or not any change in size or distribution in the carbides took place during embrittlement.

3) Whether the etching attack observed with the light microscope on embrittled structures was an actual attack or merely an etching stain.

Specimens that were examined with the electron microscope were electrolytically polished. Initially, etching was carried out from ten seconds to two and one-half minutes in ethereal picric solution. Considerable difficulty was encountered in stripping replicas from specimens etched longer than sixty seconds. Emphasis was placed on shorter etching times, not because of the ease of replica production, however, but because it was felt that if any phase was present at the grain boundary, the longer etching exposures would result in the complete removal of these constituents and make subsequent observation of them impossible. Observations on specimens etched for longer times were made but they showed only a more severe etching attack and actual grooves in the boundary areas. For these reasons only the electron micrographs obtained on specimens etched for ten seconds are included.

Figure 11 is a typical electron micrograph of a tough structure obtained by 1 hour at 1250°F after hardening. This and subsequent electron micrographs were taken originally at 10,000 diameters and then enlarged to 20,000 diameters. The black ball appearing on the micrograph is a polystyrene-latex particle of a given size such that it's diameter in millimeters times 4000 gives the magnification. The presence of these polystyrene-latex particles is also helpful in interpreting the micrographs properly. It must be remembered that these particles were placed on the stripped replica prior to shadowing with palladium. Therefore, these particles were actually above the surface of the replica, causing the white rim that is found near them on the electron micrographs.

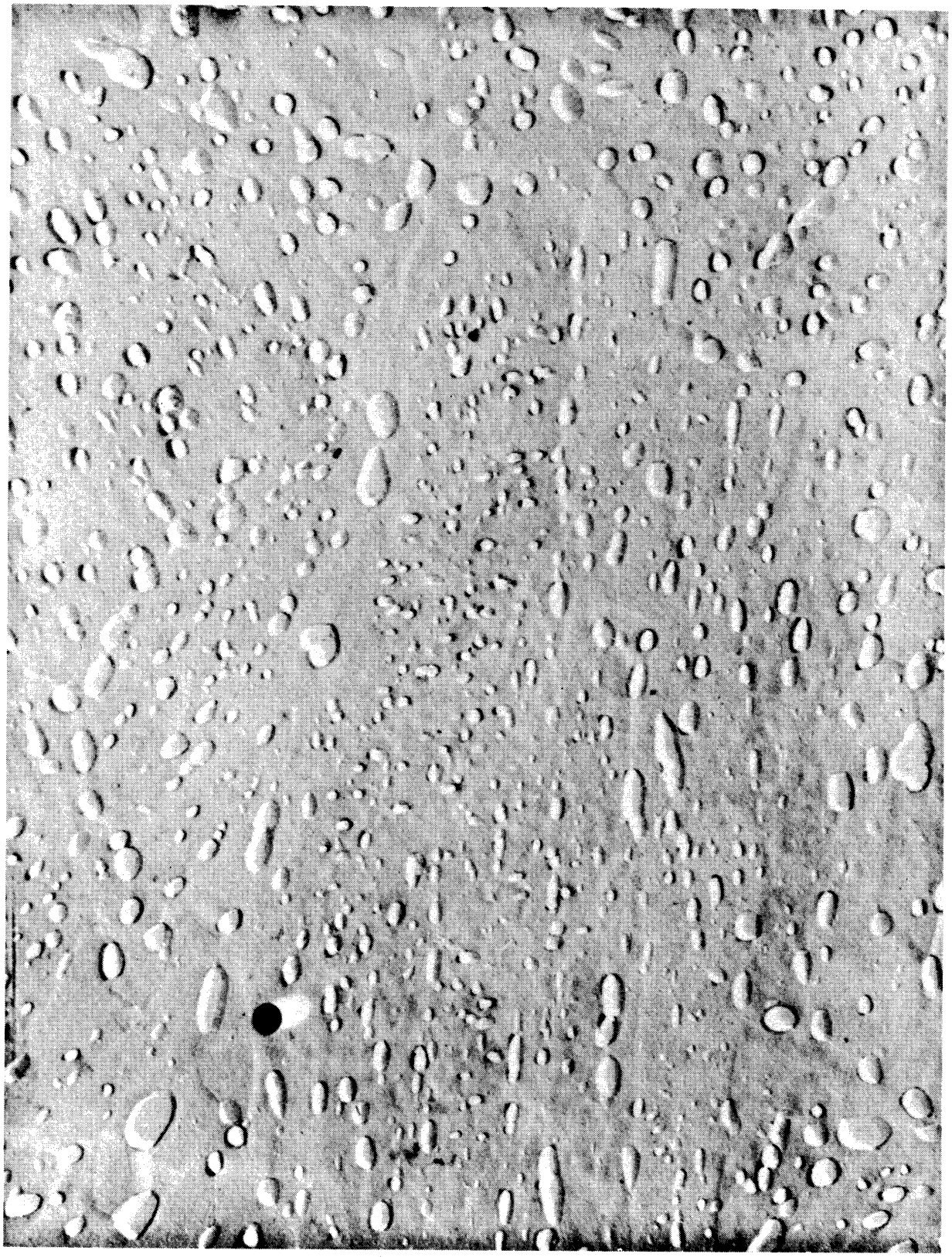


Figure 11. Electron Micrograph of Structure Produced by Tempering 1 Hour at 1250°F, W.Q., Tough Condition. Electrolytic Polish. Ethereal Picric Immersion Etch - 10 Seconds. Magnification 20,000 x

The side on which the white rim exists was the side that "saw" more palladium atoms during shadowing. Therefore, any particle having a white rim on a side opposite that of the polystyrene-latex particle is a depression in the replica, and consequently a particle in relief on the original metal surface. A white rim on the same side of a particle or impression as that which exists near the polystyrene-latex particle means that, on the replica, this particle or impression was in relief above the surface of the replica, and consequently below the surface of the original metal.

Even though with the light microscope no grain boundary attack is evident in a tough structure when etched with ethereal picric solution, a slight indication of such an attack is revealed in the electron micrograph of Figure 11. This attack seems to follow the outline made by the larger carbide particles found in the structure. Figures 12 through 16 are electron micrographs of temper-embrittled structures of increasing degrees of embrittlement. A slight increase in grain boundary attack over that observed in the tough condition is noted even by an embrittling treatment of 1 hour at 850°F, Figure 12. As the severity of embrittlement at 850°F increases, due to holding times of 1, 10, 50, 100, and 500 hours, the boundary attack becomes more severe. At 100 and 500 hours the outlines of larger grains, which are believed to be prior austenite grain boundaries, are very well delineated from the matrix. In most cases the prior austenite grain boundaries follow the larger carbide particles found in the structure. However, no change in size or distribution of these carbide particles can be discerned between the tough and temper-embrittled structures. Furthermore, no evidence of a continuous film around the prior austenite grain boundaries is evident

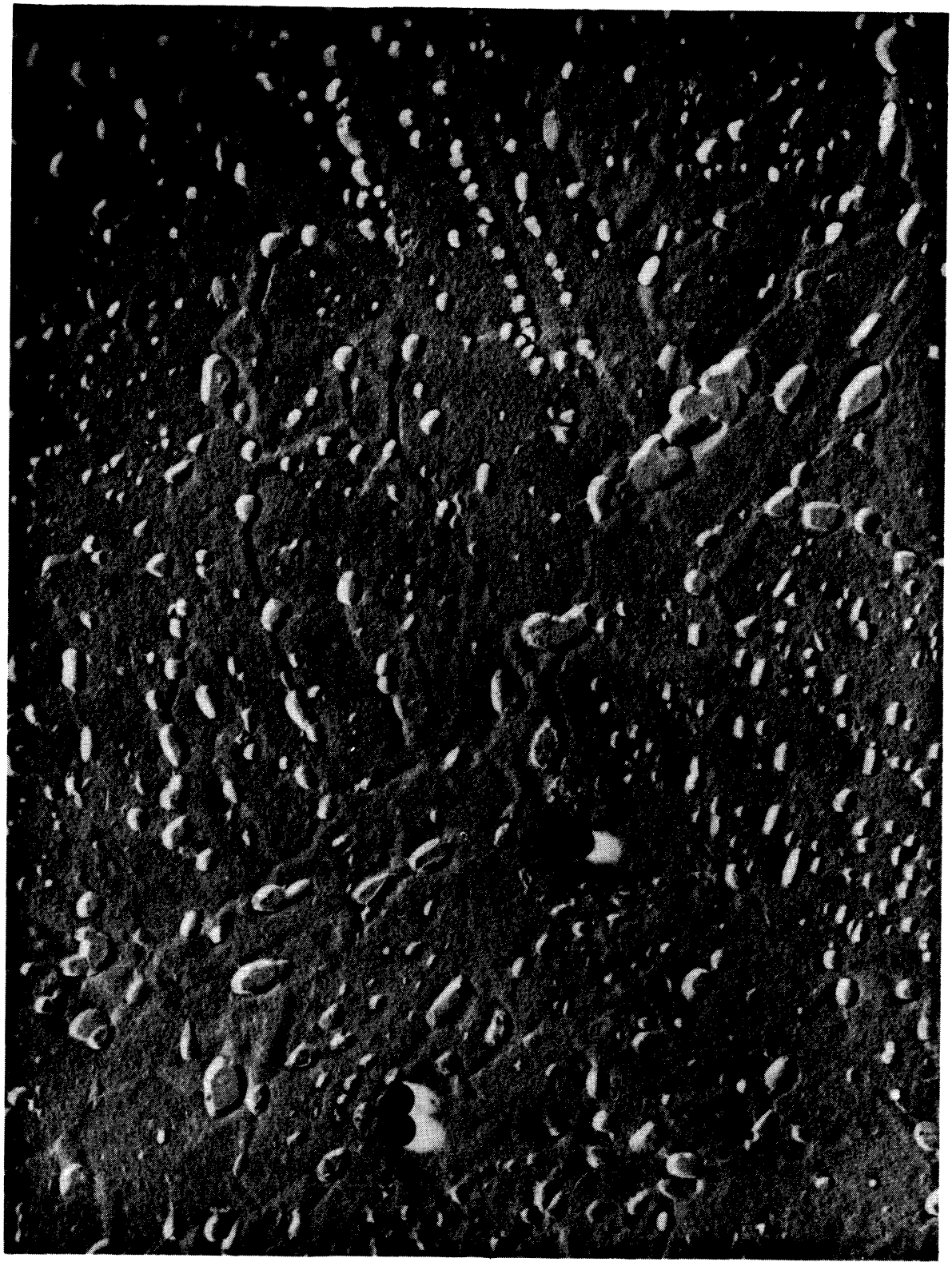


Figure 12. Electron Micrograph of Structure Produced by Embrittling Cycle of 1 Hour, 850°F, W.Q., Preceded by 1 Hour, 1250°F, W.Q. Electrolytic Polish. Ethereal Picric Immersion Etch - 10 Seconds. Magnification 20,000 x

Figure 13. Electron Micrograph of Structure Produced by Embrittling Cycle of 10 Hours, 850°F, W.Q. Preceded by 1 Hour, 1250°F, W.Q. Electrolytic Polish. Ethereal Picric Immersion Etch-10 Seconds. Magnification 20,000 x

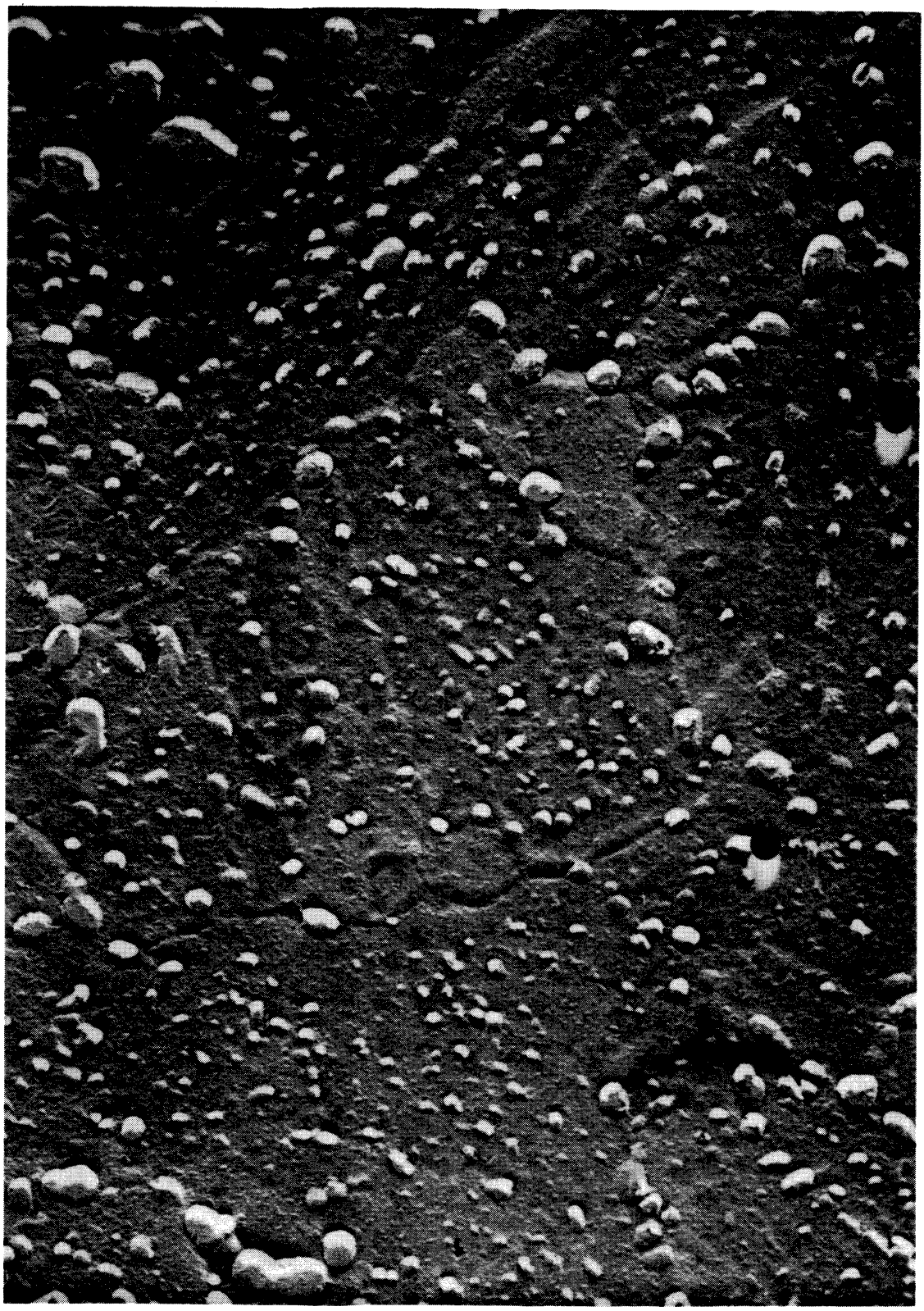


Figure 14. Electron Micrograph of Structure Produced by Embrittling Cycle of 50 Hours, 850°F, W.Q. Preceded by 1 Hour, 1250°F, W.Q. Electrolytic Polish. Ethereal Picric Immersion Etch-10 Seconds. Magnification 20,000 x

Figure 15. Electron Micrograph of Structure Produced by Embrittling Cycle of 100 Hours, 850°F, W.Q. Preceded by 1 Hour, 1250°F W.Q. Electrolytic Polish. Ethereal Picric Immersion Etch-10 Seconds. Magnification 20,000 x

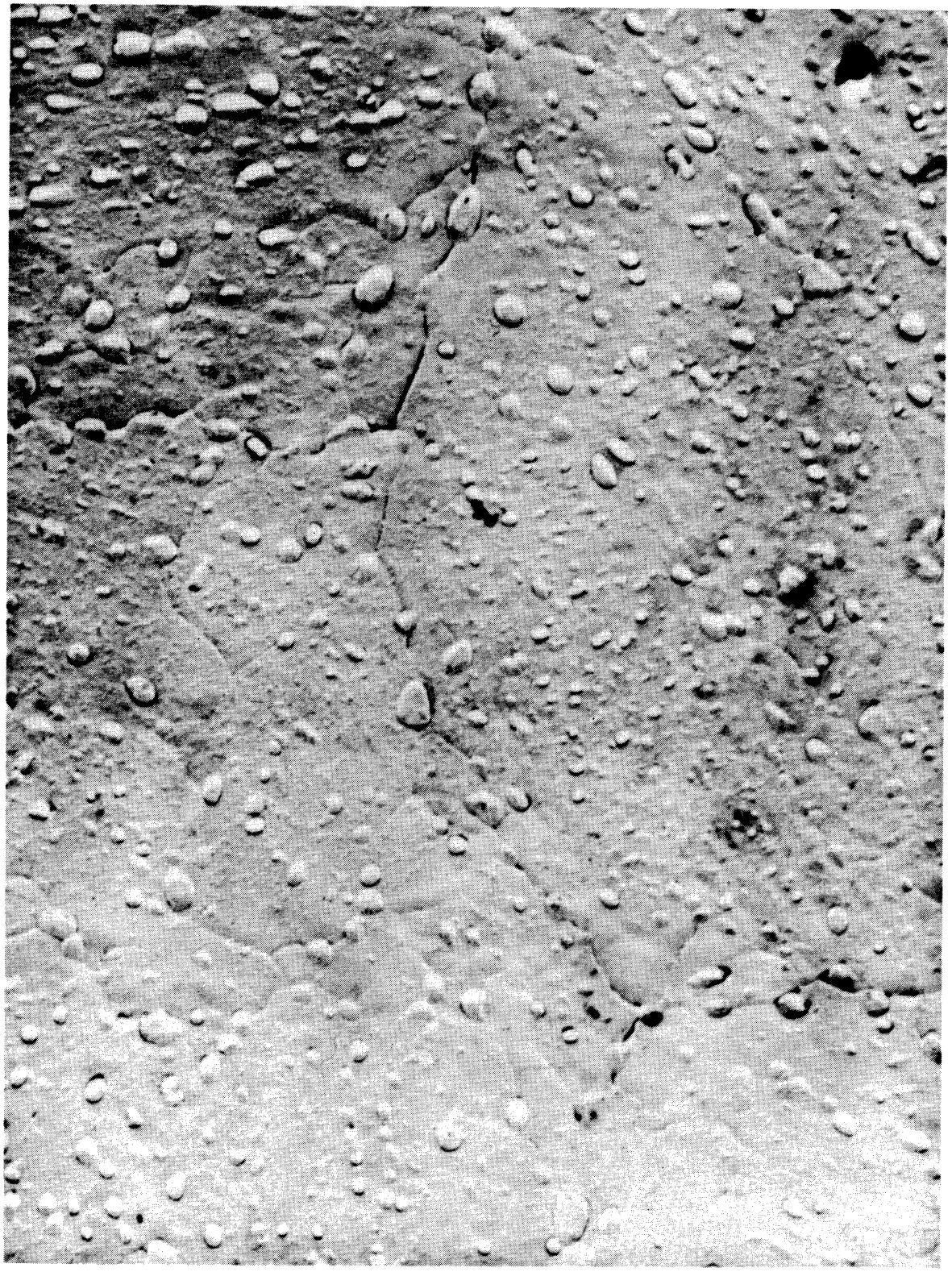


Figure 16. Electron Micrograph of Structure Produced by Embrittling Cycle of 500 Hours, 850°F, W.Q. Preceded by 1 Hour, 1250°F, W.Q. Electrolytic Polish, Ethereal Picric Immersion Etch-10 Seconds. Magnification 20,000 x

from these micrographs. It is apparent that the outlines of these prior austenite grains are grooves in the original metal surface and not an etching stain. The particles appearing in the matrix and grain boundaries are in relief on the metal surface and not holes created by an etching reaction which eats away the particles, leaving the matrix behind.

Carbon-14 Studies

The object of the carbon-14 experiments was to attempt to follow the distribution of dissolved carbon and combined carbon in the form of carbides in a tough and temper-embrittled structure. Prior to the actual microscopic observation for carbide distribution, considerable effort was necessary to prepare samples with radioactive carbon-14. It was first necessary to obtain a material free of carbon with the same alloy content as was used for the other work in this investigation. Next, the carbon-14 had to be introduced by carburization prior to the actual heat treatments developing tough and temper-embrittled structures. Finally, the evaluation and detection of the radioactive carbon distribution had to be made using autoradiographic and counting rate determination techniques.

Decarburization

Material that was used for the radioactive carbon work was initially obtained from the SAE 3140 steel as received bar stock previously mentioned. Thin slugs of about 0.06 inches were cut from 0.75 diameter bars. These slugs were then decarburized in a wet hydrogen atmosphere at 1550°F for 100 hours. This procedure eliminated most of the carbon in the original steel. A typical photomicrograph of a decarburized sample is presented in Figure 17. Each slug after decarburization was examined microscopically to be sure that decarburization was accomplished.

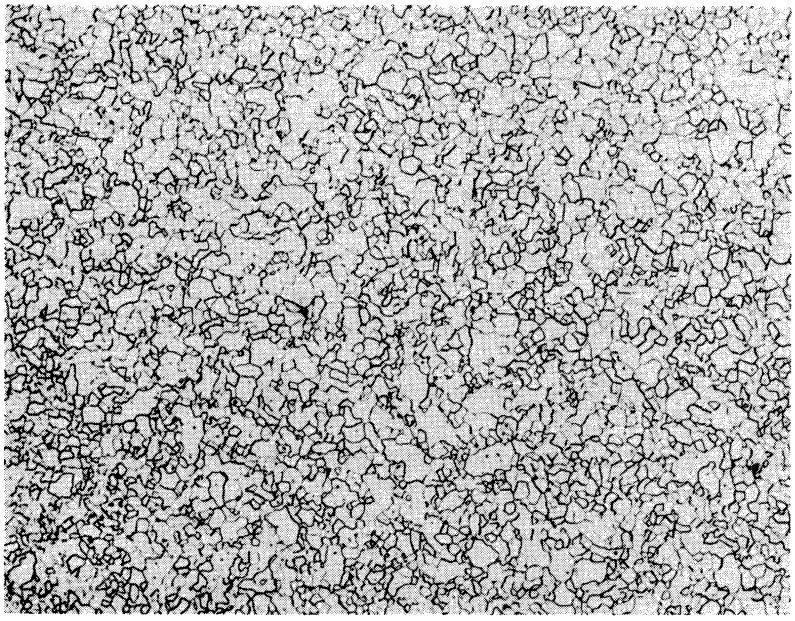


Figure 17. SAE 3140 Steel Decarburized 100 Hours at 1550°F in Wet Hydrogen. Electrolytic Polish. Nital Etch. Magnification 100 x

Recarburization

After the decarburized slugs had been examined microscopically they were recarburized with a mixture containing active and inactive carbon atoms. Carburization was accomplished in a steel welded capsule for 72 hours at 1700°F. Table V lists the compositions of the various charges used for introducing radioactive carbon-14 into the metal. In all, nine carburization experiments were performed as listed. These resulted in a total of 27 specimens containing carbon-14 for subsequent autoradiographic examination. After carburization each sample was examined microscopically to see whether or not a homogeneous distribution of carbon was accomplished. In each case satisfactory carburization had taken place. A typical carburized structure is shown in Figure 18. This sample was taken from Carburizing Run Number 9. Although no chemical analysis was performed on any of the carburized samples to determine the extent of carbon restoration, an estimate of the carbon content was made microscopically. These estimates appear in the last column of Table V.

Autoradiography

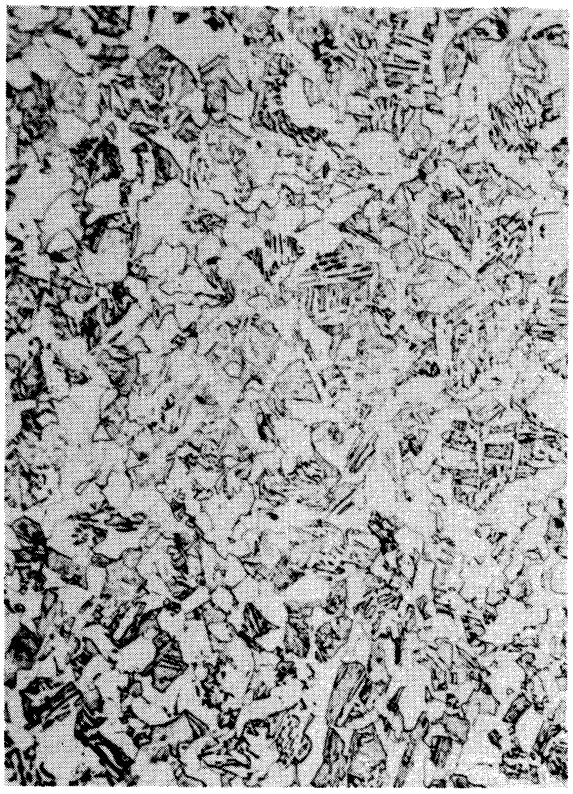
After carburization the samples were heat treated to give tough and temper-embrittled structures. Autoradiographs of these samples were taken using the stripping film technique.[148] In these experiments four variables were considered. These were: 1) effect of etching time, 2) effect of film exposure time, 3) effect of specific activity level, and 4) effect of carbon level.

In Figures 19 and 20 are shown typical autoradiographs obtained on tough and temper-embrittled samples of Carburizing Run Number 5 showing the effect of etching time on the amount and distribution of carbon in the steel. Both autoradiographs were obtained with 6 day exposures.

TABLE V. CHARGE DISTRIBUTION FOR CARBON-14 CARBURIZING RUNS

Carbon-14 Carburizing Run No.	Active* $BaCO_3$, mg.	Inactive $BaCO_3$, mg.	Charcoal mg.	Weight of Metal, mg.	Estimated Carbon Content After Carburizing, %
1	10.0	220.0	15.0	1405.8	0.4
2	4.8	224.5	15.0	1450.3	0.4
3	11.1	220.3	15.0	1410.2	0.4
4	10.2	220.6	15.4	1390.7	0.4
5	20.1	210.0	15.0	1435.1	0.4
6	3.1	113.2	7.1	1769.8	0.4
7	2.9	13.1	1.0	2024.2	0.1
8	4.7	31.2	2.2	2155.6	0.2
9	5.1	47.6	3.2	1589.1	0.3

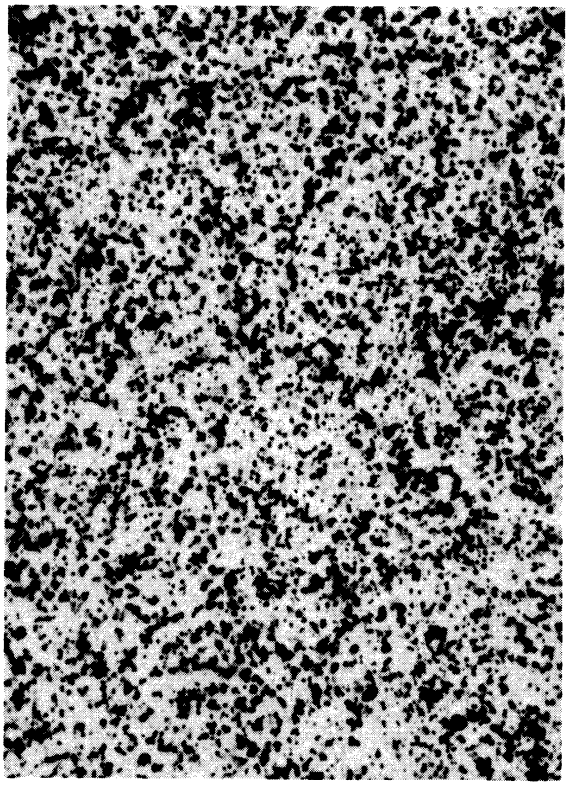
* Specific Activity of $BaCO_3$ - 0.0282 millicuries per milligram of $BaCO_3$.



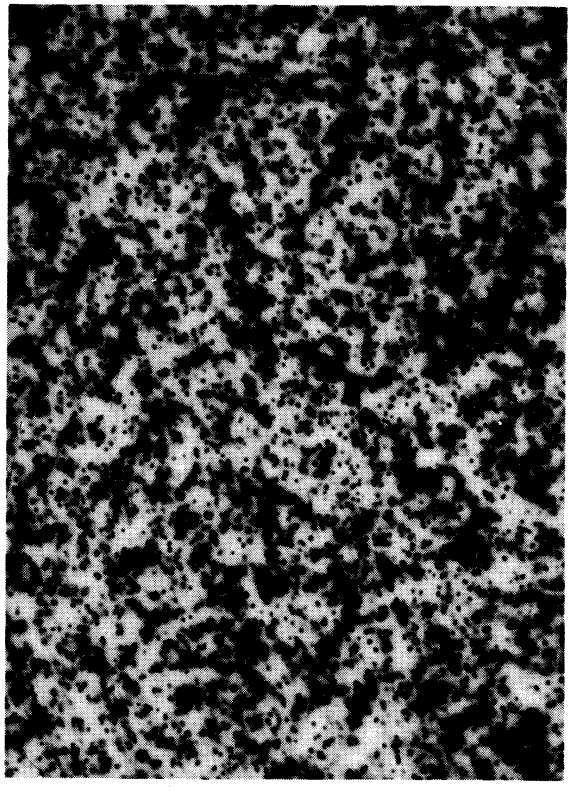
a) Magnification 500 x

b) Magnification 1000 x

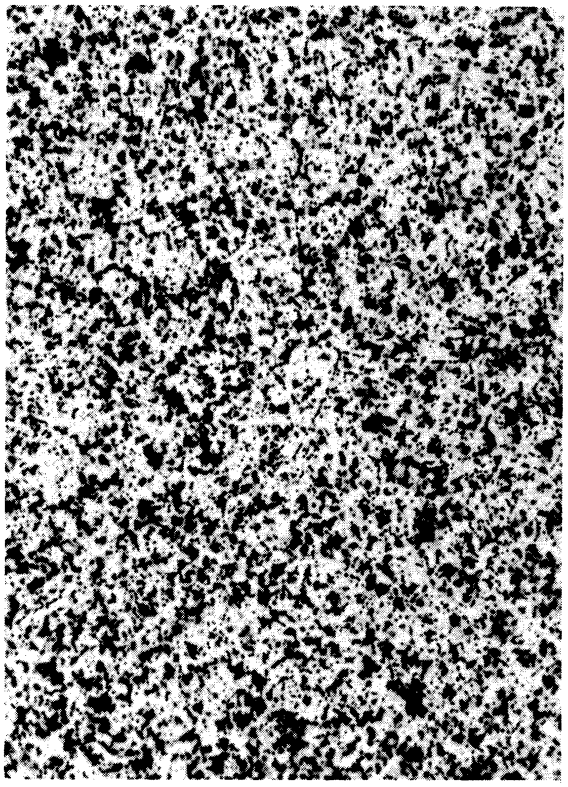
Figure 18. Photomicrographs Illustrating the Uniformity of Carbon Distribution after Carburizing a Decarburized Sample of SAE 3140 Steel 72 Hours at 1700°F. Sample Taken from Carburizing Run Number 9. Hand Polished, Nital Etch.



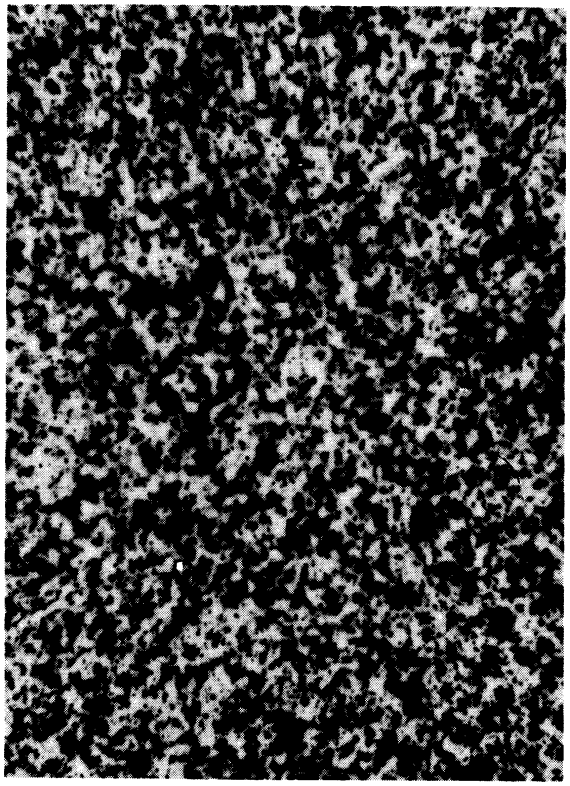
a) 1 Hour, 1250°F, W.Q.
Tough Condition. Focus on Metal



b) 1 Hour, 1250°F, W.Q.
Tough Condition. Focus on Film

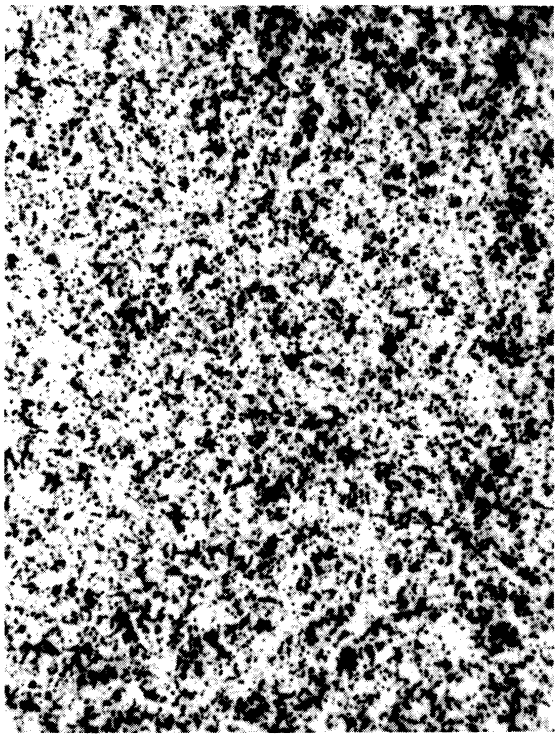


c) Tough Condition + 100 Hours,
850°F, W.Q. Focus on Metal

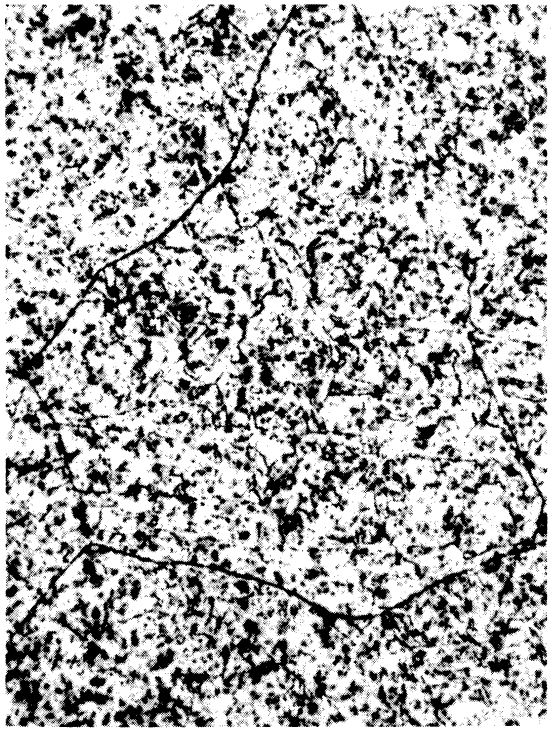


d) Tough Condition + 100 Hours,
850°F, W.Q. Focus on Film

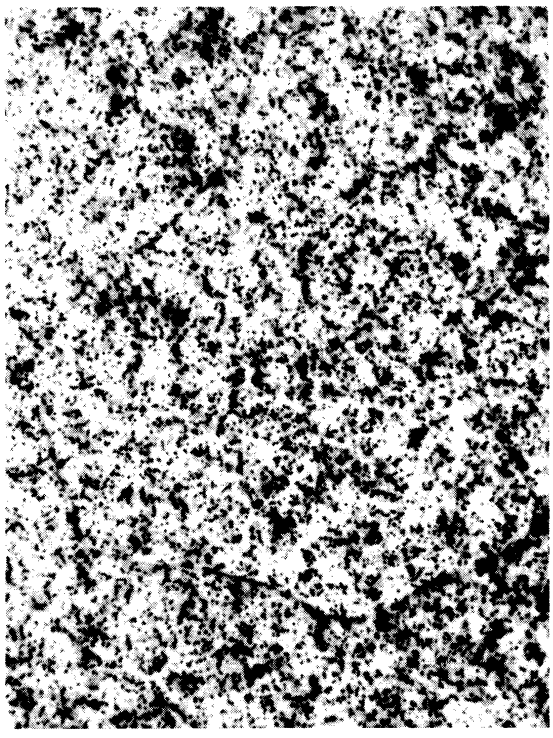
Figure 19. Six Day Exposure Autoradiograph on Samples of Carburizing Run Number 5. Carburized with 20.1 mg. of Active Barium Carbonate. Hand Polished, Ethereal Picric Immersion Etch-10 Seconds. Magnification 1000 x



a) 1 Hour, 1250°F, W.Q., Tough Condition
Focus on Film



b) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Metal



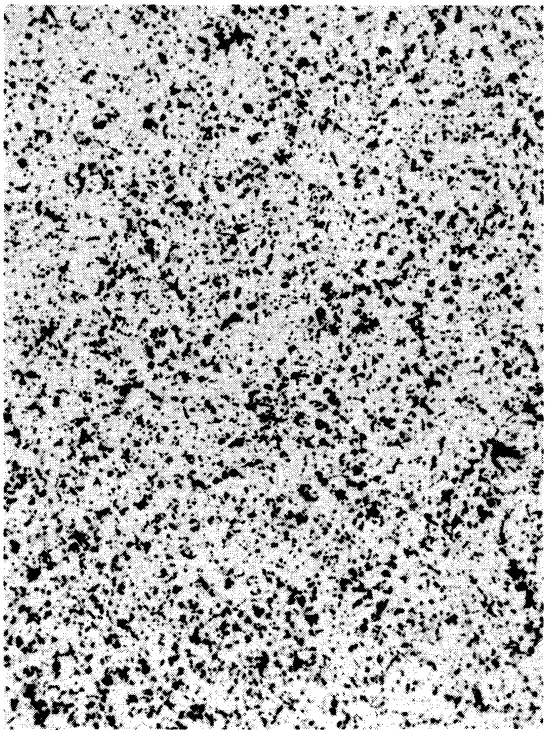
c) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Film

Figure 20. Six Day Exposure Autoradiograph on Samples of Carburizing Run Number 5. Carburized with 20.1 mg. of Active Barium Carbonate Hand Polished. Ethereal Picric Immersion Etch-2.5 Minutes. Magnification 1000 x

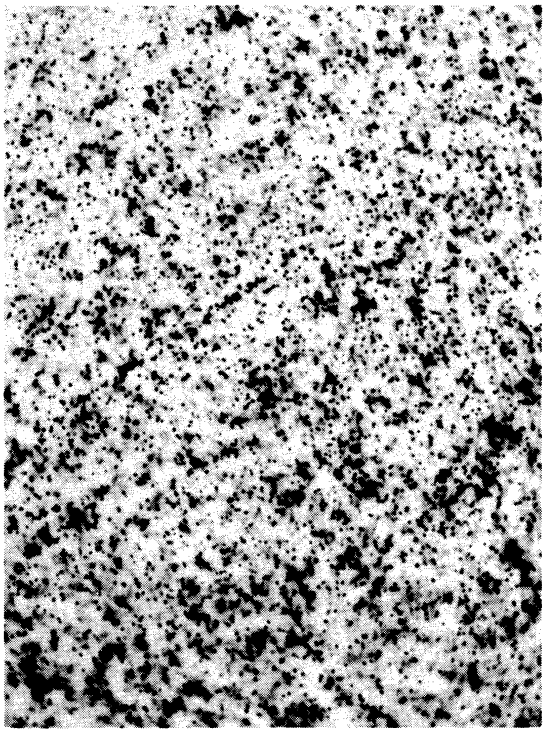
Figure 19 compares the tough and temper-embrittled structures as revealed by a 10 second ethereal picric immersion etch. The tough sample shows no grain boundary attack; whereas, the temper-embrittled structure shows a slight grain boundary attack even with this light etch. The black specks appearing above the grain boundaries in the embrittled structure are the result of carbon-14 particles that have exposed the film emulsion in intimate contact with the metallic surface. Figure 20 shows the same samples etched for two and one-half minutes in ethereal picric solution. In this case the etchant has attacked the grain boundary of the embrittled steel to a great extent. Again there are activity centers directly above the delineated grain boundaries in the embrittled structure.

In Figure 21 is shown the microstructure obtained from the samples used for Figure 20 but with a three and one-half day autoradiographic exposure. It can be noted that the activity in Figure 21 is considerably less than that of Figure 20, and that activity centers that appear directly above the grain boundaries are also found in the embrittled structure.

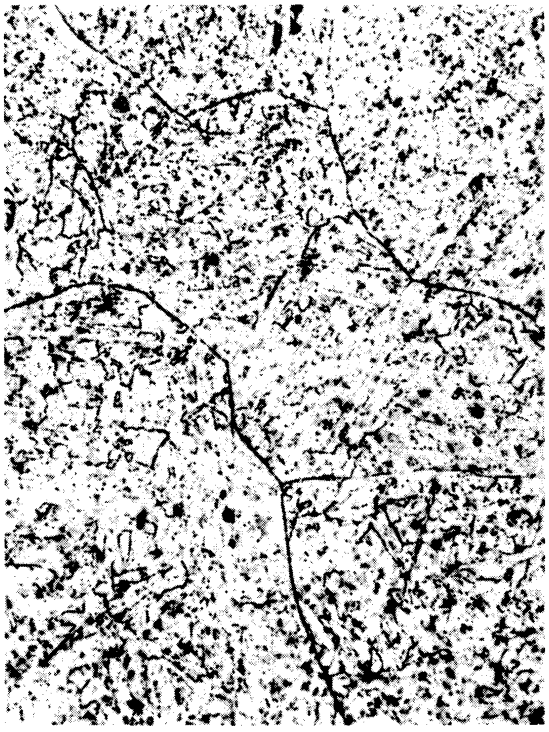
A series of autoradiographs showing the effect of specific activity level on the microstructures of tough and temper-embrittled steels is presented in Figures 22 and 23. These have been taken on samples etched two and one-half minutes with three and one-half day film exposures. The comparison can be further extended by including Figure 21 previously shown. Figures 24 and 25 compare the effect of specific activity level on the microstructure of tough and temper-embrittled steels at a six day exposure level.



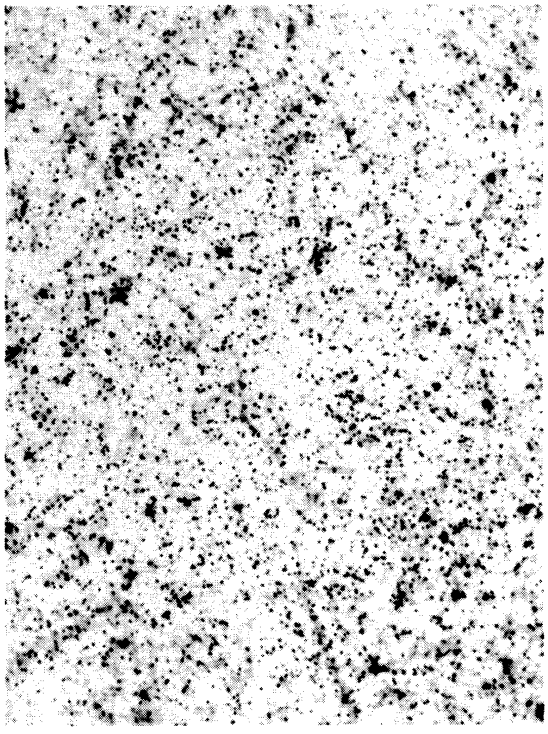
a) 1 Hour, 1250°F, W.Q., Tough Condition. Focus on Metal



b) 1 Hour, 1250°F, W.Q., Tough Condition. Focus on Film

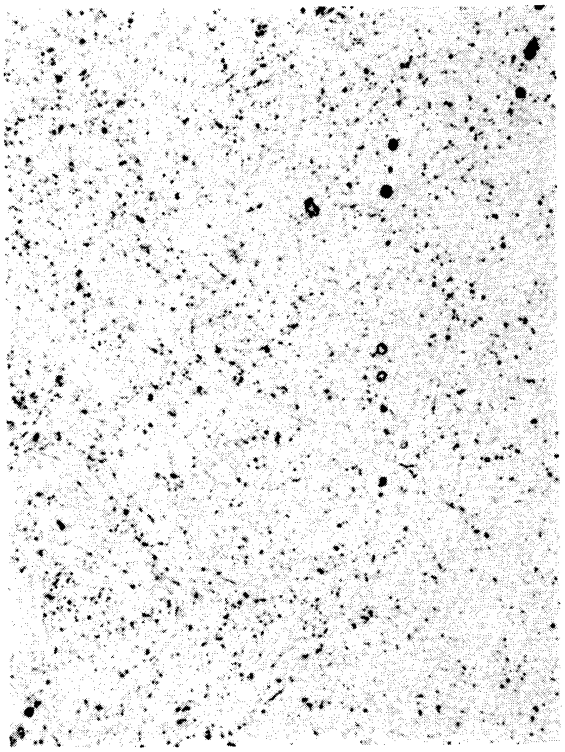


c) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Metal

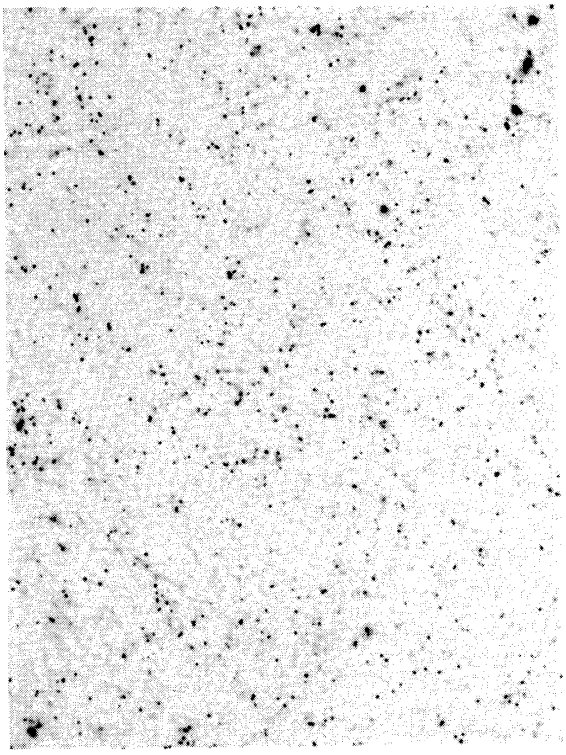


d) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Film

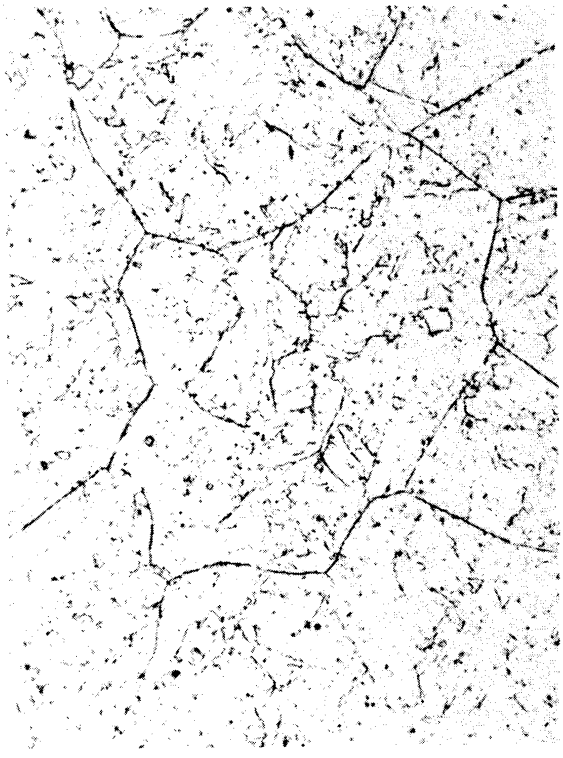
Figure 21. Three and One-Half Day Exposure Autoradiograph on Samples of Carburizing Run Number 5. Carburized with 20.1 mg. of Active Barium Carbonate. Hand Polished. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x



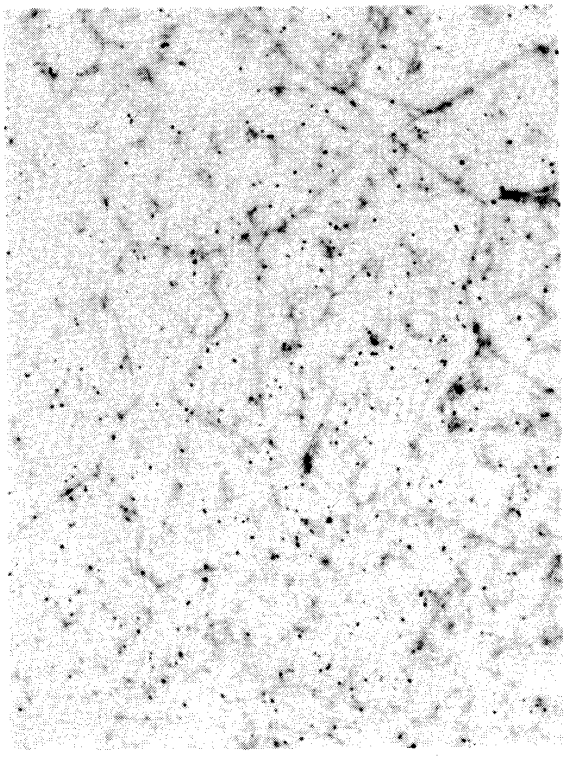
a) 1 Hour, 1250°F, W.Q., Tough Condition. Focus on Metal



b) 1 Hour, 1250°F, W.Q., Tough Condition. Focus on Film

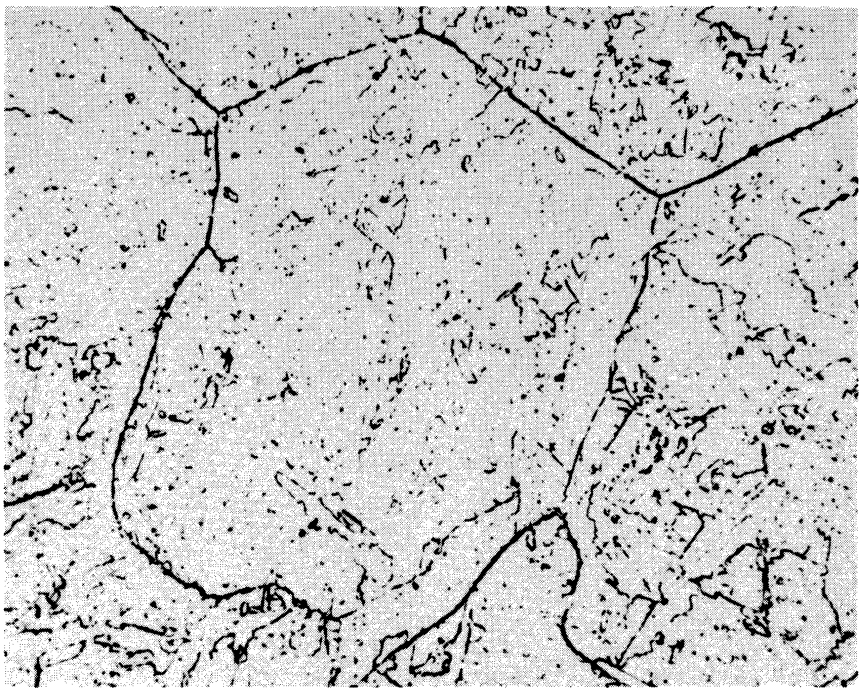


c) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Metal

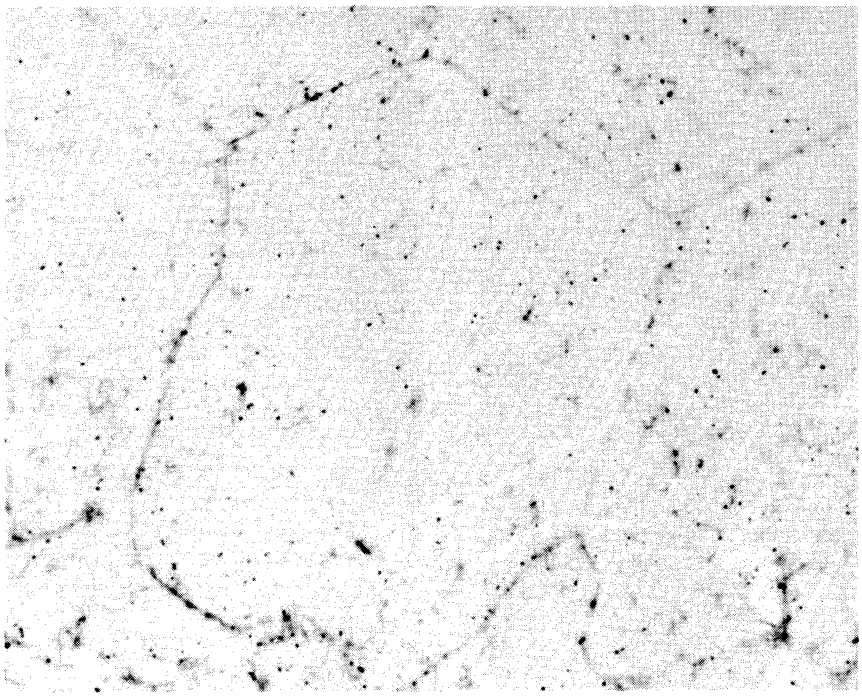


d) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Film

Figure 22. Three and One-Half Day Exposure Autoradiograph on Samples of Carburizing Run Number 2. Carburized with 4.8 mg. of Active Barium Carbonate. Hand Polished. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x

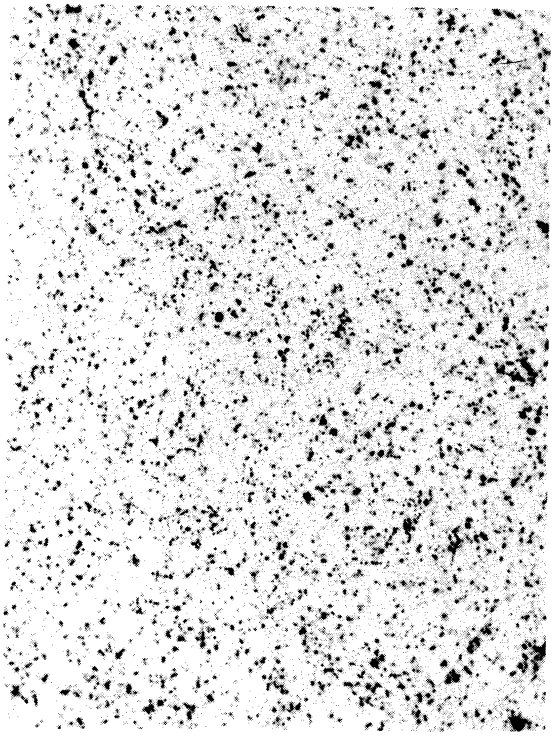


e) Tough Condition + Embrittled 500 Hours, 850°F, W.Q. Focus on Metal

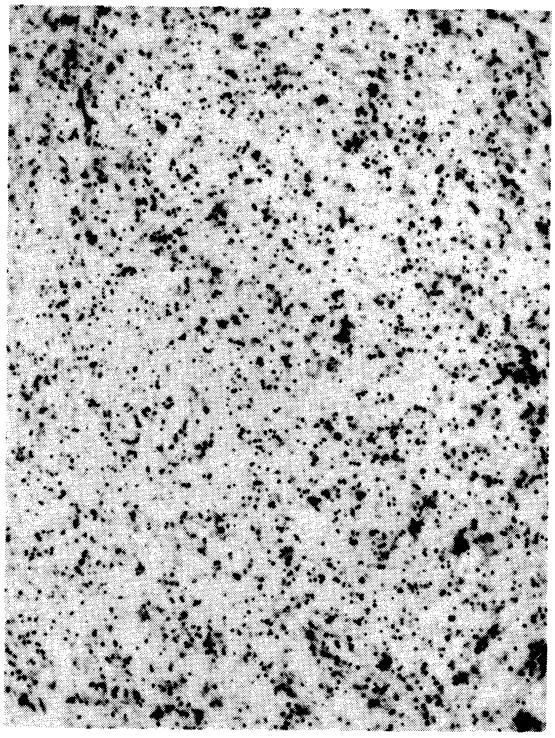


f) Tough Condition + Embrittled 500 Hours, 850°F, W.Q. Focus on Film

Figure 22. Three and One-Half Day Exposure Autoradiograph on Samples of Carburizing Run Number 2. Carburized with 4.8 mg. of Active Barium Carbonate. Hand Polished. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x

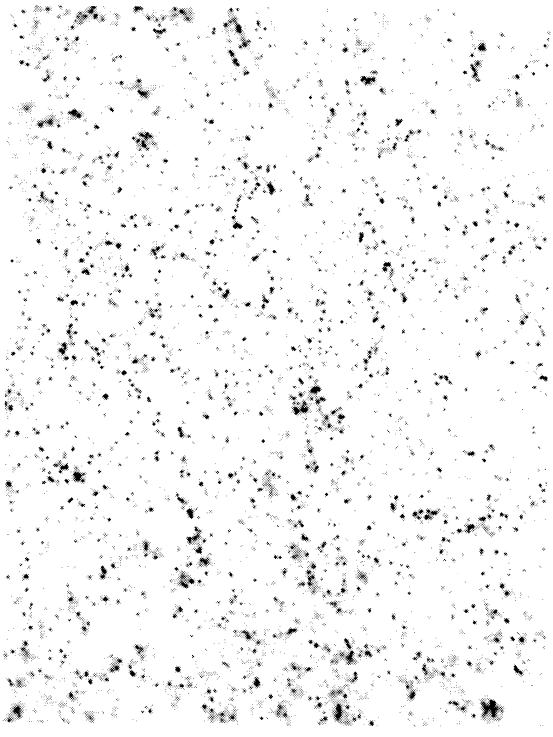


a) 1 Hour, 1250°F, W.Q., Tough Condition. Focus on Metal



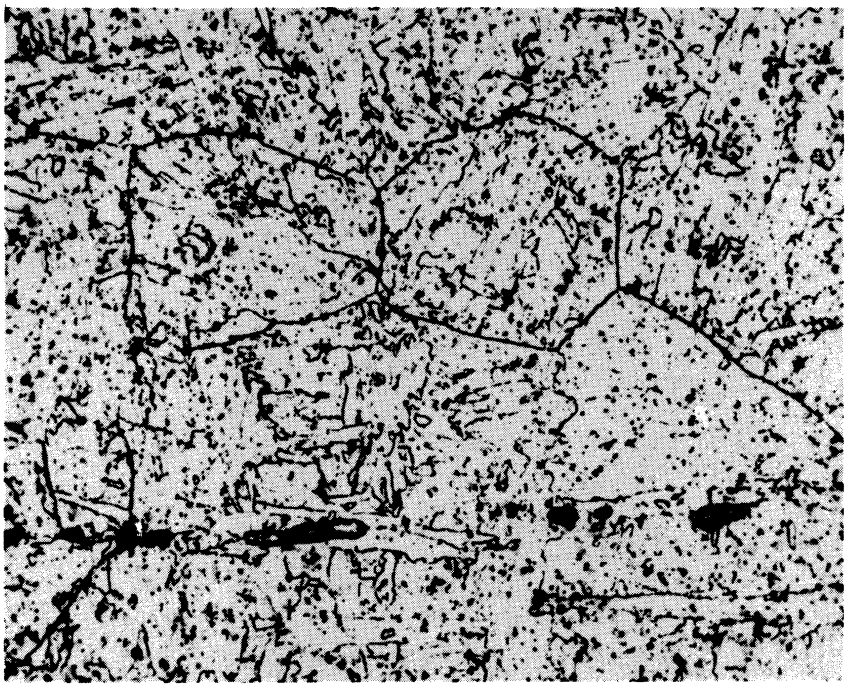
b) 1 Hour, 1250°F, W.Q., Tough Condition. Focus on Film

c) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Metal

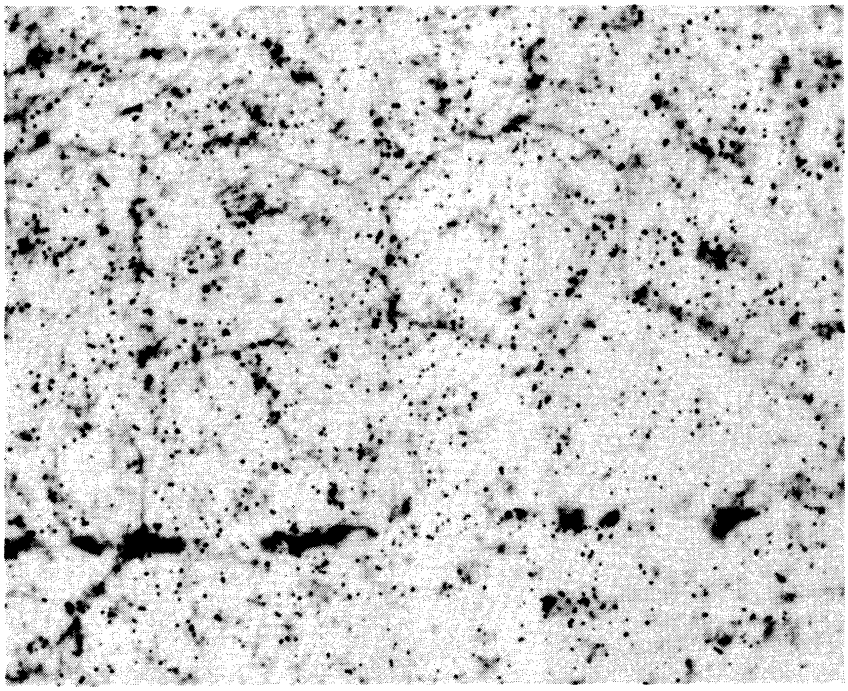


d) Tough Condition + Embrittled 100 Hours, 850°F, W.Q. Focus on Film

Figure 23. Three and One-Half Day Exposure Autoradiograph on Samples of Carburizing Run Number 3. Carburized with 11.1 mg. of Active Barium Carbonate. Hand Polished. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x

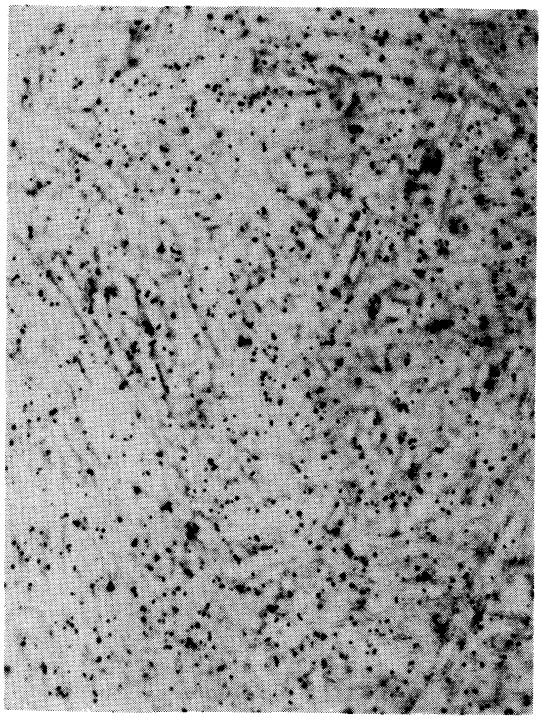


e) Tough Condition + Embrittled 500 Hours, 850°F, W.Q. Focus on Metal

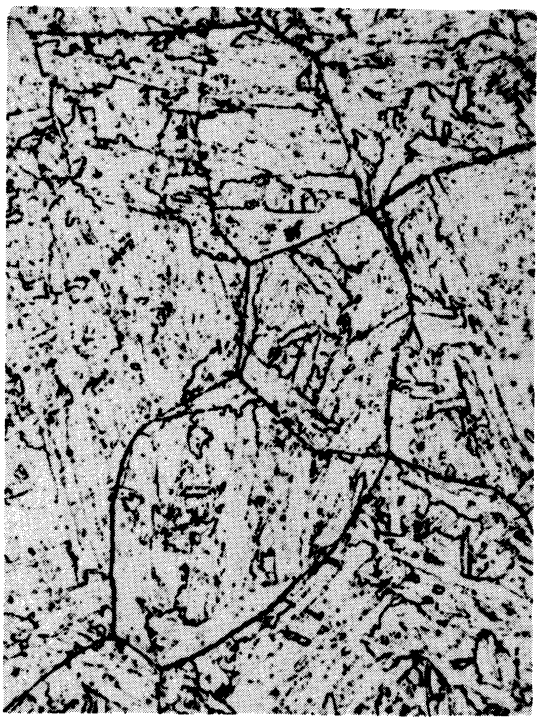


f) Tough Condition + Embrittled 500 Hours, 850°F, W.Q. Focus on Film

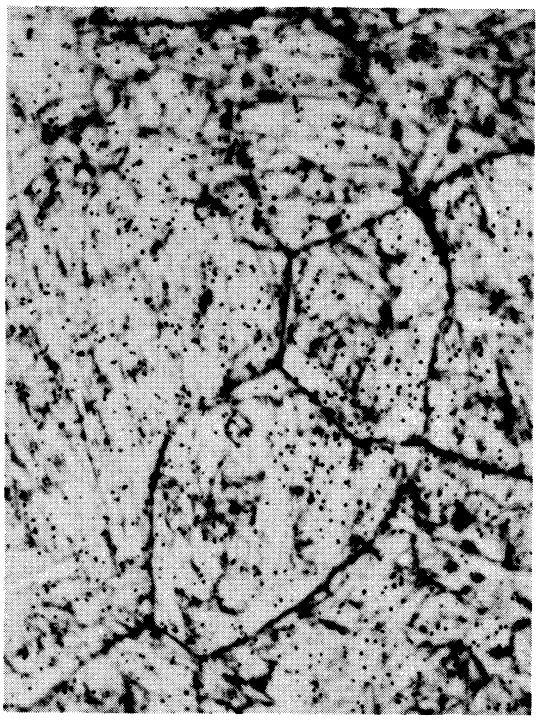
Figure 23. Three and One-Half Day Exposure Autoreadiograph on Samples of Carburizing Run Number 3. Carburized with 11.1 mg. of Active Barium Carbonate. Hand Polished, Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x



a) 1 Hour, 1250°F, W.Q., Tough Condition
Focus on Film

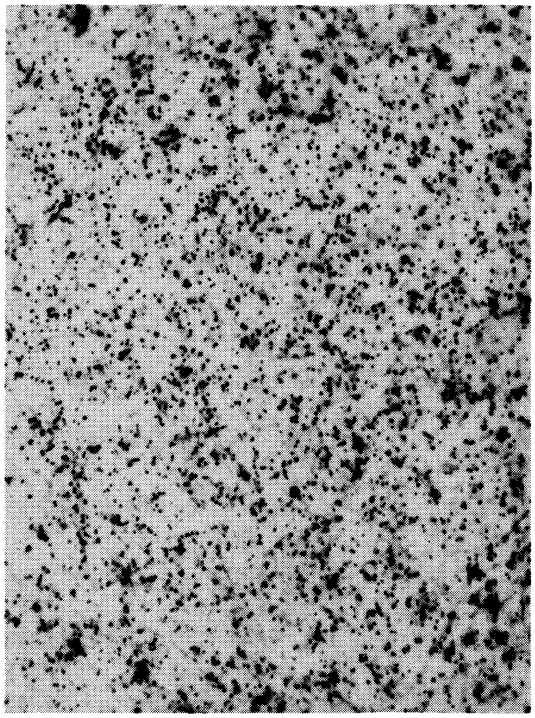


b) Tough Condition + Embrittled 500 Hours, 850°F, W. Q. Focus on Metal

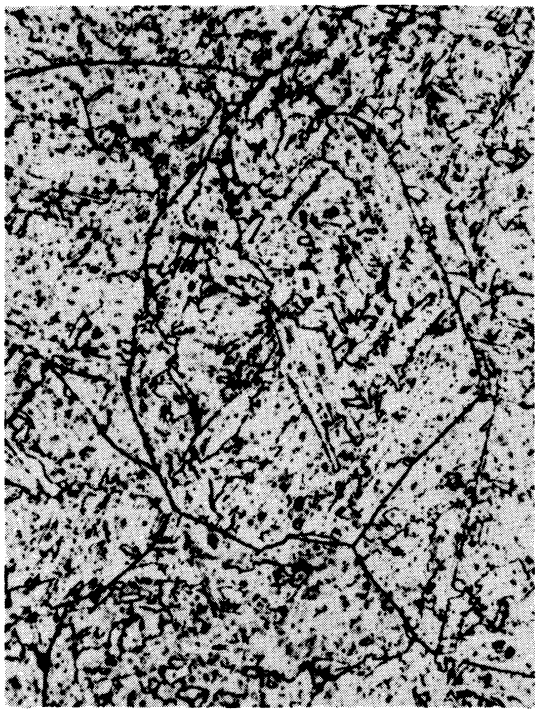


c) Tough Condition + Embrittled 500 Hours, 850°F, W.Q. Focus on Film

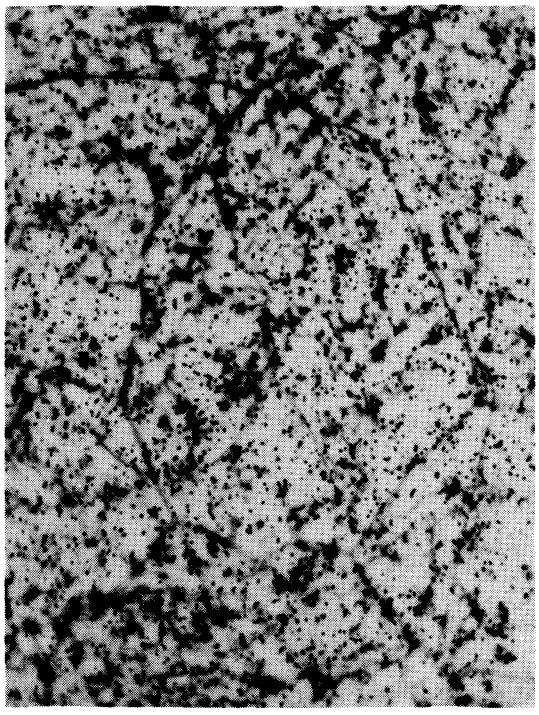
Figure 24. Six Day Exposure Autoradiograph on Samples of Carburizing Run Number 2. Carburized with 4.8 mg. of Active Barium Carbonate. Hand Polished. Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x



a) 1 Hour, 1250°F, W.Q., Tough Condition
Focus on Film



b) Tough Condition + Embrittled 500
Hours, 850°F, W.Q. Focus on Metal



c) Tough Condition + Embrittled 500
Hours, 850°F, W.Q. Focus on Film

Figure 25. Six Day Exposure Autoradiograph on Samples of Carburizing Run Number 3. Carburized with 11.1 mg. of Active Barium Carbonate. Hand Polished, Ethereal Picric Immersion Etch - 2.5 Minutes. Magnification 1000 x

An examination of the presented autoradiographs shows that although some carbon is found in the prior austenite grain boundaries of a temper-embrittled steel, it is by no means extensive. The presence of activity centers in the grain boundaries is certainly expected since inspection of microstructures with light and electron microscopes reveal their presence. However, no gross segregation of carbon or carbides to the prior austenite grain boundaries appears evident. Nor can a continuous film of carbon or carbide be detected by the autoradiographs obtained in this investigation. Although in some cases a line-up of activity centers can be observed in the autoradiographs of the embrittled structures, it is not believed that these are due to temper-embrittlement since similar line-ups can be seen in the tough structures also. Attempts at reducing the total carbon content of the steel were made in an effort to produce a more favorable condition for detecting any segregation of carbon or carbides to the prior austenite boundaries. Autoradiographs of these samples did not reveal any greater amounts of activity centers in prior austenite grain boundaries of temper-embrittled structures.

Counting Rate Studies

Since it is known that temper-embrittled steels exhibit an intergranular fracture when broken below their transition temperature, an experiment was performed in an attempt to detect a difference in carbon-14 present in the grain boundaries of tough and temper-embrittled steels that were fractured at a temperature of dry ice. Subsize specimens containing carbon-14 were notched and broken at a temperature of dry ice and counting rates determined on their fractured surfaces. A comparison of the counting rates obtained using a gas-flow counter for a tough and temper-embrittled structure is shown in Table VI. In

Table VI the embrittled sample is shown to have given the higher counting rate, indicating that more carbon-14 is present in the grain boundaries of an embrittled structure than a tough structure. However, the results are not conclusive in that the fractured surface of the brittle sample was much rougher than the tough sample. Thus, the observed difference in counting rate can be attributed to the greater area of the roughened brittle sample's fractured surface.

TABLE VI. COUNTING RATES OBTAINED ON FRACTURED SURFACES OF TOUGH AND 500 HOUR TEMPER EMBRITTLED CARBON-14 SPECIMENS OF CARBURIZING RUN NUMBER 5 FRACTURED AT -112°F

Condition	Total Counts	Counts/Min.	Corrected Counts/Min. for Area Difference
Tough[a]	3606	1803	1803
	3841	1921	1921
Tough[b]	4011	2006	2006
	4089	2045	2045
Brittle[a]	4668	2334	2180
	4757	2379	2222
Brittle[b]	5268	2634	2460
	4878	2439	2280

[a] One half of specimen.

[b] Other half of same specimen.

Tough Condition - Normalized, austenitized, + 1 hour at 1250°F, W.Q.

Brittle Condition - Tough condition + 500 hours at 850°F, W.Q.

Counting rates on polished and etched samples of tough and temper-embrittled structures were also determined. Table VII presents these results on samples obtained from carburizing Run Number 3. Again no significant difference in surface activity was observed. The small difference that was observed can be accounted for by the inhomogeneities of the samples tested.

Vacuum Fusion Analysis

In the course of this investigation it was observed that the prepared carbon-14 specimens responded differently to the ethereal picric etch than the material used for the impact and fatigue specimens. The prior austenite grain boundaries in the brittle condition of the specimens that were run through the decarburizing-carburizing cycle were attacked to a lesser extent by the ethereal picric etch than the material which had not been given the carburizing cycle treatment. The difference was thought to be caused by a possible change in nitrogen content of the materials that had been decarburized in wet hydrogen. To check this premise vacuum fusion analyses were obtained on samples in the as received, decarburized, recarburized, tough, and temper-embrittled conditions. The results are presented in Table VIII. No substantial change in nitrogen was observed in any of the samples tested.

Minor Phase Extraction Studies

Since electron microscope examination of tough and temper-embrittled structures revealed large amounts of minor phase material, which was thought to be some carbide phase, it was hoped that an analysis of these carbides might reveal a clue as to the process of temper-

TABLE VII. SURFACE COUNTING RATES ON POLISHED AND ETCHED SAMPLES OF CARBURIZING RUN NUMBER 3 FOR TOUGH AND 500 HOUR TEMPER-EMBRITTLED CONDITIONS

Condition	Total Counts	Counts/Min.	Corrected Counts/Min. for Area Difference
Tough	8,519	1,704	1,178
Tough	16,644	1,664	1,151
Brittle	6,355	1,271	1,271
Brittle	12,957	1,296	1,296

Tough Condition - Normalized, austenitized, + 1 hour at 1250°F, W.Q.

Brittle Condition - Tough condition + 500 hours at 850°F, W.Q.

TABLE VIII. VACUUM FUSION ANALYSIS OF SAE 3140 STEEL IN VARIOUS CONDITIONS OF HEAT TREATMENT

Sample No.*	% N_2	% H_2	% O_2
1	0.01025	0.000040	0.00127
2	0.00816	0.000065	0.01460
3	0.00848	0.000029	0.00111
4	0.01360	0.000066	0.00621
5	0.00963	0.000100	0.00424
6	0.00845	0.000044	0.00131

* 1. As received
2. Decarburized in wet H_2 at 1550°F for 100 hours
3. 500 hour at 850°F, temper-embrittled, from impact bar
4. 500 hour at 850°F, temper-embrittled, from carburized specimen
5. Tough - from carburized specimen
6. Tough - from impact bar

embrittlement. Maloof[109] has reported a change in chromium content in the complex carbides brought about by the temper-embrittling process. However, in Maloof's work the comparison was made on samples that were quenched and slowly cooled through the susceptible temperature region, and whether or not the change in chromium content was responsible for the embrittlement was not made clear by his work.

Extractions were obtained on material in the tough and temper-embrittled conditions. In addition, extractions were obtained on material put through the reversible temper-embrittlement heat treatment, i.e., 1 hour at 1250°F + 500 hours at 850°F + 1 hour at 1250°F. This last condition was performed as a check run; for, if temper-embrittlement is associated in some manner with carbide composition, the removal of temper-embrittlement should also cause a revertment of the carbides to the condition that exists in the tough structure. The extractions were analyzed using X-ray diffraction and spectrographic techniques.

X-Ray Analysis

The "d"-values obtained from the Debye-Scherrer X-ray diffraction powder patterns are listed in Table IX. Along with the values obtained on the three heat treatments investigated is presented the "d"-values for Fe_3C obtained from the literature.[142] A comparison of the "d"-values obtained on the extracts with those of Fe_3C shows that Fe_3C exists in the extracts. The four "d"-values that are not accounted for by Fe_3C have not been conclusively identified. However, they seem to agree with the patterns obtained on chromium carbides of type 1) Cr_7C_3, 2) Cr_3C_2, or 3) CrC. The unidentified "d"-values are probably from chromium carbides, since the existence of chromium in the extracts was verified by spectroscopic analysis.

TABLE IX. COMPARISON OF "d" - VALUES OBTAINED BY X-RAY DIFFRACTION ON EXTRACTS OF AN SAE 3140 STEEL AND AN IRON CARBIDE STANDARD

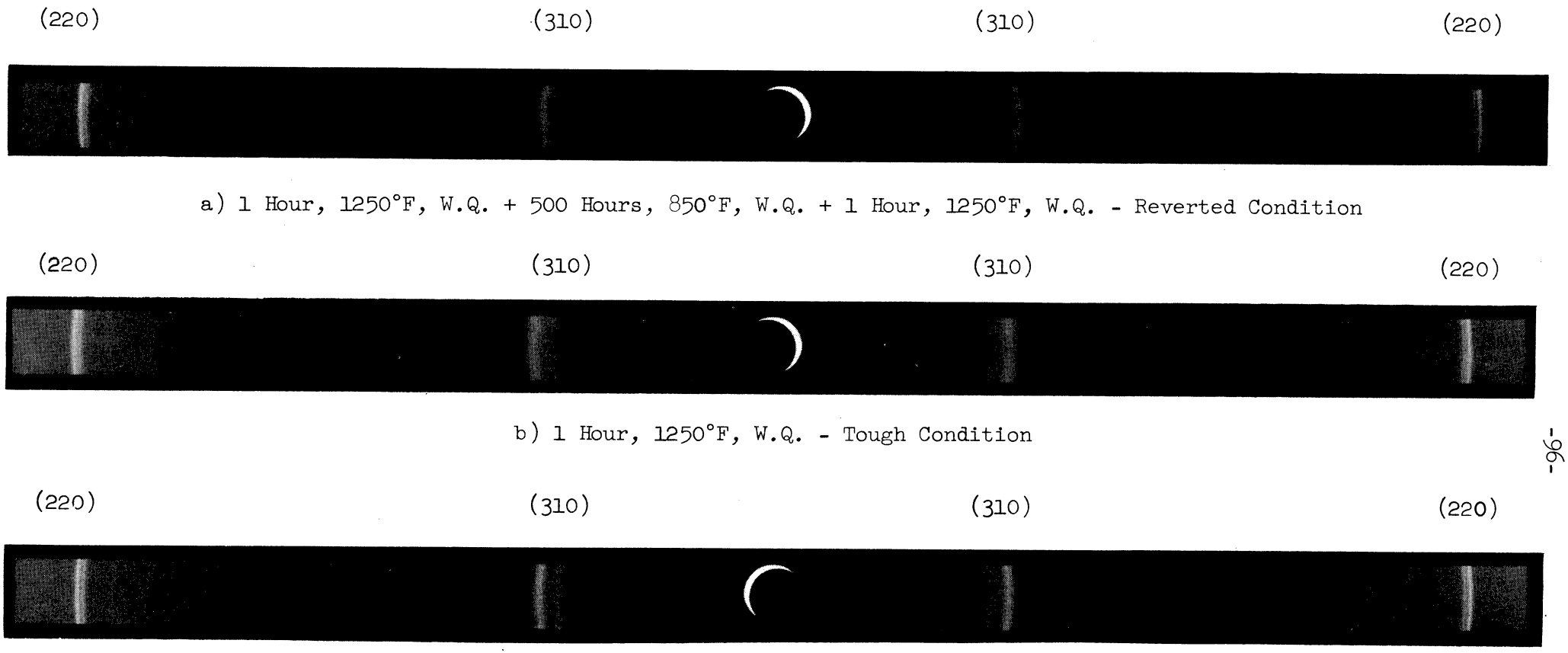
Reversible Heat Treatment[1]		Tough[2]		Brittle[3]		Fe_3C	
d	I	d	I	d	I	d	I
2.353	5	2.352	4	2.357	4	2.39	M
2.231	2	2.221	1	2.228	1	2.265	W
2.187	2	2.186	1	2.176	1		
2.075	6	2.078	6	2.072	5	2.115	M
2.041	6	2.040	6	2.032	5	2.07	M
1.995	10	1.998	10	1.989	10	2.04	S
1.954	6	1.954	6	1.942	5	1.99	M
1.854	6	1.851	5	1.849	6	1.86	S
1.828	6	1.828	5	1.824	6		
1.745	4	1.739	3	1.738	3	1.766	W
1.668	4	1.672	3	1.682	3	1.69	W
1.623	1	1.627	1	1.633	1	1.645	VW
1.568	4	1.569	3	1.584	3	1.593	W
1.528	1	1.527	1	1.543	1	1.55	VW
1.497	3	1.498	2	1.508	2	1.512	W
1.393	1	1.408	1	1.406	1	1.415	W
1.318	6	1.320	5	1.327	5	1.335	MS
1.250	1	1.249	1	1.258	1	1.262	VW
1.215	8	1.212	9	1.218	9	1.22	M
1.184	1	1.184	1	1.191	1		
1.154	6	1.156	5	1.161	5	1.165	S
1.146	3	1.148	2	1.134	3	1.133	S
1.120	9	1.122	9	1.113	9	1.127	S
1.099	5	1.098	6	1.091	5	1.112	S
1.050	1	1.049	1	1.043	1		
0.983	9	0.984	9	0.979	9	0.987	S

1. Brittle Condition + 1 Hour at 1250°F, W.Q.
2. Normalized, Austenitized, + Tempered 1 Hour at 1250°F, W.Q.
3. Tough Condition + 500 Hours at 850°F, W.Q.

The X-ray patterns obtained from the extracts were identical for all three heat treatments studied. Thus, if temper-embrittlement is caused by a new chromium or iron carbide phase forming at the embrittling temperature, there is no X-ray evidence to support this contention. However, the X-ray evidence does not preclude the possibility of a modified carbide being responsible for temper-embrittlement. Since chromium and iron have the same atomic radius, it is quite likely that the solution of chromium in an iron carbide lattice would have little effect on the resulting X-ray diffraction characteristics.

Back Reflection X-Ray Studies

The back-reflection X-ray patterns obtained on structures produced by 1) one hour at 1250°F plus 500 hours at 850°F plus one hour at 1250°F, 2) one hour at 1250°F, and 3) one hour at 1250°F plus 500 hours at 850°F are presented in Figure 26. The two pairs of lines near the center and ends of the films are associated with the (310) and (220) planes of the iron lattice, respectively. A comparison of the line broadness of the three patterns shows that lines of the temper-embrittled structure are sharper and better defined than those of the tough and reheated structures. This observation is in direct contrast to that reported by McKay and Arnold,[115] Weill,[140,141] and Maloof,[109] who attributed the line broadening to the presence of strain caused by temper-embrittlement. However, it must be pointed out that these authors examined structures obtained by slow cooling conditions which could have very well brought about a strained iron lattice due to nitride precipitation that occurs below 750°F in steels. The lack of line broadening observed on the temper-embrittled specimen in this investigation does not preclude the presence of strain existing at prior austenite grain



a) 1 Hour, 1250°F, W.Q. + 500 Hours, 850°F, W.Q. + 1 Hour, 1250°F, W.Q. - Reverted Condition

b) 1 Hour, 1250°F, W.Q. - Tough Condition

c) 1 Hour, 1250°F, W.Q. + 500 Hours, 850°F, W. Q. - Temper-Embrittled Condition

Figure 26. Effect of Heat Treatment on the Broadening of Iron Lattice Lines Obtained on an SAE 3140 Steel

boundaries, since it is unlikely that X-ray line broadening would result from localized regions of strain on this scale. On the other hand, the increase in sharpness that was observed on the embrittled sample could also be the result of the differences in carbon solubilities at 1250 and 850°F.

Spectrographic Analysis

The extracts as obtained from the chemical separation were also analyzed with a light spectrograph. No attempt was made to quantitatively determine the composition of the extracts. However, a qualitative examination was made of the amounts of iron and chromium present on the basis of relative intensities of suitable iron and chromium lines as described previously under Experimental Procedures. The results are shown in Table X below.

TABLE X. COMPARISON OF RELATIVE INTENSITIES OF IRON AND CHROMIUM SPECTROGRAPHIC LINES OBTAINED ON EXTRACTS FROM THREE CONDITIONS OF HEAT TREATMENTS

Condition	Intensity of Fe line 2926.58 Å	Intensity of Cr line 2855.68 Å	Intensity Ratio Cr/Fe
1 hr. at 1250°F, Tough	0.095	0.40	4.21
1 hr. at 1250°F + 500 hrs. at 850°F, Brittle	0.065	0.30	4.62
1 hr. at 1250°F + 500 hrs. at 850°F + 1 hr. at 1250°F, Tough	0.32	0.52	1.63

It can be seen that no significant change in the ratio of the relative intensity of chromium to iron occurred between the tough and temper-embrittled specimens. However, the ratio of intensity of chromium to iron did change notably in the reversible, heat treated specimen. It was expected that the ratio for this condition would be very close to that obtained on the tough specimen. The fact that no logical explanation is apparent to explain the decrease in chromium content of the carbides of the reheat treated specimen leads one to suspect the technique employed.

Electron Diffraction

Electron diffraction experiments were run on tough and temper-embrittled materials in two ways.

First, if one considers the possibility of temper-embrittlement being caused by a precipitate or film in the prior austenite grain boundaries, then an electron diffraction pattern of the grain boundaries of an embrittled material should reveal the presence of such a precipitate or film. To test this hypothesis Charpy impact fractures on tough and temper-embrittled structures were obtained at a temperature of liquid nitrogen. Electron diffraction patterns were then obtained on the fractured surfaces. Analysis of these patterns revealed the presence of iron only. Either the electron beam did not "see" any carbides in the boundary or they were not there after fracture.

Secondly, electron diffraction was obtained on tough and temper-embrittled structures etched with ethereal picric. The action of this etchant is to leave the carbide particles in relief as was shown in the electron micrographs previously presented. Distinct evidence of iron carbide existed but no other carbides could be identified. The

patterns obtained for the temper-embrittled structure appeared to be very similar to that published by Heidenreich[64] on a temper-embrittled steel supplied to him by Maloof.[109] Heidenreich has suggested that these patterns represent a transition lattice of carbide precipitates. The pattern obtained by Heidenreich was not conclusively identified but was thought to be due to a "transition lattice in the formation of carbide precipitates."

Fatigue Properties

Approximately eighty fatigue specimens were run at 0°F in this investigation to determine the effect of temper-embrittlement on the fatigue properties of an SAE 3140 steel. Twenty-two specimens were tested to establish the finite region of the S-N fatigue curves while the remainder of the specimens were used in determining the susceptibility of a tough and temper-embrittled structure to damage by cycles of overstress.

The S-N curve for the tough and temper-embrittled structures was determined by running three or four specimens of each type at three stress levels of 90,000, 85,000, and 75,000 psi. Damage determinations were obtained at cycles of overstress at 90,000, 85,000, and 80,000 psi using a runout stress of 75,000 psi. Again, either three or four specimens of each type were run at each stress level. Only five specimens were run below 75,000 psi to qualitatively determine the endurance limit.

Hardness determinations were made on all fractured fatigue specimens between the bearing surfaces at each end of the bars. These readings were corrected for the curvature of the specimen where the hardness impressions were made. The average and range in Rockwell C hardness obtained on specimens used for the establishment of the S-N curves are presented in Table XI and in Table XII for specimens used for

TABLE XI. ROCKWELL C HARDNESS OF S-N CURVE FATIGUE SPECIMENS OF AN SAE 3140 STEEL

Heat Treatment	Test Stress, psi	Average R_C Hardness	Range		No. of Specimens[3] Tested
			Maximum	Minimum	
Tough[1]	70,000	21.0	-	-	1
Tough	72,000	20.5	21	20	2
Brittle[2]	72,000	21.0	22	20	2
Tough	75,000	22.6	23	22	4
Brittle	75,000	22.1	23	21	4
Tough	85,000	22.6	23	21	4
Brittle	85,000	21.5	22	20	4
Tough	90,000	20.6	21	20	3
Brittle	90,000	20.3	21	20	3

[1] Tough - Normalized 1-1/2 hour at 1650°F + 1 hour at 1600°F, O.Q. + 1 hour at 1250°F, W.Q.

[2] Brittle - Same as tough condition + 500 hours at 850°F, W.Q.

[3] Eight readings were taken on each specimen. These were corrected for the curvature of the specimen's surface.

TABLE XII. ROCKWELL C HARDNESS OF PRESTRESS FATIGUE SPECIMENS OF AN SAE 3140 STEEL

Heat Treatment	Prestress psi	Average Hardness	Range		No. of Specimens[3] Tested
			Maximum	Minimum	
Tough[1]	80,000	21.2	22	20	12
Brittle[2]	80,000	20.8	22	20	12
Tough	85,000	20.9	22	20	8
Brittle	85,000	20.5	22	20	8
Tough	90,000	20.8	22	20	9
Brittle	90,000	19.8	20	19	9

[1] Tough - Normalized 1-1/2 hour at 1650°F + 1 hour at 1600°F, O.Q. + 1 hour at 1250°F, W.Q.

[2] Brittle - Same as tough condition + 500 hours at 850°F, W.Q.

[3] Eight readings were taken on each specimen. These were corrected for the curvature of the specimen's surface.

the damage experiments. In general the hardnesses obtained are essentially uniform and agree well with the hardnesses obtained on impact bars with flat surfaces.

S-N Curves

The S-N curve established for the tough condition is shown in Figure 27 and for the temper-embrittled condition in Figure 28. The center curve in the figures is the best curve drawn through the antilog of the mean log of cycles to failure determined at the three stresses. The line on either side of the center curve represents the upper and lower limits of the 95 percent confidence interval for the mean. This interval is such that 95 percent of the time the mean of a set of samples tested at these stresses will fall within the indicated band.

A comparison of Figures 27 and 28 will show that the individual test points exhibit more scatter for the tough condition than for the temper-embrittled condition at the same stress level. Furthermore, for both types of specimens, the scatter in the test points increases as the stress approaches the endurance limit.

In Figures 27 and 28 are also plotted the test data obtained at stresses in the endurance limit region. Runouts were obtained on tough samples at 72,000 and 70,000 psi, while one specimen failed at 72,000 psi. In the temper-embrittled condition two specimens tested at 72,000 psi both failed. From the limited data no attempt can be made at establishing the endurance limit or its range. However, it must be less than 75,000 psi and possibly as low as 65,000 psi.[37] In any case the endurance limit for this steel appears to be more than 50 percent of the tensile strength.

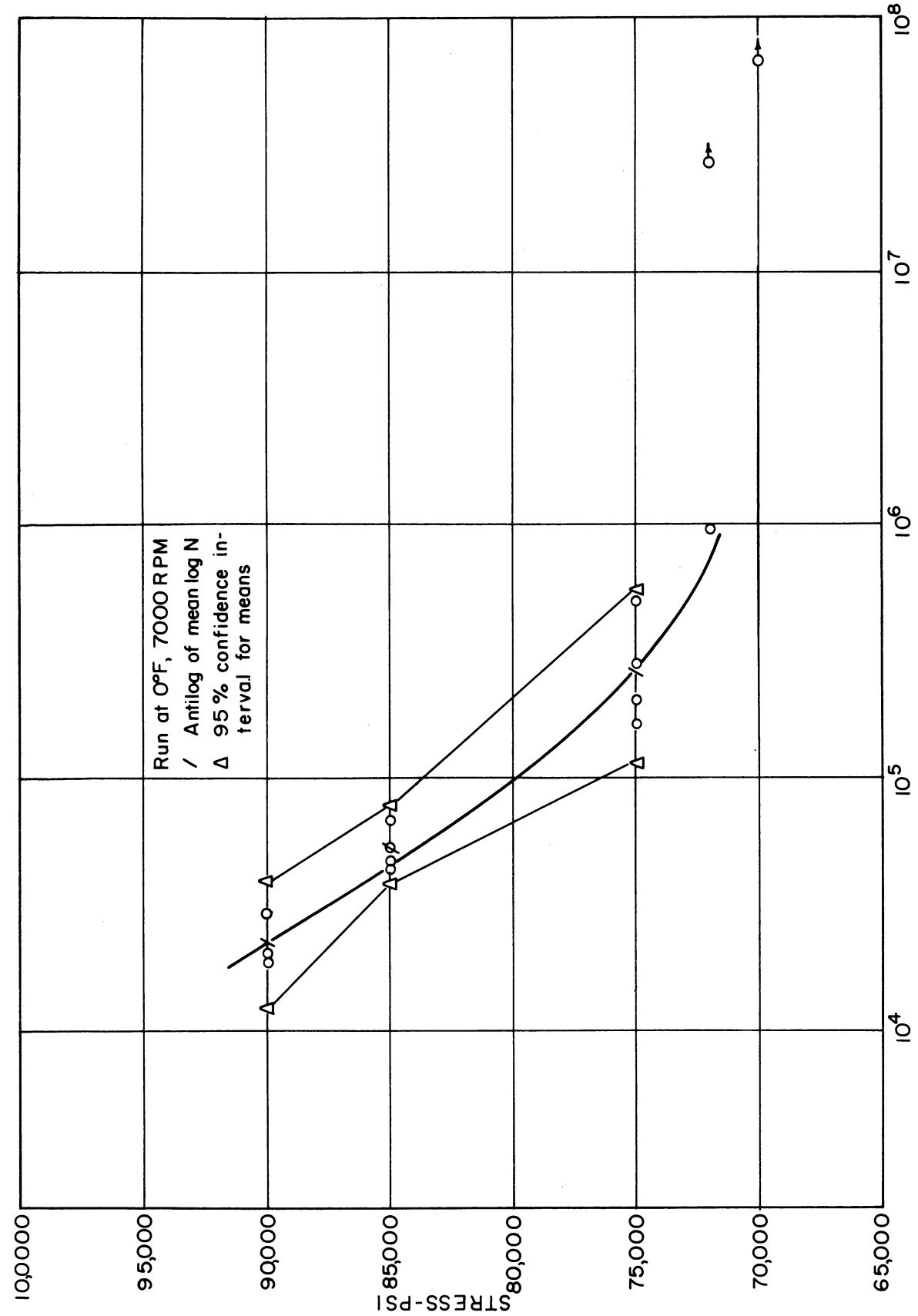


Figure 27. Fatigue Properties of an SAE 3140 Steel Tempered 1 Hour at 1250°F, Tough Condition.

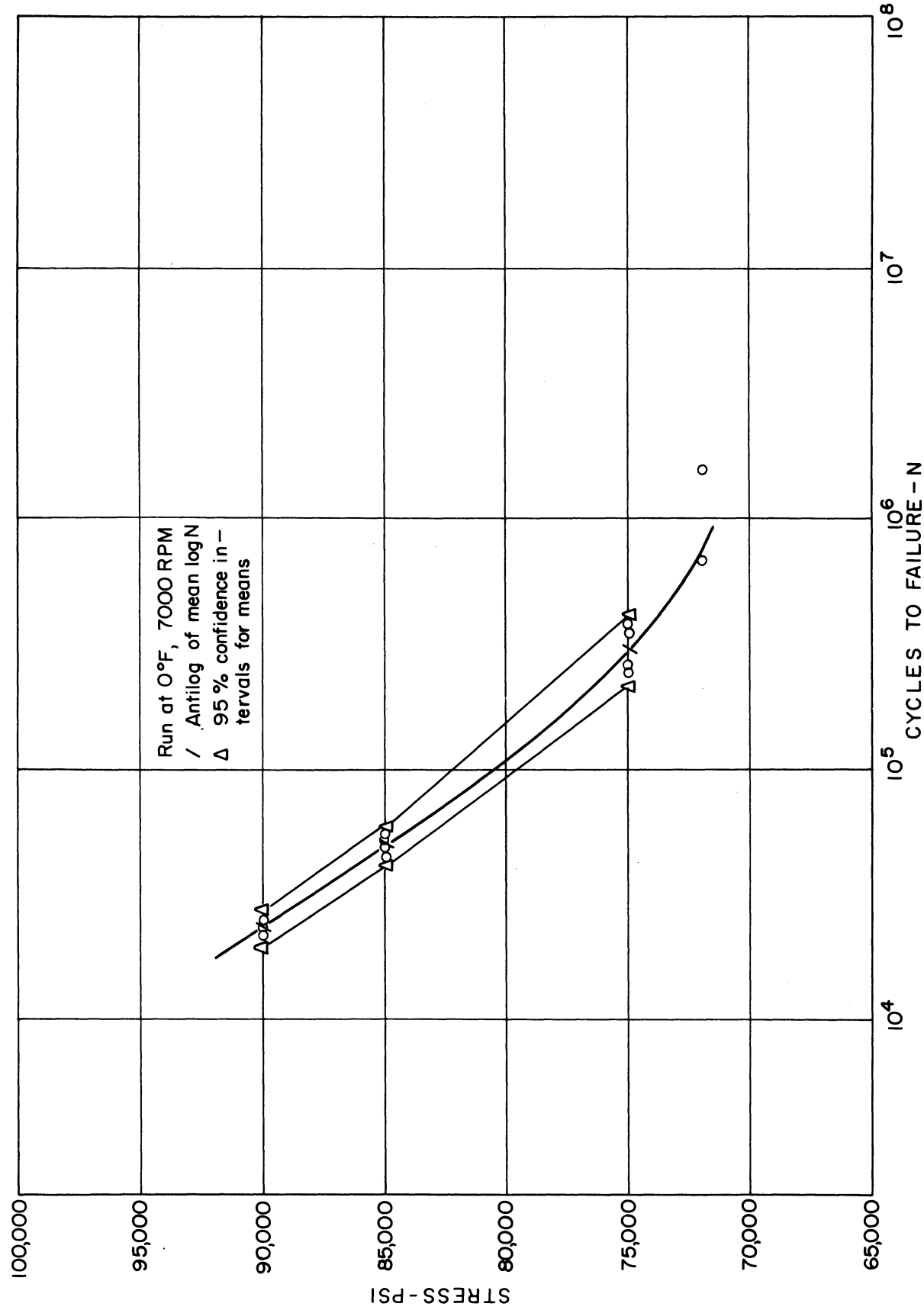


Figure 28. Fatigue Properties of an SAE 3140 Steel Temper-Embrittled 500 Hours at 850°F.

Statistical Comparison of S-N Curves

A tabulation of the fatigue data obtained in establishing the S-N curves for the tough and temper-embrittled structures is shown in Tables XIII and XIV, respectively. The cycles to failure at a given stress have been also transformed to the log of thousands of cycles to failure in these tables since statistical evaluation of fatigue data is based on a logarithmic normal distribution. Table XV presents some statistical parameters calculated from the S-N curve data obtained on tough specimens. A similar tabulation is given in Table XVI for the temper-embrittled specimens.

The calculation of the mean and variance of a set of samples is a familiar operation. However, when considering a small sample size, less than 30, it is necessary to make an adjustment to the variance calculated from the raw data. It has been shown by statisticians that the variance, as calculated from the raw data of a small sample size, is a biased estimate of the universe variance, $(\sigma')^2$. To obtain the universe variance, the variance of the sample, σ_x^2, must be multiplied by a factor of $\frac{N}{N-1}$ where N is the sample size. Furthermore, the square root of the universe variance, $(\sigma')^2$, is not an unbiased estimate of the standard deviation, σ'. It has been shown that an unbiased estimate of the standard deviation of small samples is actually equal to $\frac{\sigma_x}{C_2}$ where σ_x is the standard deviation calculated from the raw data and C_2 is a correction factor that is a function of sample size and may be found tabulated in standard statistical texts. Unbiased estimates of the standard deviation were used in computing the scatter band of the S-N curves. Unbiased estimates of the variance were needed in testing for real

TABLE XIII. S-N CURVE FATIGUE DATA FOR SAE 3140 STEEL IN TOUGH CONDITION[1] OBTAINED AT 0°F AND SPEED OF 7000 RPM

Stress-psi	Thousands of Cycles of Stress, N	Log_{10} N
70,000	68,000+	4.83251
72,000	960	2.98227
72,000	27,000+	4.43136
75,000	161	2.20683
75,000	202	2.30535
75,000	282	2.45025
75,000	503	2.70157
85,000	44	1.64345
85,000	47	1.67210
85,000	53	1.72428
85,000	68	1.83251
90,000	19	1.27875
90,000	20	1.30103
90,000	29	1.46240

[1] Austenitized 1 hour at 1600°F, oil quench plus 1 hour at 1250°F water quench.

+ Specimen did not fail.

TABLE XIV. S-N CURVE FATIGUE DATA FOR SAE 3140 STEEL IN TEMPER-EMBRITTLED CONDITION[1] OBTAINED AT 0°F AND SPEED OF 7000 RPM

Stress-psi	Thousands of Cycles of Stress, N	Log_{10} N
72,000	680	2.83251
72,000	1,570	3.19590
75,000	247	2.39270
75,000	257	2.40993
75,000	352	2.54654
75,000	375	2.57403
85,000	44	1.64345
85,000	49	1.69020
85,000	54	1.73239
85,000	55	1.74036
90,000	22	1.34242
90,000	23	1.36173
90,000	25	1.39794

[1] Austenitized 1 hour at 1600°F, oil quench + 1 hour at 1250°F, water quench + 500 hours at 850°F, water quench.

TABLE XV. STATISTICAL PARAMETERS OF LOG N, LOG OF THOUSANDS OF CYCLES TO FAILURE, FOR S-N CURVE OF FATIGUE SAMPLES OF AN SAE 3140 STEEL IN THE TOUGH CONDITION

Stress psi	Number of Tests	Log N = x								
		$\bar{X}$	Antilog $\bar{X}$	σ_x^2	$(\sigma_x')^2$	σ_x	σ_x'	$\bar{X} \pm 2\sigma_x'$	Antilog $\bar{X} \pm 2\sigma_x'$	95% Confidence Intervals for Mean, $\bar{X}$
75,000	4	2.416000	260.60	0.034679	0.046239	0.186224	0.233394	1.949212 to 2.882787	88.96 to 763.40	118.50 to 573.00
85,000	4	1.718085	52.25	0.005204	0.006938	0.072139	0.090412	1.537262 to 1.898908	34.45 to 79.23	38.51 to 70.90
90,000	3	1.347393	22.25	0.006696	0.008929	0.081834	0.113094	1.121207 to 1.573580	13.22 to 37.46	12.54 to 39.49

TABLE XVI. STATISTICAL PARAMETERS OF LOG N, LOG OF THOUSANDS OF CYCLES TO FAILURE, FOR S-N CURVE OF FATIGUE SAMPLES OF AN SAE 3140 STEEL IN THE TEMPER-EMBRITTLED CONDITION

Stress psi	Number of Tests	Log N = x							Antilog	95% Confidence Intervals for Mean, $\bar{X}$
		$\bar{X}$	Antilog $\bar{X}$	σ_x^2	$(\sigma_x')^2$	σ_x	σ_x'	$\bar{X} \pm 2\sigma_x'$	$\bar{X} \pm 2\sigma_x'$	
75,000	4	2.480800	302.60	0.006449	0.008599	0.080308	0.100649	2.279500 to 2.682099	190.30 to 480.90	215.40 to 425.00
85,000	4	1.701600	50.30	0.001490	0.001987	0.038606	0.048385	1.604831 to 1.798369	40.25 to 62.87	42.72 to 59.23
90,000	3	1.367360	23.30	0.000531	0.000707	0.023033	0.031831	1.303700 to 1.431025	20.13 to 26.98	19.83 to 27.38

differences in mean life for the S-N curves as well as in the evaluation of the damage experiments.

Two general types of statistical tests were employed in evaluating the fatigue data of the tough and temper-embrittled conditions. One type was concerned with establishing whether or not the variance of all the sample sets was the same. The other type of statistical test was concerned with establishing whether or not the means of two sample sets differed significantly.

The homogeneity of variance was tested by using the "F"-test,[36] which considers the variances between two sample sets only, and a special test by Fisher,[44] which compares the significance of an aggregate based on the product of the probabilities individually observed in the "F"-test.

When applying the "T"-test for determining a significant difference in sample means, it is necessary to consider whether or not the variance of the two samples is homogeneous. If the variance is homogeneous, then the standard "T"-test can be employed.[36] However, if the variance is found to be non-homogeneous, then another type of "T"-test must be used which is less efficient in distinguishing between means of different samples.[2,24]

In Table XVII the results of the "F"-tests on individual sample variances for the S-N curves are presented. Although the variances for the tough and temper-embrittled samples appear homogeneous when compared at a common stress level, it is not the case when tough and temper-embrittled samples are compared at different stress levels on the basis of the "F"-test. The existence of a non-homogeneity in variances is further supported by the results of the Fisher test on the significance of the product of the individual probabilities presented in Table XVIII.

TABLE XVII. STATISTICAL COMPARISON OF VARIANCES AND MEANS OBTAINED FROM S-N CURVE DATA ON AN SAE 3140 STEEL IN THE TOUGH AND TEMPER-EMBRITTLED CONDITIONS

Stress psi	Condition of Specimen	Variance Ratio S_1^2/S_2^2	"F"-Test, $f_{0.025}$	Significant Difference in Variance	Mean Life, Cycles to Failure	Calculated "t" value for Means	Critical "t"-values for "t" 0.025, $\sigma_1^2 = \sigma_2^2$	Critical "t"-values for "t" 0.025, $\sigma_1^2 \neq \sigma_2^2$	Significant Difference in Means
75,000	Tough Brittle	5.38	15.4	No	260,600 302,600	0.554	±2.447	±3.182	No
85,000	Tough Brittle	3.5	15.4	No	52,250 50,300	0.342	±2.447	±3.182	No
90,000	Tough Brittle	12.6	39.0	No	22,250 23,300	0.382	±2.776	±4.303	No
75,000 85,000	Tough Tough	5.18	15.4	No	-	-	-	-	-
75,000 90,000	Tough Brittle	65.4	39.2	Yes	-	-	-	-	-
75,000 90,000	Tough Tough	5.2	39.2	No	-	-	-	-	-
75,000 90,000	Brittle Brittle	12.08	39.2	No	-	-	-	-	-

TABLE XVIII. TEST FOR THE SIGNIFICANCE OF THE PRODUCT OF A NUMBER OF INDEPENDENT PROBABILITIES OBTAINED FROM THE VARIANCES OF THE S-N CURVE DATA

Stress psi	Variance Ratio S_1^2/S_2^2	Probability Level, P	$-\text{Log}_e$ P	Calculated Chi^2 Value	Degrees of Freedom
75,000	5.38	0.100	2.303	-	2
85,000	3.5	0.175	1.743	-	2
90,000	12.6	0.075	2.592	-	2
			Σ = 6.638	13.276	Σ = 6

The chi square value obtained from the data fell between 2.5 and 5 percent. This means that the probability of the aggregate of the three "F"-tests employed occurring by chance is only between 2.5 and 5 percent. Therefore, a significant difference in variance exists.

The question of whether or not the variances for the tough and temper-embrittled specimens are uniform is not unequivocally answered by statistical tests. However, the evidence appears to favor a non-uniform variance if one compares the scatter band of the individual test points of the two S-N curves shown in Figures 27 and 28.

Since the question of non-homogeneity or homogeneity of variance is not definitely established, both types of "T"-tests were used in comparing the means of tough and temper-embrittled samples. In Table XVII the critical "T"-values for a two tail test on a 5 percent significance level, which are computed on a basis of uniform and non-uniform variance, are compared to the calculated "T"-values for the means. Both tests show no significant difference between the means of the tough and temper-embrittled samples. One thing can be observed in the relationship of the calculated "T"-value and the critical "T"-values. If the "T"-test made on the basis of a uniform variance shows no significance, the "T"-test made on the basis of a non-uniform variance will certainly not show a significant difference either. However, if the "T"-test made on the basis of a uniform variance shows significance, the other "T"-test may or may not show significance, depending upon the degree of the non-homogeneity in variance.

Statistical Evaluation of Prestress Data

A compilation of all of the prestress data for the tough specimens is presented in Table XIX and for the temper-embrittled specimens

TABLE XIX. PRESTRESS FATIGUE DATA OBTAINED AT 0°F AND SPEED OF 7000 RPM FOR SAE 3140 STEEL IN THE TOUGH CONDITION. RUNOUT STRESS, 75,000 PSI

Prestress psi	Cycles at Prestress	Total Cycles to Failure x 1000	Log_{10}, Total Cycles to Failure, N
80,000	32,000	191	2.28103
		205	2.31175
		219	2.34044
		245	2.38917
80,000	54,000	164	2.21484
		383	2.58320
		555	2.74429
		871	2.94002
80,000	65,000	118	2.07188
		188	2.27416
		192	2.28330
		244	2.38739
85,000	5,000	195	2.29003
		201	2.30320
		243	2.38561
		364	2.56110
85,000	15,000	93	1.96848
		112	2.04922
		166	2.22011
		178	2.25042
90,000	1,000	194	2.28780
		231	2.36361
		359	2.55509
90,000	3,000	105	2.20119
		107	2.02938
		168	2.22531
90,000	7,000	66	1.81954
		95	1.97772
		100	2.00000

in Table XX. The number of cycles run at any prestress was determined by taking a fraction of the average of the means of the log of cycles to failure for the tough and temper-embrittled specimens taken together at a common stress level on the S-N curves.

A statistical comparison of the data obtained on tough and temper-embrittled specimens was made at each combination of prestress and cycles at the prestress to determine the relative behavior of these structures to damage by a short time overload in stress. In Table XXI the results of the "F"-tests based on individual variances between tough and temper-embrittled specimens are presented. On the basis of these individual "F"-tests, evidence of a non-homogeneous variance exists in the data obtained at a prestress of 80,000 psi; however, at 85,000 and 90,000 psi levels the variance appears to be homogeneous.

The Fisher test was applied to these "F"-tests also. The results are shown in Table XXII. The chi square value obtained by combining all of the prestress data fell between 2.5 and 5 percent. Again, as in the case of the S-N curve data, a non-homogeneity in variance is indicated.

Since the homogeneity of the variance was not clearly established, both types of "T"-tests were employed in comparing the means of the tough and temper-embrittled samples. In Table XXI the critical "T"-values for a two tail test on a 5 percent significance level, which were obtained on the basis of uniform and non-uniform variance, are compared to the calculated "T"-values for the means. The hypothesis that the means for the tough and temper-embrittled structures are equal cannot be accepted for the prestress conditions at:

1) 80,000 psi for 32,000 cycles

TABLE XX. PRESTRESS FATIGUE DATA OBTAINED AT 0°F AND SPEED OF 7000 RPM FOR SAE 3140 STEEL IN THE TEMPER-EMBRITTLED CONDITION. RUNOUT STRESS, 75,000 PSI

Prestress psi	Cycles at Prestress	Total Cycles to Failure x 1000	Log_{10}, Total Cycles to Failure, N
80,000	32,000	348	2.54158
		424	2.62737
		477	2.67852
		784	2.89432
80,000	54,000	248	2.39445
		361	2.55751
		555	2.74429
		585	2.76716
80,000	65,000	191	2.28103
		191	2.28103
		325	2.51188
		386	2.58659
85,000	5,000	103	2.01284
		110	2.04139
		147	2.16732
		193	2.28556
85,000	15,000	41	1.61278
		61	1.78533
		62	1.79239
		63	1.79934
90,000	1,000	126	2.10037
		147	2.16732
		165	2.21748
90,000	3,000	87	1.93952
		153	2.18469
		286	2.45637
90,000	7,000	86	1.93450
		86	1.93450
		120	2.07918

TABLE XXI. STATISTICAL COMPARISON OF VARIANCES AND MEANS OBTAINED FROM PRESTRESS DATA ON AN SAE 3140 STEEL IN THE TOUGH AND TEMPER-EMBRITTLED CONDITIONS. RUNOUT STRESS, 75,000 PSI

Prestress psi	Cycles at Prestress	Condition of Specimen	Variance Ratio S_1^2/S_2^2	"F"-Test $f_{0.025}$	Significant Difference in Variance	Mean Life, Cycles to Failure	Calculated "t" value for Means	Critical "t"-values for "t" $_{0.025}$, $\sigma_1^2 = \sigma_2^2$	Critical "t"-values for "t" $_{0.025}$, $\sigma_1^2 \neq \sigma_2^2$	Significant Difference in Means
80,000	32,000	Tough Brittle	10.60	15.4	No	213,800 484,200	4.520	±2.447	±3.182	Yes
80,000	54,000	Tough Brittle	3.08	15.4	No	416,900 412,100	0.026	±2.447	±3.182	No
80,000	65,000	Tough Brittle	1.43	15.4	No	179,500 260,000	1.560	±2.447	±3.182	No
80,000	32,000 54,000	Tough Brittle	44.80	15.4	Yes	213,800 412,100	-	-	-	-
85,000	5,000	Tough Brittle	1.00	15.4	No	242,100 133,700	2.830	±2.447	±3.182	Yes-No
85,000	15,000	Tough Brittle	2.26	15.4	No	132,400 55,850	4.650	±2.447	±3.182	Yes
90,000	1,000	Tough Brittle	5.55	39.0	No	252,300 144,900	2.780	±2.776	±4.303	Yes-No
90,000	3,000	Tough Brittle	5.02	39.0	No	123,300 156,000	0.621	±2.776	±4.303	No
90,000	7,000	Tough Brittle	1.39	39.0	No	85,510 95,940	0.675	±2.776	±4.303	No
90,000	1,000 3,000	Brittle Brittle	19.4	39.0	No	144,900 156,000	-	-	-	-

TABLE XXII. TEST FOR THE SIGNIFICANCE OF THE PRODUCT OF A NUMBER OF INDEPENDENT PROBABILITIES OBTAINED FROM THE VARIANCES OF THE PRESTRESS DATA

Prestress psi	Cycles at Prestress	Variance Ratio S_1^2/S_2^2	Probability Level, P	$-\text{Log}_e P$	Calculated Chi^2 Value	Degrees of Freedom
80,000	32,000	10.6	0.045	3.107		2
	54,000	3.08	0.180	1.715		2
	65,000	1.43	0.300	1.204		2
85,000	5,000	1.00	0.500	0.693		2
	15,000	2.26	0.230	1.470		2
90,000	1,000	5.55	0.095	2.355		2
	3,000	5.02	0.105	2.255		2
	7,000	1.39	0.310	1.171		2
				$\Sigma = 13.970$	27.940	$\Sigma = 16$

2) Possibly at 85,000 psi for 5,000 cycles

3) 85,000 psi for 15,000 cycles

4) Possibly at 90,000 psi for 1,000 cycles

The "T"-test as applied above indicated a difference in response of tough and brittle samples to a certain prestress condition at each prestress level employed. However, the question of whether damage was done or not is not indicated by these tests.

To test whether or not damage was inflicted on the fatigue specimens by any prestress it was necessary to compare the means established for a given prestress condition with the mean of a set of samples run to failure at 75,000 psi without prior prestressing. A comparison of the means of tough and temper-embrittled specimens run at all of the prestress conditions with the means established at 75,000 psi for the S-N curves is presented in Table XXIII. Again, "T"-tests were made on the basis of a uniform and non-uniform variance.

At 80,000 psi prestress no damage was observed for any stress-cycle combination. Although the "T"-tests indicated no improvement in life caused by prestressing at 80,000 psi either, the response to prestressing of both tough and temper-embrittled samples, especially the latter, appears to be different than at other prestress levels. Improvements in fatigue life were obtained as indicated in Table XXIII but the "T"-tests did not indicate these improvements as being significant. The improvements that were observed seemed to be greater for the temper-embrittled specimens than for the tough specimens.

The improvement in fatigue life by periods of overstress near the endurance limit has been reported by other investigators.[97,131] It is possible that by prestressing at 80,000 psi an effect similar to

TABLE XXIII. STATISTICAL COMPARISON OF VARIANCES AND MEANS BETWEEN 80,000, 85,000, AND 90,000 PSI PRESTRESS AND 75,000 PSI S-N CURVE FATIGUE DATA ON AN SAE 3140 STEEL IN THE TOUGH AND TEMPER-EMBRITTLED CONDITIONS

Stress or Prestress psi	Cycles at Prestress	Condition of Specimen	Variance Ratio s_1^2/s_2^2	"F"-Test $f_{0.025}$	Significant Difference in Variance	Mean Life, Cycles to Failure	Calculated "t" value for Means	Critical "t"-values for "t" $_{0.025}$, $\sigma_1^2 = \sigma_2^2$	Critical "t"-values for "t" $_{0.025}$, $\sigma_1^2 \neq \sigma_2^2$	Significant Difference in Means
75,000 80,000	- 32,000	Tough Tough	21.8	15.4	Yes	260,600 213,800	0.777	±2.447	±3.182	No
75,000 80,000	- 32,000	Brittle Brittle	2.63	15.4	No	302,600 484,200	2.320	±2.447	±3.182	No
75,000 80,000	- 54,000	Tough Tough	2.04	15.4	No	260,600 416,900	1.090	±2.447	±3.182	No
75,000 80,000	- 54,000	Brittle Brittle	3.56	15.4	No	302,600 412,100	1.370	±2.447	±3.182	No
75,000 80,000	- 65,000	Tough Tough	2.65	15.4	No	260,600 179,500	1.290	±2.447	±3.182	No
75,000 80,000	- 65,000	Brittle Brittle	2.90	15.4	No	302,600 260,000	0.720	±2.447	±3.182	No
75,000 85,000	- 5,000	Tough Tough	2.97	15.4	No	260,600 242,100	0.250	±2.447	±3.182	No
75,000 85,000	- 5,000	Brittle Brittle	1.83	15.4	No	302,600 133,700	4.550	±2.447	±3.182	Yes
75,000 85,000	- 15,000	Tough Tough	2.52	15.4	No	260,600 132,400	2.310	±2.447	±3.182	No
75,000 85,000	- 15,000	Brittle Brittle	1.06	15.4	No	302,600 55,850	11.300	±2.447	±3.182	Yes
75,000 90,000	- 1,000	Tough Tough	2.44	39.2	No	260,600 252,300	0.104	±2.571	±3.580	No
75,000 90,000	- 1,000	Brittle Brittle	2.49	39.2	No	302,600 144,900	5.550	±2.571	±3.570	Yes
75,000 90,000	- 3,000	Tough Tough	3.46	39.2	No	260,600 123,300	2.560	±2.571	±3.500	No
75,000 90,000	- 3,000	Brittle Brittle	7.77	16	No	302,600 156,000	1.840	±2.571	±4.190	No
75,000 90,000	- 7,000	Tough Tough	4.78	39.2	No	260,600 85,510	3.990	±2.571	±3.44	Yes
75,000 90,000	- 7,000	Brittle Brittle	1.23	39.2	No	302,600 95,940	7.45	±2.571	±3.77	Yes

coaxing is brought about which strengthens the metal. The tendency for the temper-embrittled specimens to be more prone to this apparent coaxing effect can be attributed to the presence of a more favorable stress distribution in these structures. The tensile test data presented in an earlier section show that temper-embrittled specimens exhibit a different characteristic yield point than corresponding tough specimens. This difference in the yield point phenomenon may explain the greater tendency for coaxing in the temper-embrittled samples. Corten[27] has reported that materials that are more subject to strain aging are also more subject to coaxing.

At 85,000 psi prestress both types of "T"-tests indicated that the temper-embrittled specimens were damaged by 5,000 and 15,000 cycles of prestress, but the tough samples were not damaged.

At 90,000 psi prestress the temper-embrittled specimens were damaged by 1,000 and 7,000 cycles of prestress and the tough specimens by 7,000 cycles of prestress. No damage was indicated by the "T"-tests for the tough specimens for 1,000 and 3,000 cycles of prestress and for the temper-embrittled specimens for 3,000 cycles of prestress.

In considering the overall damage data obtained in this investigation, it appears that temper-embrittlement produces a structure which is more prone to damage by periods of overstress that are relatively high above the runout stress of 75,000 psi than are the tough structures. With periods of overstress just above the runout stress a coaxing effect is indicated for both tough and temper-embrittled specimens with the latter being more prone to the beneficial effects of coaxing.

The damage data also indicate that the amount of damage produced by a given prestress condition is governed by the number of cycles at the prestress as well as the prestress level. For the embrittled samples, at 80,000 psi prestress and 65,000 cycles no significant damage was observed; but at 85,000 psi prestress damage occurred with only 5,000 cycles, and at 90,000 psi prestress damage occurred at a still smaller number, 1,000 cycles of prestress. For the tough samples no damage was observed except for 7,000 cycles of prestress at 90,000 psi.

The different response to prestress cycles for tough and embrittled structures can be attributed to the different residual stress patterns in the two structures. A temper-embrittled structure is believed to have a stress concentration across the prior austenite grain boundaries, whereas the tough structure does not. At prestress levels considerably above the runout stress no coaxing for either tough or temper-embrittled structures is expected, since the coaxing phenomenon is only observed when the initial stress is close to the runout stress employed. Apparently, at the higher prestress levels the residual stress pattern in the embrittled structure in conjunction with the initial prestress promotes a more rapid deterioration of the metal at a lower stress level than in the case of the tough structure.

In the straight runout fatigue tests, used for establishing the S-N curves, both structures were damaged to the same extent as the test proceeded. The residual stress patterns in the two structures did not influence the fatigue properties determined at a single stress. The residual stress patterns were only of consequence when a second, lower runout stress was employed.

MECHANISM OF TEMPER-EMBRITTLEMENT

An examination of the literature on the phenomenon of temper-embrittlement will reveal that although many conflicting experimental evidence has been reported, a number of well established facts emerge clear. These observed facts regarding the phenomenon are:

1) Loss in impact properties while other mechanical properties are virtually unaffected.

2) Increase in grain boundary attack observed with special etches as the degree of embrittlement increases.

3) Intergranular brittle failure of an embrittled material along prior austenite grain boundaries.

4) The large number of alloying elements that seem to promote temper-embrittlement.

5) The reversibility of the embrittling reaction as a function of time as well as temperature.

6) The progressive increase in transition temperature to a maximum value with time.

7) The lack of an identification of any precipitated phase that is associated with the embrittling reaction.

Any theory that will fully explain the complex phenomenon of temper-embrittlement must embrace all of the observed facts listed above.

A mechanism for temper-embrittlement which appears to satisfy the listed observations has been proposed by Bush.[22] His idea is as follows:

> "To explain the entire embrittlement phenomenon it is necessary to assume a dual rate reaction. This reaction is temperature dependent, as is customary in rate reactions. It is postulated that the diffusion of interstitial constituents occurs at a rapid rate, while substitutional constituents diffuse at a lower rate. The constituents diffuse from within the lattices to areas of misfit occurring at the grain boundaries. These areas of misfit serve two purposes; the first is to

> act as sink holes to entrap the particles diffusing from the lattice; the second is to act as centers for dislocations. These dislocations serve to lock in the components, and areas of pronounced localized strain occur. When sufficient strain is developed the dislocations tend to diffuse from these centers, releasing the constituent and causing a reversal in the transition temperature."

The proposal that the interstitial elements are responsible for early embrittlement is in harmony with known diffusion rates of carbon and nitrogen. An analysis of the transition shifts reported by Bush[22] using a reaction rate type plot of log embrittling rate versus reciprocal temperature leads to an activation energy for temper-embrittlement of about 39,000 calories per mole. Since the activation energy for carbon diffusion in alpha iron is of the order of 20,000 calories per mole, whereas for substitutional elements the value is closer to 80,000 calories per mole, it substantiates the claim that carbon is contributing to the early stages of temper-embrittlement as outlined by the theory.

Further evidence of carbon contributing to the embrittling reaction has been noted in studies on the effect of alloying elements on temper-embrittlement as was discussed in the Literature Survey. Evidence of a contribution to the embrittling reaction by substitutional elements, P, Cr, Mn, etc. is well founded.

Although the theory outlined above does not recognize the existence of a precipitated phase or film existing about the prior austenite grain boundaries, it does postulate the existence of segregation of interstitial and substitutional elements to these regions. It is pertinent to inquire the amount of this segregation which is necessary to affect the fracture behavior of a susceptible steel. A clue as to

how much carbon would be necessary can be found in the work of Cottrell.[28] In the application of dislocation theory to the yield point phenomenon observed in mild steels, Cottrell reports that a concentration of 10^{-3} to 10^{-2} weight percent carbon is necessary to lock a dislocation into position, even though only about 10^{-7} weight percent of carbon is dissolved in ferrite at room temperature. The build up of from 10,000 to 100,000 times as much carbon around a dislocation as exists in the ferrite matrix is brought about by diffusion of carbon from localized supersaturated ferrite as well as from existing carbides.

In the mechanism of temper-embrittlement as put forth by Bush,[22] the concept of carbon diffusing to prior austenite grain boundaries, locking dislocations into position, is paramount. If it is assumed that an equivalent amount of carbon is necessary to lock dislocations in position in both the yield point and temper-embrittlement phenomena, then at least 10^{-3} weight percent of carbon is associated with the prior austenite grain boundaries of a temper-embrittled steel, and this concentration is from 1,000 to 10,000 times as much carbon as is dissolved in the ferrite matrix at the embrittling temperature of 850°F.

It is believed that a concentration difference of at least 10 to 1 was distinguishable using this autoradiographic technique. This sensitivity of the autoradiographic technique was established by Yukawa[148] using radioactive nickel-63. Since the energies of the beta particles emitted by carbon-14 and nickel-63 are of the same order of magnitude, it is believed that the sensitivity results obtained with nickel-63 are applicable to the carbon-14 work done in this investigation.

However, the results of the autoradiographic studies in this investigation do not indicate the presence of carbon segregated to the

prior austenite boundaries in any form. Therefore, it appears that the idea of carbon segregating to the prior austenite grain boundaries, locking dislocations into position and thereby building up a strained region in this vicinity, accounting for intergranular brittle fracture, is open to question. Experimental evidence does not support the carbon segregation hypothesis.

A second hypothesis, which is similar in nature to that just discussed, is the one proposed by Woodfine.[147] In this hypothesis, substantial elements are believed to diffuse to the prior austenite grain boundaries on a submicroscopic scale from the ferrite matrix at the embrittling temperature as well as from the austenite matrix existing during the initial hardening of the steel. The result of this segregation is to lower the cohesion between grains across the prior austenite grain boundaries which results in brittle fracture. To explain the reversal that occurs in the transition temperature with embrittling time, which has been observed by Woodfine[147] and Bush and Siebert,[22] he postulates a diffusion of chromium from the matrix into the carbide particles, thereby reducing the chromium content of the ferrite in the immediate vicinity of existing carbides. This reaction is supposed to result in an increase in the ferrite cleavage strength, thus producing a partial recovery from brittle fracture.

Woodfine's theory is not consistent with the fact that the activation energy for temper-embrittlement is closer to that of an interstitial element and not of any of the substitutional elements that contribute to temper-embrittlement. Nor is it easy to see how a reaction occurring in the austenite can influence temper-embrittlement when embrittlement can be completely removed by a retempering operation just below the

critical temperature where the material is completely ferritic. Furthermore, carbon additions to a susceptible steel increase further the degree of embrittlement attainable by a given heat treatment. Further evidence against the theory of substitutional elements diffusing to prior austenite grain boundaries leading to embrittlement is the fact that plain nickel steels are not susceptible to temper-embrittlement. It therefore appears that this theory is not fully adequate to explain the complexity of the temper-embrittlement phenomenon.

The fact that the prior austenite grain boundaries are preferentially attacked in a brittle structure and that this attack progressively increases with increasing embrittlement suggests two alternatives. Either the chemical composition of the boundary regions is altered so that they are selectively etched or that a severe stress concentration is present in these regions which leads to a stress-corrosion type of attack. Since in the preceding discussion the idea of segregation to the prior austenite boundaries of interstitial and substitutional elements was shown to be unsupported by existing experimental evidence, the strain mechanism for temper-embrittlement needs to be examined as a possible explanation.

If it is postulated that during the embrittling process a modification of carbides in the matrix comes about through a reaction between the existing carbides and the alloying elements dissolved in the matrix, then a strain could be developed across the prior austenite grain boundaries by virtue of a contraction in each individual ferrite grain. Maloof[109] has reported such a change occurring in carbide composition brought about by the temper-embrittlement reaction. It is known that the lattice parameter of a temper-embrittled steel is smaller

than a corresponding tough steel. This means that the alloying elements dissolved in the matrix have come out of solution from the ferrite. Since these elements are not believed to diffuse to the prior austenite grain boundaries, they must diffuse somewhere and the only remaining positions that they can occupy are associated with the existing minor phases. In this manner carbon could still be responsible for the initial rapid rate of embrittlement and the substitutional elements with the later stages of embrittlement. The reversal in transition temperature with time at the embrittling temperature could be explained on the basis of a partial balance between the strain developed across the prior austenite boundaries and the increase in cleavage strength of the ferrite as dissolved alloying elements are removed.

The return of toughness by reheating a temper-embrittled structure below the lower critical temperature could be explained on the basis of relief of strain at the higher temperature and a re-solution of some substitutional alloying elements into the ferrite matrix. The fact that plain carbon steels are not generally considered to be susceptible to temper-embrittlement supports the theory of carbide modification by alloying elements as outlined here.

As yet no direct experimental evidence is available in support of this hypothesis. An attempt to analyze the extracted carbides of an embrittled steel was made in this investigation in an effort to substantiate this theory but the limited results were not reliable. It is suggested that work involving radioactive isotopes of the substitutional elements as well as a program involving the identification of the carbides in an embrittled steel be conducted to gain a better understanding of the phenomenon of temper-embrittlement.

CONCLUSIONS

An analysis of the results of this investigation has led to the following conclusions:

1. Autoradiographic studies using carbon-14 and electron microscope observations of embrittled structures of a 3140 steel do not reveal the presence of carbon atoms or carbides segregated to the prior austenite grain boundaries.

2. No continuous film of any kind was observed in the prior austenite grain boundaries of a temper-embrittled structure in this heat of 3140 steel.

3. Carbides are present in the prior austenite grain boundaries of a temper-embrittled structure but it is unlikely that in these sites their presence has any effect on temper-embrittlement.

4. The prior austenite grain boundaries as revealed by an ethereal picric etch are actual grooves between the grains and not a staining effect of the etchant.

5. X-ray back reflection lines for embrittled structures are less diffuse than corresponding tough structures.

6. Temper-embrittlement is brought about by a strain developed across the prior austenite grain boundaries at the embrittling temperature as a result of contraction of the ferrite lattice when dissolved alloying elements are removed from solution in the ferrite by a reaction with the existing minor phases.

7. Within the scope of this investigation the fatigue properties of a 3140 steel in the finite life region of the S-N curve were not found to be affected by temper-embrittlement.

8. Temper-embrittled structures are more susceptible to damage by periods of fatigue overstress than are tough structures.

9. The initiation of damage by a prestress above the runout stress is a function of cycles at the prestress as well as the prestress level.

10. The scatter in fatigue life at a given stress is greater for the tough specimens than for the temper-embrittled specimens.

11. The tough and temper-embrittled structures of a 3140 steel are both subject to a strengthening action by periods of overstress at 80,000 psi followed by a runout stress of 75,000 psi.

12. Temper-embrittled structures are more prone to a strengthening action by a prestress of 80,000 psi followed by a runout stress of 75,000 psi than are tough structures.

13. Temper-embrittled structures exhibit a greater drop in the beam at the yield point in the tensile test than tough structures.

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