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TEMPER-EMBRITTLEMENT STUDIES

R. F. KNIGHT

PHYSICAL METALLURGY DIVISION

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TEMPER-EMBRITTLEMENT STUDIES

by

R. F. Knight*

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ABSTRACT

The extensive literature relating to temper-embrittlement phenomena is reviewed critically, and various aspects are examined, particularly as related to the effects of progressive increments of molybdenum in a series of low-alloy steels.

The experimental results show over-all agreement with the trends reported in general terms in the literature. Attempts are made to define effects quantitatively for the particular experimental conditions involved, particularly concerning the quantity of molybdenum required to achieve the optimum improvements in impact transition temperature and in the degree of susceptibility to temper embrittlement.

The distinctive grain-boundary etching effect is shown to be related to the condition of heat treatment, and not to the degree of susceptibility to temper embrittlement of the sample being examined.

Various effects of molybdenum on the room-temperature and reduced-temperature tensile behaviour of the experimental steels are presented.

Difficulties relating to the selection of criteria to define the susceptibility to temper embrittlement are discussed, and a new approach is suggested.

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Direction des mines

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ÉTUDE DE FRAGILISATION AU REVENU

par

R. F. Knight*

RÉSUMÉ

L'auteur fait l'étude critique des nombreux ouvrages traitant des phénomènes de fragilisation au revenu et en examine divers aspects, en particulier l'effet produit par l'addition progressive de molybdène à des aciers faiblement alliés.

Les résultats des expériences s'accordent dans l'ensemble avec les conclusions énoncées en termes généraux dans les ouvrages consultés. L'auteur s'est efforcé de traduire quantitativement ces résultats, dans les conditions expérimentales qui prévalaient au cours des essais, surtout en ce qui a trait à la quantité de molybdène nécessaire pour améliorer les caractéristiques de la température de transition (dans l'essai de résilience) et du degré de susceptibilité à la fragilisation au revenu.

Les essais permettent de conclure que l'effet d'attaque aux joints des grains est dû au mode de traitement thermique et non pas au degré de susceptibilité de l'échantillon étudié à la fragilisation au revenu.

L'auteur décrit certains effets de l'addition de molybdène aux aciers étudiés sur les caractéristiques mécaniques, à la température ambiante et à des températures plus basses.

Il relate également les difficultés éprouvées dans la sélection de critères pour définir la susceptibilité à la fragilisation au revenu et il suggère une nouvelle méthode d'aborder le problème.

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1. REVIEW OF THE LITERATURE

General

Temper brittleness has been defined as "brittleness that results when certain steels are held within, or are cooled slowly through, a certain range of temperature below the transformation range. The brittleness is revealed by notched-bar impact tests at room temperature or at lower temperatures" (1).

Two types of embrittlement which occur on tempering these steels are: upper-nose (irreversible) embrittlement, and lower-nose (reversible) embrittlement. It is generally held that both should be considered as temper embrittlement processes, but the causes and many of the effects of the two phenomena are different. Most of the interest has been centred on reversible temper brittleness and, in general, upper-nose embrittlement is covered in this review only as required to make necessary distinctions.

Excellent reviews on the subject have been published by Woodfine (2) and Goodchild (3), and more recently by Singh (4). This review will summarize the main views of the above authors, and incorporate some of the findings of other investigators as required.

Evaluation of Susceptibility to Temper Brittleness

In the early literature, the usual method used to show the susceptibility of a steel to temper embrittlement was to determine the "susceptibility ratio", S, as given by

$$S = Iu/Ie,$$

where Iu was the room-temperature impact energy absorbed by an impact specimen in the tough (unembrittled) condition of heat treatment, and Ie was the room-temperature impact strength of the embrittled sample.

Vidal and Jolivet (5) showed in 1944 that the use of room-temperature impact properties is not a satisfactory method to reveal temper brittleness. Many steels which would be rated as "not susceptible" by the room-temperature susceptibility ratio, would in fact be shown to be markedly susceptible at lower testing temperatures. Since that time, most investigators have obtained full impact-energy versus testing-temperature curves for both conditions of heat treatment (embrittled and unembrittled). The extent to which the curve is displaced to higher testing temperatures by the embrittling treatment is taken as indicative of the degree of susceptibility to temper embrittlement. In order to simplify the presentation of data, investigators measure the temperature of transition from ductile to brittle fracture mode, and report the increase in the transition temperature caused by the embrittling treatment.

Sadovskii (6) presented the viewpoint held until very recently by most Russian workers when he stated that there is no sound foundation for quantitative comparative evaluation of susceptibilities from the difference between the transition temperatures for tough and embrittled steels. Most of the Russian work has been based on a comparison of the impact-energy levels of tough and embrittled steels, with most of the work being done exclusively at ambient temperature. Mikhailov-Mikheev (7) proposed that even if the transition temperature of a steel is increased by the embrittlement treatment, the steel should not be considered susceptible to temper embrittlement if for both conditions the transition from ductile to brittle fracture occurs below some accepted temperature (he chose 32°F). The earlier viewpoint of the Russians emphasizes the lowering of the impact strength as a more practical manifestation of the embrittling effect, and their conclusions were, for the most part, based solely on impact values. Unfortunately, selecting one or two testing temperatures will not guarantee that the most severe degree of embrittlement will be detected. Recent Russian papers do refer to the shift in the transition temperature as the measurement of embrittlement.

The use of the impact transition temperature is also complicated by several factors. It does not take into account the actual degree of impairment of impact strength. Susceptibility ratings based solely on differences in transition temperature make no distinction of the actual temperatures involved. Furthermore, there is no universal agreement on the proper choice of transition-temperature criterion.

This subject of susceptibility is developed further in a later section of this report.

Choice of Transition Temperature

One of the problems involved in the use of transition-temperature measurements to simplify the presentation of data obtained by impact testing is concerned with the selection of the criterion to define the transition temperature. Disagreement exists on whether the value should be based on the impact-energy curve, the appearance of the fracture, or a combination of both. The most common methods which have been used can be summarized as follows:

Based on fracture appearance:

a - the lowest temperature for which the fracture appears to be 100 per cent fibrous, i.e., the point of first appearance of brittle fracture.

b - the temperature corresponding to a given percentage of fibrous fracture, usually 50 per cent.

Based on impact-energy curves:

a - the testing temperature at which the fracture energy is reduced to some arbitrary value (15, 25, 30 and 50 ft-lb levels are common).

b - the temperature corresponding to a given percentage, usually 50 per cent, of the maximum energy absorption (upper-shelf of impact energy).

c - the temperature at the point of inflection in the transitional range.

d - the temperature at which the impact strength is at a given level, usually midway, between the impact strengths at high and low testing temperatures (upper-shelf and lower-shelf) (8).

Based on a combination of both concepts:

a - the temperature corresponding to a given amount, usually 50 or 75 per cent, of the energy absorption at the lowest temperature for 100 per cent fibrous fracture.

Many of the foregoing methods give comparable results but the choice of method seems to depend to a great extent on the type of steel being tested. It would be desirable to try to standardize on one criterion so that the results of independent investigations might more realistically be compared.

Vidal and Popoff (9) reported an attempt to define a transition temperature in a low-temperature tensile test. While the value of the transition temperature they obtained was quite reproducible, it would appear to be dependent on the static load level applied. In any case, no attempt was made to differentiate between tough and embrittled conditions of steels.

Embrittlement Temperature Ranges

Woodfine stated that temper embrittlement takes place in nickel-chromium steels up to the Ae_1 temperature. Jaffe and Buffum (10) examined the isothermal embrittlement of SAE 3140 steel and found two separate regions where isothermal embrittlement develops rapidly. The lower-nose or reversible embrittlement range extended downward from around 600°C (1112°F), with the most rapid embrittlement between 550 and 490°C (1022 and 914°F), and continued to low temperatures where embrittlement became very slow. The upper-nose or irreversible brittleness range started at the Ae_1 temperature where embrittlement was most rapid, and continued downward, overlapping the lower range, with the rate of upper-nose embrittlement becoming very small as the temperature was lowered. The maximum degree of lower-nose embrittlement attainable on isothermal holding increased as the temperature was lowered, at least to 500°C (932°F).

To obtain these results, Jaffe and Buffum made use of the isoembrittlement diagram, which was well described by Proebstle (11). The ordinate of this diagram is the tempering temperature, and the abscissa is the logarithm of the time at temperature. Curves are plotted showing combinations of tempering temperature and time that result in the same transition temperature, or similar amounts of embrittlement. Each curve depicts a given increase in transition temperature from a base transition temperature. Proebstle showed that the most suitable base transition temperature, the lowest transition temperature attainable, is found by means of a short, high-temperature preliminary temper followed by fast cooling.

The lower-nose embrittlement can be removed by heating above 600°C (1112°F) and fast cooling through the embrittlement range by quenching to a temperature below 350°C (662°F). High-temperature embrittlement occurs up to the lower critical temperature and cannot be reversed by any treatment short of re-austenitizing. Jaffe and co-workers (10, 12, 13) cite evidence that embrittlement occurring during slow cooling proceeds at a faster rate than does embrittlement during isothermal treatment. Cooling an SAE 3140 steel from 675 to 325°C (1247 to 617°F) over a 20-hour period gave an embrittlement comparable to that resulting from holding at the temperature indicated by the nose of the isoembrittlement diagram for a period of 48 hours. It is apparent that the embrittlement occurring during slow cooling is associated primarily with lower-nose embrittlement, and in the case of the SAE 3140 steel under discussion most of the embrittlement took place above 498°C (930°F).

Effect of Variation of Heat Treatment

Woodfine (2) pointed out that for investigations of temper embrittlement the steels should be in the quenched-and-tempered condition before the embrittling treatment is applied. If they are embrittled directly from the quenched condition, it is practically impossible to sort out the separate effects of temper embrittlement and the normal tempering effects. For steels to be embrittled by the slow-cooling method, it was recommended that both quenched samples should be tempered at 650°C (1200°F), with one furnace-cooled and the other water-cooled. Then both should be re-tempered at 650°C (1200°F), followed by water cooling of the first sample to give a retoughened material, and furnace cooling of the other sample to embrittle it. For isoembrittlement treatments, the preliminary high-temperature temper should be just long enough to minimize hardness and microstructural differences between the tough and embrittled samples, so as to minimize the occurrence of upper-nose embrittlement.

Woodfine noted that a tempered martensitic structure is the most susceptible for the development of temper brittleness, but that it can be produced in unhardened steels. Buffum and Jaffe (14) found that, for a fixed embrittlement treatment, less brittleness developed in pearlitic and proeutectoid ferrite than in tempered martensite of the same composition.

Goodchild (3) illustrated the effect of sensitizing time for a 0.25% C, 0.32% Si, 0.30% Mn, 1.38% Cr steel, oil-quenched from 825°C (1520°F) and pretempered for 2 hours at 650°C (1200°F). He showed that the greater part of the displacement in the impact-energy curve, due to the embrittling treatment at 525°C (975°F), had occurred after a period of 3 to 10 hours.

Woodfine (15) found that, with constant grain size, neither the austenitizing time nor the quenching temperature affects the subsequent development of temper brittleness. Jaffe, Carr and Buffum (16) also found that lowering the quenching temperature has little effect on the transition temperature. Sadovskii (6), in referring to this point, cited unpublished work in which some mitigation of temper embrittlement was apparent due to dropping the temperature from 1200°C to 900°C (2190°F to 1650°F) and holding at that temperature prior to quenching.

Some confusion as to "recovery" and "stabilization" effects due to extended tempering at high temperatures was apparent. Mikhailov-Mikheev (7) reported that holding above 600°C (1110°F) did not embrittle the steel he was testing, and claimed that such holding renders steels insusceptible to temper embrittlement. However, Prosvirin and Kvashnina (17) found a drop in the impact strength of the same steel at sub-zero temperatures. They stated, as did Smirnov and Sadovskii (18), that such "stabilization" is due only to softening. Prosvirin and Kvashnina also reported that when a quenched, or quenched-and-tempered, steel is tempered for a long period in the embrittlement range, the room-temperature impact strength initially decreases, and subsequently rises to values about normal for quenched-and-tempered steels. Unfortunately they published no hardness data. Woodfine (15) felt that the softening occurring in steels tempered for long periods at high temperatures cannot completely explain the observed decrease in embrittlement. He based this view on the supposition that a given decrease in hardness should result in a constant decrease in transition temperature, and to him the decrease in embrittlement observed appeared to be greater than would be expected from the drop in hardness. It should be pointed out, while considering the subject of stabilization, that Jaffe and Buffum (13) found that the temper brittleness of SAE 3140 steel was not reduced by increasing the holding time at any temperature below the Ae_1 temperature, at least up to 240 hours.

Woodfine (2) stated that increased austenitic grain-size increases the rate and degree of temper embrittlement. Jaffe, Buffum and Carr (12) stated that there are apparent exceptions, notably SAE 2345 steel and high-temperature embrittlement in SAE 3140 steel. Proebstle (11) found the rate of high-temperature embrittlement to be highly dependent on the rate of ferrite grain growth.

Sadovskii (6) stated that the susceptibility to temper brittleness is greatly reduced by requenching from between the Ac_1 and Ac_3 temperatures, but there was no indication whether or not this conclusion was based on more than room-temperature impact values. Jaffe, Buffum and Carr (12) noted that slow heating to the isoembrittlement temperature introduced the same amount of embrittlement as did slow cooling from this temperature, within the experimental limits.

Woodfine (15) showed that if double tempering treatments are carried out, the second treatment is the controlling one and there is no effect of the previous treatment. Bhat and Libsch (19) found no difference in the rate of high-temperature embrittlement, whether or not low-temperature embrittlement preceded it. However, they found low-temperature embrittlement to be structure-sensitive, in that previous high-temperature embrittlement retards low-temperature embrittlement. The results of Jaffe, Buffum and Carr (12) indicated that when several embrittlement treatments are applied to the same specimen, each contributes to the embrittlement. When the previous transition temperature was high, the introduction of an additional embrittlement treatment did not produce as large an increase in transition temperature as when the previous transition temperature was low. In a later paper, Jaffe and Buffum (10) showed that when a steel embrittled at one temperature is heated to a higher temperature (below the Ae_1) and held, the degree of embrittlement rapidly approaches that attributable to the higher temperature alone. When a steel embrittled by holding at one temperature is held at a lower temperature, the final degree of embrittlement is generally only slightly greater than if the treatment responsible for the higher degree of embrittlement had been applied alone. An apparent exception was when both treatments led to lower-nose embrittlement. In this case a transfer to lower temperatures sometimes produced a slight initial decrease in brittleness, which became an increase with longer holding.

Effect of Deformation

Sadovskii (6) reported that cold plastic deformation increases the impact values of steels in the brittle state and reduces the susceptibility to temper brittleness. He stated that plastic deformation in the austenitic state resulted in a great reduction in the susceptibility if the specimen was quenched immediately after the deformation. The deformation temperature was above the recrystallization temperature, but the immediate quench was found to prevent the occurrence of recrystallization. Recent work by Druzhinin, Taraska and Grdina (20) has confirmed that plastic deformation in the austenitic state, followed by immediate quenching to prevent recrystallization, inhibits the development of temper embrittlement.

Buffum and Jaffe (21) found that a reduction of the strain rate lowers the transition temperature, and that the transition temperature is affected in both the tough and embrittled conditions. They tested SAE 3140 steel with two strain rates differing by 5 million to 1. The effect seemed to be somewhat greater for the unembrittled specimens.

Effect of Embrittlement on Properties

A - Hardness and tensile properties

Woodfine (2) stated that temper brittleness does not affect hardness, yield stress, maximum stress, or tensile elongation. In severely temper-embrittled specimens a slight decrease in the reduction of area was apparent in a few steels. Lea and Arnold (22) also reported a slight decrease in percentage reduction of area, due to temper embrittlement. They also showed the embrittled condition to be associated with a star-type fracture with irregular radial cracks, whereas the same material in the tough condition had a cup-cone type fracture. Similar tensile fracture features were encountered by the author (23) in a previous investigation.

Woodfine (2) reported that embrittlement treatments resulted in significant decreases in the percentage reduction of area and in the maximum stress in tensile tests carried out at -196°C (-320°F). Sadovskii (6) also noted that temper brittleness can be revealed by low-temperature testing, both static-bending and static-tensile.

This topic of tensile behaviour is the subject of further consideration in a later section of this report.

B - Fatigue and physical properties

While Woodfine (2) stated that temper brittleness does not affect the fatigue properties, recent work by Weiss, Niedzwiedz and Breuer (24) on high-silicon, high-manganese steels has indicated that, although temper embrittlement has no effect on the fatigue limit, at stresses just above this limit there is a reduction in endurance. With increasing stress this influence diminishes, and at high stresses it disappears almost completely.

Woodfine (2) referred to the slight differences in the lattice parameters, specific gravity and electrical resistivity of steels in the tough and embrittled conditions but stated that they were not sufficiently significant to aid in distinguishing between the two conditions. Maloof (25) reported differences he had observed in the lattice parameters in the tough and embrittled conditions. McLean and Northcott (26) found a difference in the electrode potential of the embrittled grain boundary as compared with that of the non-embrittled boundary. Sadovskii (6) referred to the reported increase in internal friction in the temper-brittle state, and this effect has been employed in recent studies by Grdina, Glikman and Piguzov (27,28).

C - Impact properties

Notched-bar impact testing remains the only test giving anything approaching quantitative results. The effect of temper embrittlement is both to lower the impact values and to displace the ductile-to-brittle transition to higher temperatures. According to Woodfine (15), temper brittleness reduces the brittle fracture stress of a susceptible steel without affecting the yield stress or strain-hardening. Uzhik and Zuikova (29) stated that steel in the embrittled condition has a considerably smaller capacity for the development of plastic deformation under conditions where cracks have already developed.

An important aspect of the temper-embrittlement phenomenon, and the factor which allowed temper brittleness to be detected in many steels with only a room-temperature impact test, is the impairment of the upper-shelf of impact energy. Carr, Goldman, Jaffe and Buffum (30) found that for SAE 3140 steel the maximum impact-energy level (within the hardness range Rc $20\frac{1}{2}$ to 38) was approximately 10 ft-lb higher for unembrittled samples than for embrittled samples. They found a linear relationship between impact-energy level and hardness in the range Rc 27 to 38. Buffum and Jaffe (31) found that for the same steel, within the hardness range, Rc $24\frac{1}{2}$ to $32\frac{1}{2}$, the transition temperatures for tough and embrittled materials rise in a parallel fashion with an increase of hardness.

Another characteristic of temper brittleness revealed by impact testing is the brittle fracture mode. Jaffe and Buffum (10) noted that the crystalline fractures of impact test specimens, broken at low temperatures, appeared different, even to the naked eye, for the unembrittled material, or for material embrittled in the temperature region associated with lower-nose embrittlement, than for material embrittled in the upper region. Microscopical examination of metallurgical sections revealed that upper-zone embrittlement caused the brittle fracture to follow predominantly along the ferrite grain boundaries. Lower-zone (reversible) embrittlement caused a fracture along prior austenite grain boundaries. The fracture of the unembrittled material was predominantly trans-crystalline cleavage. Woodfine (15) pointed out that the transition from the unembrittled state to that of the embrittled is gradual, and that as the degree of embrittlement increases there is a progressive decrease in the number of cleavage facets observed.

Special Etching Effects

Many metallographic etchants have been developed to reveal the grain structure in quenched-and-tempered steels, and much of the interest in this subject was prompted by investigations of temper-embrittlement phenomena.

Woodfine (15) described the distinctive etching behaviour noted for temper-brittle steels, whereby samples which have been embrittled display a preferential grain boundary attack, whereas unembrittled samples do not. This effect is probably due to the difference in electrode potential of the embrittled grain boundary as compared with that of the unembrittled boundary, as demonstrated by McLean and Northcott (26). Woodfine showed, by means of thermal etching techniques, that the main grain boundaries delineated by the etch correspond to the prior austenite grain boundaries, and that the sub-boundaries observed generally correspond to ferrite grain boundaries. He reported that no continuous grain-boundary material is observed by electron microscopical examination, but, rather, only grooves which widen continuously with longer etching times.

Jaffe and Buffum (10) noted that material embrittled in the range of temperature resulting in reversible temper embrittlement was more rapidly attacked than was the unembrittled, and that material embrittled in the upper region was, if anything, attacked less than the unembrittled material.

Cohen, Hurlich and Jacobson (32) noted that with extended etching times the grain-boundary attack can be produced in non-embrittled samples as well as in the embrittled, and they speculated that some temper embrittlement is inherent in all steels once they are reheated.

Most of the metallographic reagents used for this type of investigation have been based on picric acid solutions. Woodfine (15) used a 10-minute etch in a saturated solution of picric acid in distilled water, followed by a light re-polish and a light subsequent etch in picral. Sadovskii (6) recommended a solution of picric acid in xylene, and Jaffe and Buffum (10) used ethereal picric acid. The reagents used in more recent work have incorporated special wetting agents, such as zephiran chloride as recommended by Cohen, Hurlich and Jacobson (32), and sodium tridecylbenzene sulfonate as recommended by Dreyer, Austin and Smith (33).

The subject of grain-boundary etching effects and their association with temper embrittlement is treated further in a later section of this report.

Effect of Method of Manufacture

Greaves and Jones (34) reported the following order of decreasing susceptibility to temper brittleness for various steelmaking processes: acid open-hearth, basic open-hearth, electric-furnace, and crucible. However, no supporting data or reasons were presented. Proebstle (11) found that induction-melted steels exhibited a lower rate of reversible temper embrittlement than did basic electric-arc steels.

Any major differences due to process variation are probably attributable to the degree of deoxidation. Goodchild (3) stated that aluminum-killed steels are less susceptible to temper embrittlement than are steels killed solely by ferromanganese or ferrosilicon. Proebstle (11) found that deoxidation practice has a definite effect on the unembrittled transition temperature and on the rate of reversible temper embrittlement. Deoxidation with aluminum plus silicon gave a lower unembrittled transition temperature and a lower rate of reversible temper embrittlement than did deoxidation with silicon alone. It should be pointed out that this deoxidation practice also appeared to cause a slightly higher rate of high-temperature embrittlement, due to an increased rate of martensite decomposition caused by the presence of aluminum.

Effects of Chemical Composition

A - Carbon

There is general agreement that plain carbon steels exhibit no temper embrittlement when manganese contents are below 0.5 per cent. Some authors, including Libsch, Powers and Bhat (35), argued that plain carbon steels are highly susceptible, so much so, in fact, that embrittlement is developed too quickly to be suppressed by water cooling. They held to the opinion that any embrittlement occurring during tempering should be called temper embrittlement, whereas most investigators hold that only reversible embrittlement, characterized by brittle fractures along prior austenite grain boundaries, is true temper embrittlement. Other effects are considered only the normal consequence of the tempering process. Entwistle and Smith, in the discussion of a paper by Spretnak and Speiser (36), referred to a 0.4 per cent C, 0.65 per cent Mn steel in which the low-temperature fracture of the embrittled specimen was mostly characterized by cleavage, which would seem to correspond to the unembrittled state, rather than to the reversible embrittlement or upper-nose embrittlement behaviours postulated by Jaffe and Buffum (10).

The carbon content in alloy steels has a very marked effect. There is clear evidence that the susceptibility tends to decrease as the carbon content decreases to very low levels. Some steels, notably the SAE 3300 series, appear to exhibit the greatest embrittlement with carbon contents between 0.15 to 0.20 per cent. Hultgren and Chang (8) referred to results indicating that steels are susceptible down to 0.016 per cent carbon, but not at 0.003 per cent.

B - Manganese

Of all the normal alloying elements, manganese has the strongest effect in increasing susceptibility to temper embrittlement. Goodchild (3) stated that there is little effect below 0.4 per cent manganese, but that the susceptibility increases continuously with increasing manganese content, so that above 0.7 per cent the effect is quite pronounced. Small additions of manganese lower the transition temperature in the unembrittled condition, and larger additions raise it to a relatively high level. There is a lowering of the maximum impact-energy level when the manganese content is higher than 2 per cent.

The level of manganese content required to induce susceptibility to temper embrittlement is strongly influenced by the content of other alloying elements. Hultgren and Chang (8) referred to a 0.2 per cent carbon steel and showed that 2.1 per cent manganese made it strongly susceptible. However, with 3 per cent chromium present, only 0.6 per cent manganese was necessary to make the steel strongly susceptible, and in many chromium-nickel steels only 0.5 per cent is sufficient.

C - Nickel

Nickel steels are generally considered relatively insusceptible to temper brittleness. Hultgren and Chang (8) found that more than 3 per cent nickel is necessary to induce even a small embrittling effect in an Fe - 0.2 per cent C alloy. Chromium or phosphorus additions to insusceptible nickel steels render them susceptible. In like manner, nickel additions increase the susceptibility of chromium, manganese and chromium-manganese steels.

Nickel additions result in a lowering of the impact transition temperature of material in the unembrittled condition, and nickel contents over 3 per cent apparently lower the maximum impact-energy level.

D - Chromium

Early opinion, as expressed by Jolivet and Vidal (37), was that plain chromium steels are susceptible, with the susceptibility increasing steadily with increasing chromium content. However, in the absence of other elements, it is now felt that chromium results in little embrittlement. Nickel additions to insusceptible chromium steels render them susceptible, and Taber, Thorlin and Wallace (38) showed that the presence of chromium in amounts greater than about 0.6 per cent greatly increases the susceptibility of many steels.

Hultgren and Chang (8) stated that if a chromium addition gives needed hardenability, it lowers the transition temperature in the unembrittled condition. There was no effect observed on the maximum impact strength level.

E - Nickel, chromium, manganese combinations

Nickel-manganese and chromium-manganese steels are generally remarkably susceptible to temper embrittlement. The susceptibility of nickel-chromium-manganese steels generally increases with an increase in the content of any one of these components. Holloman (39) suggested that if two steels of this type have the same hardenability with respect to nickel, chromium and manganese, they will have approximately the same susceptibility to temper embrittlement.

F - Phosphorus

In the absence of alloying elements, phosphorus in the amounts normally present in steels has little effect on temper embrittlement, but it is generally agreed that temper brittleness can result from abnormally high phosphorus contents. However, phosphorus does increase the degree of susceptibility in susceptible steels, and it induces susceptibility in nickel and chromium steels.

These commonly accepted views of the effects of phosphorus seem to be somewhat at variance with recently published Russian work (27, 28), based on internal friction measurements, whereby it was concluded that the susceptibility of steels to temper embrittlement is basically determined by the phosphorus and carbon contents, and that most of the embrittlement is stimulated by the concentration of phosphorus on the grain boundaries of the alpha solid solution.

G - Silicon

Sadovskii (6) mentioned some Russian work in which additions of silicon to an insusceptible nickel steel rendered it susceptible. Columbier (40) reported that a 1.5 per cent addition of silicon to a chromium-nickel steel considerably increased the temper brittleness. Capus (41) referred to temper brittleness caused by the use of silicon as a deoxidizer.

H - Aluminum and titanium

Results available on the effect of aluminum and titanium are somewhat contradictory. Woodfine showed that a 0.5 per cent addition of aluminum to a chromium steel did not affect the embrittlement (2), and that titanium and aluminum additions to a nickel-chromium steel apparently increased the embrittlement (15). In his review (2) he referred to results that indicated a reduction in temper brittleness due to titanium (based on room-temperature results), and to other results that showed titanium additions to have no apparent effect on the embrittlement of a boron-bearing steel. Braun and Drukovskaya (42) have recently claimed that titanium decreases the susceptibility to temper embrittlement.

I - Boron, sulphur, copper, vanadium, antimony, arsenic and tin

Powers and Carlson (43) reported that a 0.0034 per cent addition of boron seemed to increase the susceptibility of a steel. Woodfine (2) referred to results showing that an increase in the sulphur content from 0.016 to 0.037 per cent in a chromium steel had no effect on the subsequent embrittlement. Goodchild (3) stated that the effects of copper and vanadium are slight. Woodfine (2) referred to a chromium steel in which the addition of 0.23 per cent vanadium appeared to increase the susceptibility slightly. Capus (41) claimed that temper embrittlement can be caused by the presence of a specific group of potent elements in minute quantities, including arsenic, antimony and tin, as well as phosphorus. Woodfine (2) felt, on the other hand, that antimony produces a specific embrittlement that is distinct from temper brittleness.

J - Molybdenum, tungsten and niobium

Braun and Drukovskaya (42) have indicated that small additions of molybdenum, tungsten and niobium, as well as titanium, served to minimize the susceptibility in cast chromium-manganese-silicon steels. Vidal (44) reported that a 3.8 per cent tungsten addition to a steel with no chromium content and low manganese caused it to be susceptible. Sadovskii (6) stated that tungsten reduces embrittlement up to a content of 1.5 per cent, where it begins to form carbides. Vidal also reported that a plain 2 per cent molybdenum steel was susceptible. However, it is universally recognized that molybdenum in suitable amounts, usually in the order of 0.25 per cent, inhibits, or to a large degree retards, the development of temper brittleness in many steels. The examination of the effects of molybdenum additions in a series of low-alloy steels is considered further in subsequent sections of this report.

Mechanisms

Bush and Siebert (45) summarized most of the theories which have been advanced as to the cause of temper brittleness, as follows:

- a - a transformation below 700°C (1290°F)
- b - an allotropic modification of iron
- c - precipitation from ferrite of such compounds as carbides, nitrides, phosphides and chromium oxides
- d - decomposition of retained austenite
- e - modification of special carbides
- f - grain boundary segregation
- g - selective distribution of carbides

The two most credible explanations are the precipitation-solution theory and the grain-boundary segregation theory. Proponents of the former approach claim that this theory explains all the characteristics of this type of embrittlement. The main problem is that no precipitate has been seen and identified, and the tensile strength, hardness and yield strength are not affected as one would expect with a precipitation mechanism.

What appears to be a more likely mechanism involves the segregation of solute atoms. Because of the tendency of any system to seek the lowest energy state, solute atoms tend to migrate to high-energy areas, such as grain boundaries and other places of imperfection, in order to lower that energy. Arkharov (46) stated that at the grain boundaries there is a critical transitional layer several atoms thick which governs the development of temper embrittlement. This layer is so thin that processes can occur which are characteristic of highly alloyed steels, since even an extremely small number of companion atoms entering the boundary layer can increase its concentration in these elements considerably. Goodchild (3) proposed that below the sensitizing range the rate of diffusion is too low for effective segregation to occur. Above the sensitizing range, the lattice is effectively large enough to accommodate solute atoms without undue strain. (Actually, the lattice parameters at high temperatures are not much bigger, but the vibration of solvent atoms is considerably greater, with the result that each position is less defined and more solute atoms can be accommodated within the grain.) Within the embrittlement range there occurs an optimum condition for the adsorption of solute atoms at the grain boundaries.

There has been no definite identification of the solute atoms involved, and the mechanism by which small additions of molybdenum minimize temper embrittlement remains in doubt. However, most current investigators accept the theory of the redistribution by physical adsorption leading to unfavourable accumulations of certain elements at the grain boundaries as the most plausible mechanism of reversible temper embrittlement.

The most widely accepted theory of the cause of irreversible, upper-nose embrittlement involves a completely distinct mechanism, associated with sub-critical ferrite grain growth. As described by Woodfine (47), the transition temperature depends on the relative values of yield stress and brittle fracture strength. At temperatures before ferrite grain growth occurs, the decrease in yield stress due to spheroidization and growth of carbides predominates, and hence the transition temperature improves. With prolonged tempering, particularly above 600°C (1110°F), the continuing effect of spheroidization and carbide growth is outweighed by the effect of decreasing brittle fracture strength produced by ferrite grain growth, and the transition temperature rises.

<u>Summary</u>

The major features of temper-embrittlement phenomena may be summarized as follows:

a - Impact testing appears to be the only practical test giving anything approaching a quantitative picture of the effects of temper brittleness. Impact-energy curves over a range of testing temperature, which includes the ductile-brittle transition temperature, should be determined. Some embrittlement-measurement criterion which would take account of both the change in transition temperature and the actual impact-energy impairment would result in a more definitive rating of susceptibility to temper embrittlement.

b - The rate of reversible temper embrittlement reaches a maximum, which for a large number of low-alloy steels occurs at an isoembrittlement temperature of about 500°C (930°F). The embrittlement which occurs on slow cooling appears to be associated primarily with lower-nose embrittlement, and occurs at a somewhat more rapid rate than does isoembrittlement.

c - Lower-nose, or reversible, temper embrittlement can be removed by reheating to 600°C (1110°F) and rapidly cooling to a temperature below the embrittling range.

d - Reversible embrittlement is characterized by a low-temperature brittle fracture along the prior austenite grain boundaries, and a rapid metallographic etching attack in these same areas.

e - Manganese, chromium, nickel and phosphorus contents in steels appear to exert the primary influence on the degree of susceptibility to temper embrittlement. While these elements individually, in the absence of the others, must be present in relatively high amounts to result in significant embrittlement, if indeed any can be developed, the presence of combinations in suitable quantities greatly increases the susceptibility.

f - Some elements, primarily molybdenum in suitable small amounts, reduce the susceptibility to temper embrittlement.

g - The mechanism of reversible temper embrittlement is thought to be associated with an accumulation by physical adsorption of solute atoms at grain boundaries and other places of lattice imperfection.

h - Irreversible embrittlement, which occurs most rapidly on isothermal holding just below the Ae_1 temperature, is characterized by low-temperature brittle fracture along the ferrite grain boundaries, and appears to be primarily due to the effect of ferrite grain growth.

2. FURTHER EVALUATION OF THE EFFECTS OF MOLYBDENUM IN LOW-ALLOY STEELS

Introduction

The program reported herein was initiated primarily to provide specific detail of the effects of various quantities of molybdenum on the impact properties of a series of experimental low-alloy steels, particularly as related to the temper-embrittlement phenomenon. Other aspects are reported in subsequent sections of this report.

In quantities usual in alloy steels (up to 0.4 per cent), it is reported (48) that molybdenum raises the impact transition temperature. It will be shown later that under the experimental conditions of the present investigation the opposite effect was observed.

In quantities up to 0.5 per cent it is recognized that molybdenum inhibits the development of reversible temper embrittlement in many steels, whereas in larger amounts it has been shown (44) to promote temper-brittle behaviour.

It was hoped that a program which would provide specific details of the magnitude of the effects observed would be useful in providing a better understanding of the mechanisms involved.

Experimental Materials

The fourteen experimental steels investigated consisted of three basic compositions modified by various molybdenum contents, as shown in Figure 1.

PERCENTAGE COMPOSITION OF
EXPERIMENTAL STEEL HEATS

STEEL	Invariant Elements, %				Variable Elements, %			
	C	S	P	Cr	Mn	Si	Al	Ni
1	0.35	0.026	0.008	0.7	0.6	0.2	-	1.6
2					0.9	0.3	0.05	0.7
3					0.6	0.1	-	1.8

MOLYBDENUM PERCENTAGE
IN INDIVIDUAL INGOTS

A	B	C	D	X*	Y*
0.01	0.1	0.2	0.3	0.5	1.0

* - Steel 3 only.

Figure 1. Composition of experimental ingots.

Steels 1 and 2 were prepared using a small induction furnace. Four 13-lb sand-moulded billets were cast from both heats, one with residual molybdenum and one each at the 0.1, 0.2 and 0.3 per cent levels. Steel 3 was prepared in a 400-lb induction melting furnace, and 50-lb ingots were poured at residual, 0.1, 0.2, 0.3, 0.5 and 1.0 per cent molybdenum levels.

Drillings for chemical analysis were obtained from bars poured concurrently with the ingots of Steel 3, and from the underside of the cropped hot-tops for Steels 1 and 2. The results of chemical analysis are detailed in Table 1.

TABLE 1

Results of Chemical Analysis

STEEL	Percentage of Element								
	C	Mn	Si	S	P	Al	Cr	Ni	Mo
1A	0.36	0.58	0.25	0.024	0.008	-	0.66	1.57	0.01
1B	0.36	0.56	0.25	0.027	0.008	-	0.67	1.57	0.10
1C	0.35	0.56	0.23	0.027	0.008	-	0.66	1.57	0.20
1D	0.36	0.56	0.21	0.029	0.008	-	0.66	1.57	0.32
2A	0.32	0.89	0.30	0.027	0.008	0.07	0.67	0.68	0.01
2B	0.33	0.90	0.31	0.027	0.008	0.05	0.66	0.68	0.11
2C	0.32	0.91	0.31	0.028	0.008	0.04	0.66	0.68	0.21
2D	0.31	0.91	0.31	0.028	0.008	0.03	0.66	0.68	0.31
3A	0.35	0.53	0.13	0.025	0.007	<0.01	0.65	1.80	0.01
3B	0.32	0.61	0.14	0.026	0.008	<0.01	0.66	1.80	0.10
3C	0.34	0.61	0.14	0.027	0.008	<0.01	0.66	1.80	0.19
3D	0.34	0.60	0.14	0.027	0.007	<0.01	0.65	1.80	0.29
3X	0.34	0.60	0.12	0.026	0.007	<0.01	0.64	1.80	0.49
3Y	0.31	0.55	0.07	0.026	0.008	<0.01	0.64	1.80	1.02

Processing

A - General

The processing details are summarized in Figure 2.

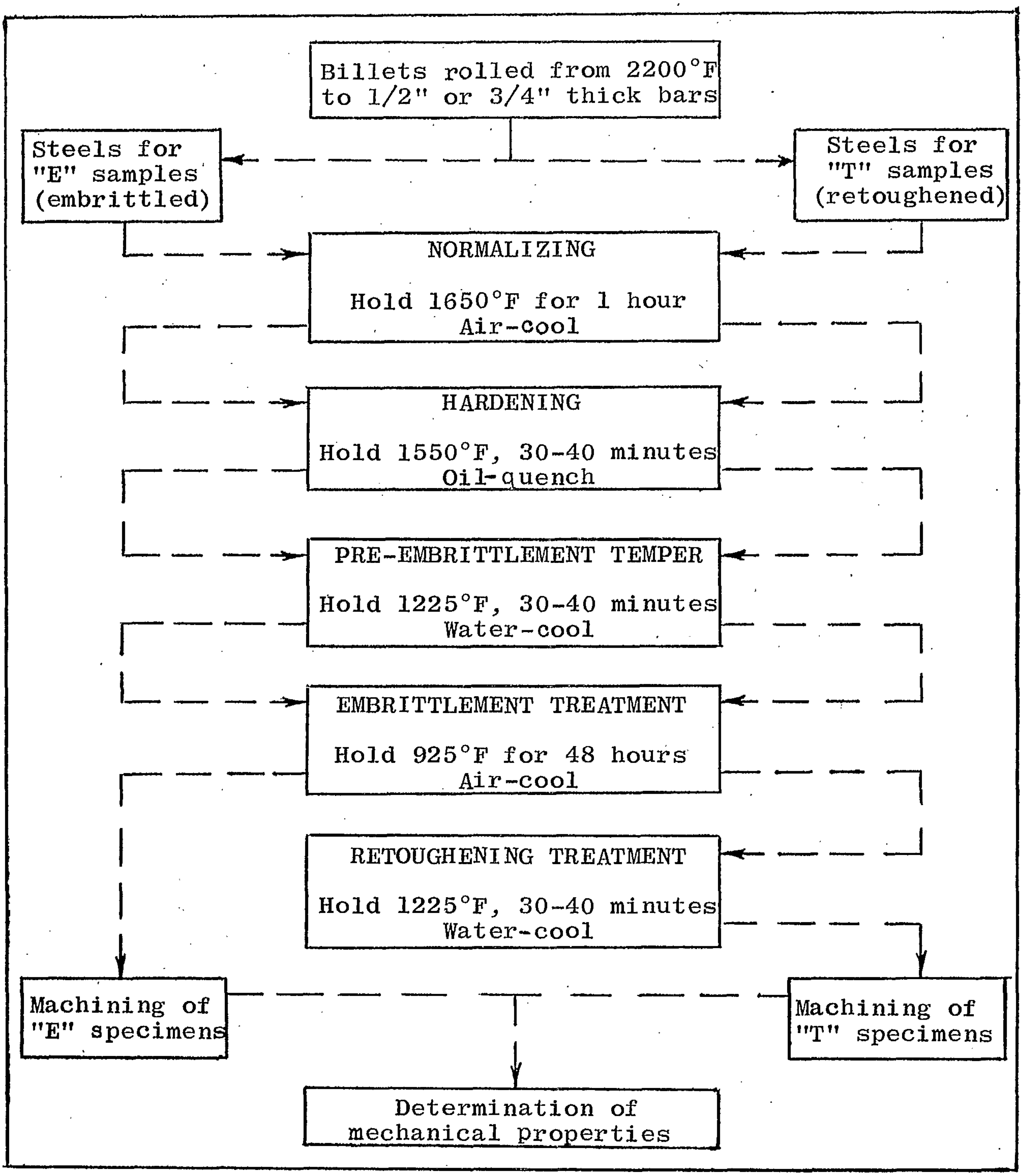


Figure 2. Summary of processing details.

B - Rolling

The cropped billets of Steels 1 and 2 were rolled from 1205°C (2200°F) to 1/2-inch-thick bars, effecting an average cross-section reduction of 65 per cent. The cropped Steel 3 ingots were rolled from the same temperature to 3/4-inch bars, giving an average reduction of about 80 per cent.

C - Heat Treatment

The steels were to be examined in two conditions, namely "embrittled" (E) and "retoughened" (T). All materials were normalized; austenitized and quenched; given a short pre-embrittlement temper at 662°C (1225°F) to minimize the hardness differential between the two test conditions; and, finally, given extended isoembrittlement treatment at a temperature corresponding to the lower nose of the isoembrittlement curve for steels of similar composition. One half of the stock from each bar was then retoughened by a further draw at 662°C (1225°F) and a rapid cooling through the embrittlement temperature range.

D - Test Specimen Preparation

From each of the experimental steels, in both the embrittled and retoughened conditions, sufficient Charpy V-notch impact specimens were obtained to establish the impact transition curves. Reliable hardness values were obtained by averaging numerous Rockwell readings, using separately machined specimens in the case of Steel 3, and broken impact bars for the other steels.

Impact Properties

The impact transition curves for all the steels in both conditions of heat treatment were established by breaking Charpy V-notch test specimens at various testing temperatures. The fracture-appearance curves were established as well, by examining and rating the fractures as to per cent crystallinity. These curves are reproduced in Figures 3, 4 and 5, with the experimental points eliminated for the sake of clarity. In these figures the dashed curves are for "E" steels, and solid curves are for "T" steels. The curves rising to the right are impact-energy curves, and those rising to the left are fracture-appearance curves.

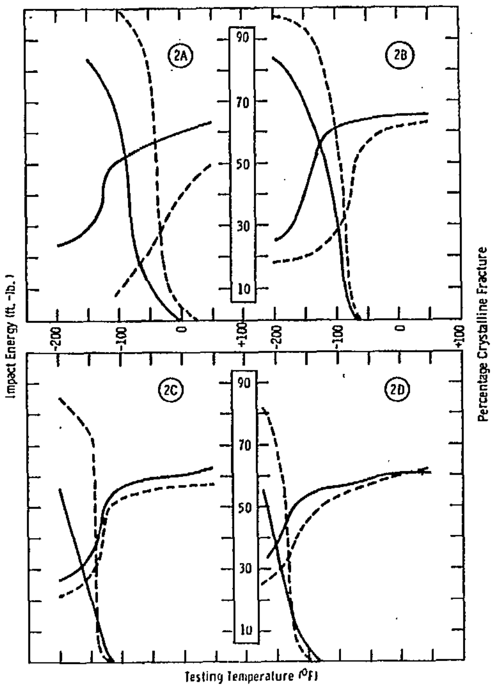


Figure 3

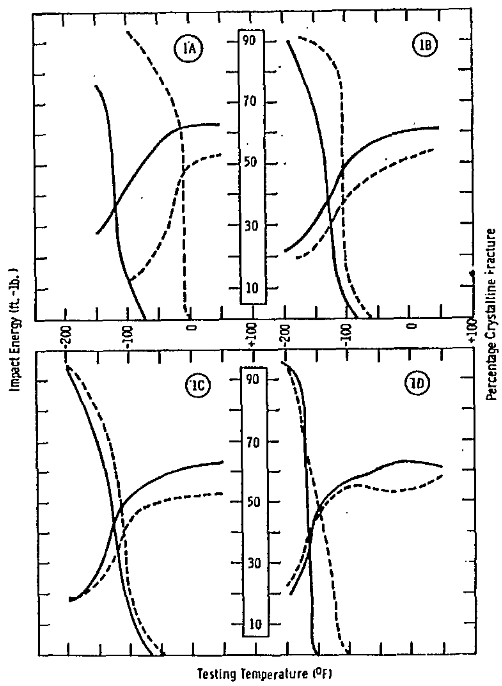


Figure 4

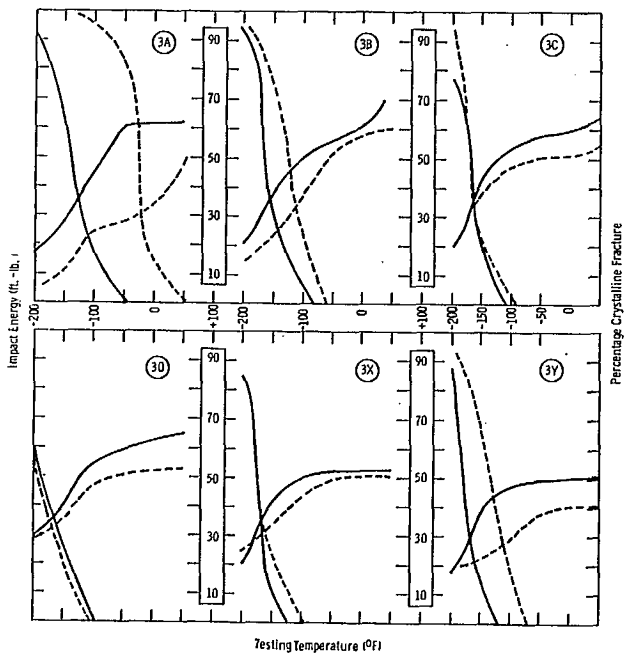


Figure 5

Figures 3, 4 and 5. Impact and fracture-appearance curves for Steels 1, 2 and 3 respectively.

For comparison purposes, the selection of the 35-ft-lb transition-temperature criterion would seem to be the most satisfactory for these steels, since at this energy level the impact curves and the fracture-appearance curves are all reasonably near the mid-range of transitional behaviour. The transition temperatures of the embrittled steels (TTe) and of the retoughened steels (TTt) are listed in Table 2.

TABLE 2

35-ft-lb Impact Transition Temperatures

% Mo Level	Steel	Transition Temperature, °F	
		TTe	TTt
0.01	1A	- 30	-122
	2A	- 13	-130
	3A	+ 6	-129
0.1	1B	-113	-137
	2B	- 84	-155
	3B	-106	-162
0.2	1C	-121	-137
	2C	-139	-153
	3C	-157	-163
0.3	1D	-167	-161
	2D	-180	-213
	3D	-146	-170
0.5	3X	-145	-165
1.0	3Y	- 71	-157

From Table 2 it can be seen that the addition of molybdenum resulted in a significant improvement in the impact transition temperature for these steels in both conditions of heat treatment. Of course, the improvement is far greater in the steels subjected to the embrittling treatment, due to the superimposed improvement in susceptibility to temper embrittlement. This effect of molybdenum on the impact transition temperature is presented graphically in Figure 6, where δ_{TT}, the improvement in the impact transition temperature as compared with that of the base composition containing residual molybdenum, is plotted for each level of molybdenum. The values shown in Figure 6 are obtained from the averages for the three steels at each molybdenum level.

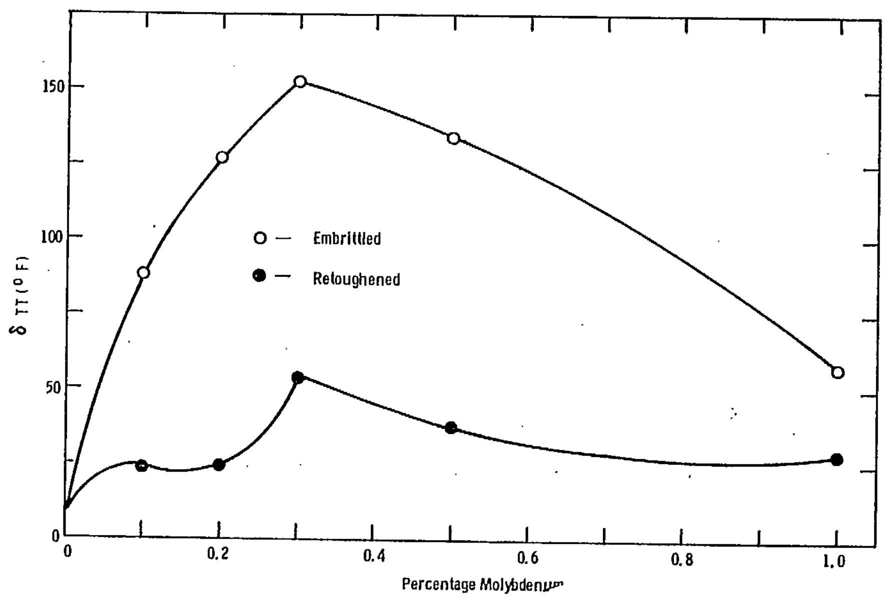


Figure 6. Effect of molybdenum content on the improvement of the 35-ft-lb impact transition temperature.

It can be seen that in both conditions of heat treatment an optimum effect is reached at the 0.3 per cent molybdenum level. For the retoughened steels the decreased effect beyond this level is attributable to a slightly higher hardness due to increased resistance to tempering. This also applied to some extent for the embrittled steels, but some of the decrease in effect must obviously be due to some other mechanism. It will be seen, later, that there is an optimum molybdenum content beyond which the susceptibility to temper embrittlement gradually returns, and it is probably this effect that is reflected here.

Embrittlement Susceptibility

Since Vidal and Jolivet (5) showed that the use of a "susceptibility ratio" based on room-temperature impact values is not a satisfactory method to reveal temper brittleness, the general practice has been to consider the degree of temperature-displacement of the impact transition due to the embrittlement treatment as a measure of susceptibility. The suitability of this technique is considered elsewhere in this report, but, with one exception, the scope of this presentation is limited to the more conventional approach, using primarily the 35-ft-lb energy-level criterion to define the impact transition temperature. Hence the susceptibility to temper embrittlement is rated in terms of Δ_{TT}, which is the difference between the 35-ft-lb transition temperatures for the two conditions of heat treatment.

The exception referred to above is a criterion based on difference in impact-energy level, which is presented for comparison purposes. This criterion was defined as Δ_{maxI}, the maximum difference in impact energy between the embrittled and retoughened steels, occurring at some temperature resulting in 20 per cent or more crystallinity in the embrittled condition. The fracture-appearance limitation was imposed to ensure that the region of the curves being considered will be in the transitional range. Without this restriction, this would not necessarily be the case for some steels exhibiting only a small degree of susceptibility.

Using these criteria, the susceptibilities of the experimental steels were calculated, and these are recorded in Table 3, along with the temperature which corresponds with the maximum difference in impact energy for each steel. Figure 7 is a graphical representation of these data.

TABLE 3

Ratings of Susceptibility to Temper Embrittlement

% Mo Level	Steel	Susceptibility Criteria		
		Transition Temperature	Impact Energy	
		Δ_{TT} (F°)	Δ_{maxI} (ft-lb)	Temp
0.01	1A	92	34	- 65°F
	2A	117	40.5	-110°F
	3A	135	32	- 50°F
0.1	1B	24	11	-105°F
	2B	71	36	-120°F
	3B	56	16	-125°F
0.2	1C	16	8.5	-120°F
	2C	14	7.5	-150°F
	3C	6	5.5	-145°F
0.3	1D	nil	0	-137°F
	2D	33	12	-177°F
	3D	24	5.5	-145°F
0.5	3X	20	8.5	-140°F
1.0	3Y	86	19	-125°F

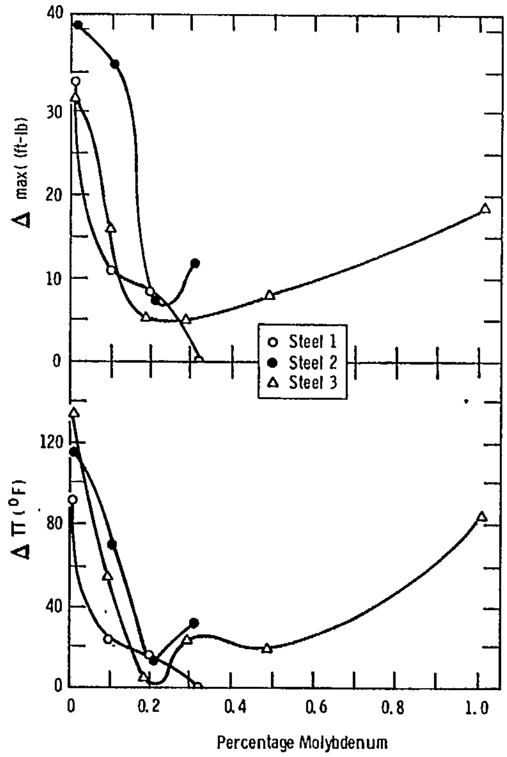


Figure 7. Effect of molybdenum content on the degree of susceptibility to temper embrittlement, as measured by two criteria.

It is apparent that the two criteria chosen exhibit similar features, and both are probably valid representations of the susceptibility behaviour of these experimental steels. Comparison of the various curves provides an illustration of some compositional effects apart from the effect of molybdenum. Steel 3 can be seen to be slightly more susceptible than Steel 1, and to require slightly more added molybdenum to reduce the susceptibility to equivalent levels. This is as would be predicted from the slightly higher nickel content of Steel 3. In the case of Steel 2, the effect of the increased manganese content would be opposed by that of the relatively large decrease in nickel content. Holloman (39) suggested that two steels having the same hardenability with respect to nickel, chromium and manganese contents would have approximately the same degree of susceptibility. Hence, it would be expected that Steels 2 and 3, for which the contributions of nickel, chromium and manganese to the hardenability multiplication factor as per Grossmann (49) are practically identical, should exhibit identical susceptibility behaviour. While no significant differences are apparent for the two molybdenum-free steels, it can be seen that for Steel 2 more added molybdenum is required to achieve equivalent levels of susceptibility than for the other steels. This difference in behaviour is probably attributable to an increase in embrittlement due to the small addition of aluminum to Steel 2, as suggested by Woodfine (15). This is in conflict with Goodchild's (3) view that aluminum-killed steels are less susceptible to temper embrittlement than are steels killed solely with ferromanganese or ferrosilicon.

It is readily apparent that the susceptibility to temper embrittlement is markedly reduced by the addition of molybdenum, and there also appears to be an optimum content beyond which the improvement begins to disappear. This is illustrated graphically in Figure 8, which shows a plot of the improvement in the susceptibility ratings ($\delta\Delta_{TT}$ and $\delta\Delta_{maxI}$), as compared with the residual-molybdenum base steels, versus the molybdenum increment (δ_{Mo}). These plots show the average improvements for the three experimental compositions. It can be seen that, for the experimental steels under consideration, the optimum molybdenum content for minimizing temper embrittlement is approximately 0.2 per cent. The decline in effectiveness beyond this point is only slight, at least up to 0.5 per cent.

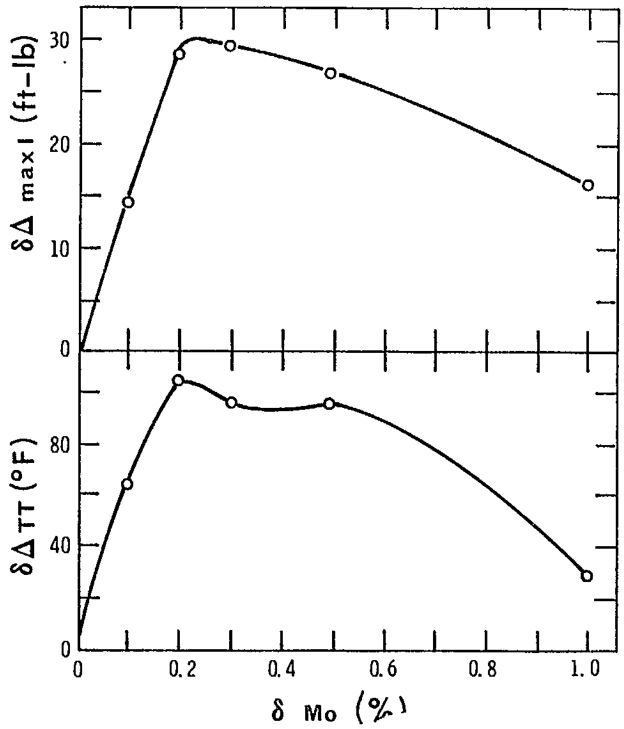


Figure 8. Improvement of the two chosen susceptibility ratings associated with various increments of molybdenum.

Since the choice of the transition-temperature criterion would in many cases have a large influence on the degree of temper embrittlement indicated, three other criteria were tested for comparison purposes, namely:

A - the lowest temperature for which the fracture appears to be 100 per cent fibrous.

B - the temperature corresponding to a 50 per cent fibrous fracture.

C - the temperature at which the impact strength is mid-way between the values at high and low testing temperatures, as proposed by Hultgren and Chang (8). (In order to use this criterion, some of the "upper -

shelf" and "lower-shelf" values had to be estimated.)

The results obtained using these three transition-temperature criteria are summarized in Figure 9, which shows the averaged values observed for Steels 1, 2 and 3. This figure illustrates the improvements associated with various molybdenum increments (δ_{Mo}), as follows:

a - the improvement in the susceptibility rating ($\delta\Delta_{TT}$);

b - the improvement in the transition temperature for the "retoughened" condition (δ_{TTt});

c - the improvement in the transition temperature for the "embrittled" condition (δ_{TTe}).

These are improvements as compared with the values for the "residual-molybdenum" steels, which are indicated on the figure for each of the three comparison-criteria.

In this particular case, the trends reflected by the comparison-criteria are in general similar to those previously described. The significant improvement in transition temperature attributable to molybdenum, with the optimum effect at 0.3 per cent, is still apparent. The fact that there is an optimum quantity of molybdenum required to minimize the development of temper brittleness is also still obvious. However, the actual molybdenum level associated with the optimum effect is not so clear. While from the impact-energy transitional behaviour the optimum seems to be at 0.2 per cent Mo, the value appears to be higher when fracture-appearance criteria are used.

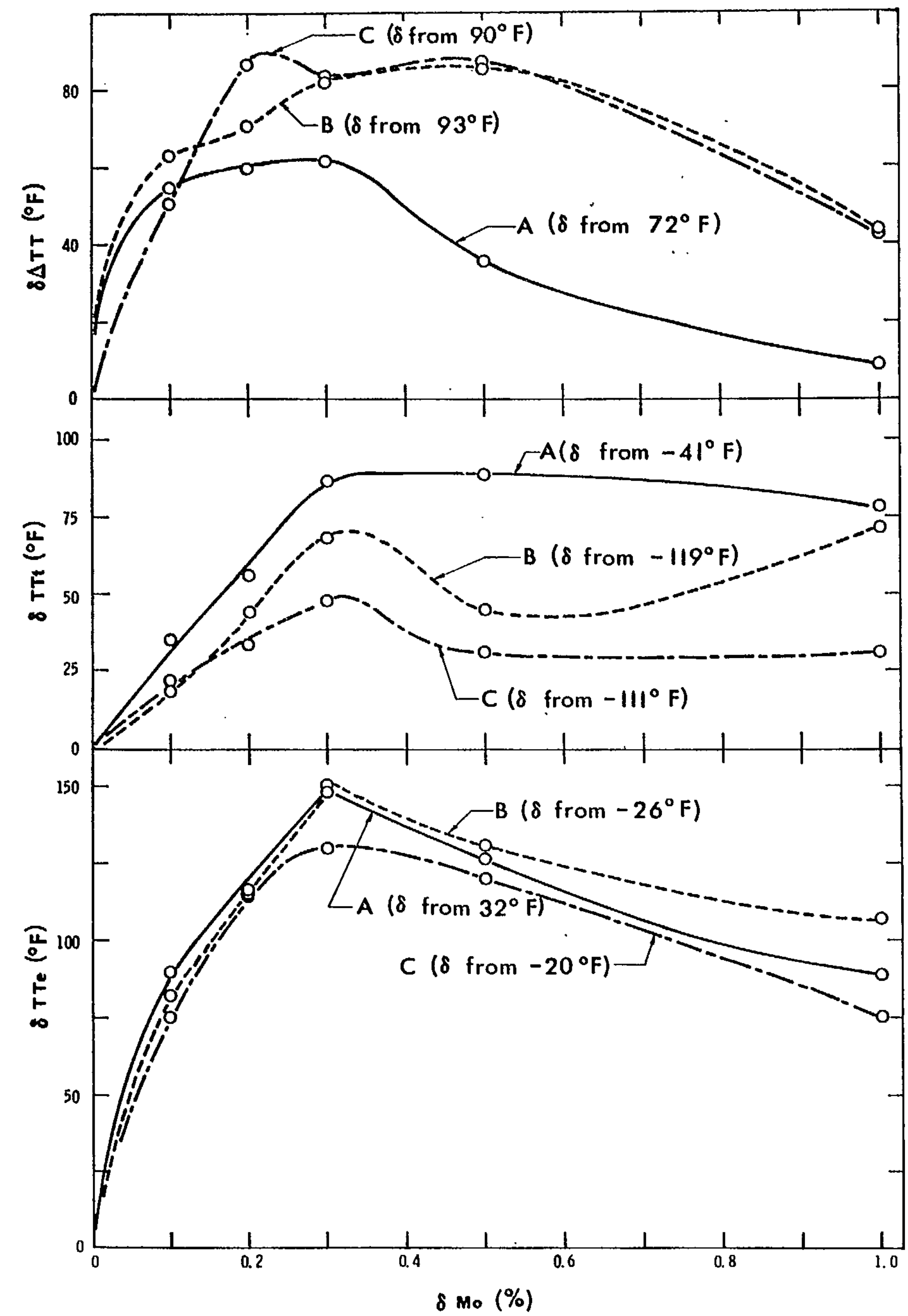


Figure 9. The effect of increased molybdenum, for comparison-criteria A, B and C, on the improvements in:
- susceptibility to temper embrittlement ($\delta\Delta TT$)
- transition temperature in the retoughened condition (δTTt)
- transition temperature in the embrittled condition (δTTe).

Summary

The addition of molybdenum to the experimental low-alloy steels investigated resulted in significant improvements in the impact transition temperatures in both the embrittled and retoughened conditions. There was a definite optimum molybdenum content associated with this effect, which occurred at 0.3 per cent molybdenum.

It was readily apparent that small additions of molybdenum caused marked improvements in susceptibility to temper embrittlement, as rated by four criteria measuring shifts in the impact transition temperature (two using the impact-energy curve, and two the fracture-appearance curve), as well as another criterion rating the maximum decrease in impact-energy level. All criteria confirmed that there is an optimum molybdenum content beyond which the susceptibility begins to return, but the value is not as definite as that for the improvement in impact transition temperature. The optimum effect appeared to be at about 0.2 per cent as rated by impact-energy data, and slightly higher as rated by fracture-appearance data. There was little actual difference in the degree of susceptibility for these steels between the 0.2 and 0.5 per cent molybdenum levels.

The data were illustrative of other compositional effects, such as the slight increase in the susceptibility to temper embrittlement associated with an increase in the nickel content. An addition of aluminum in quantities common in aluminum-deoxidation practice appeared to increase the degree of embrittlement, as indicated by the quantity of molybdenum required to lower the degree of susceptibility of the three series of steels to equivalent levels.

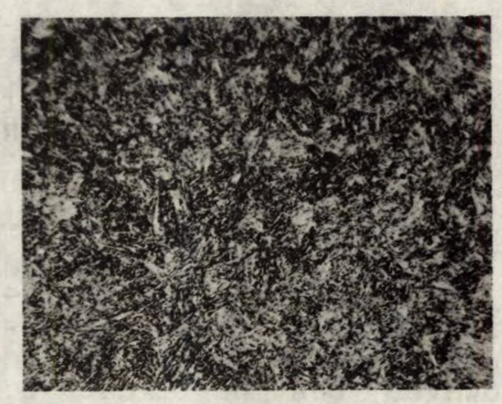
3. GRAIN-BOUNDARY ETCHING EFFECTS AS RELATED TO TEMPER EMBRITTLEMENT

Introduction

The distinctive grain-boundary etching characteristics noted in previous investigations of temper embrittlement have been summarized in an earlier section of this report. These can, however, best be illustrated by example. Since the SAE 3300 series of steels are noted for their susceptibility to temper embrittlement, some commercial SAE 3330 bar stock was given a series of heat treatments and microspecimens were prepared to represent each stage. These were all etched for the same period in a saturated solution of picric acid in distilled water, and Figure 10 shows the various microstructures associated with the following stages of heat treatment:

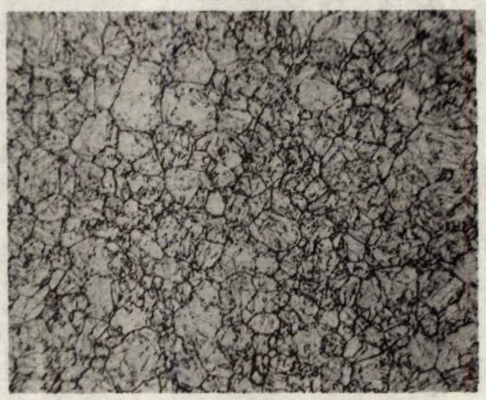
Stage I	- Normalized at 1700°F for 1 hour	- Rc $47\frac{1}{2}$
Stage II	- Hardened - austenitized at 1500°F for 1 hour and oil-quenched	- Rc $51\frac{1}{2}$
Stage III	- Pre-tempered at 1175°F for 1 hour and oil-cooled	- Rc 27
Stage IV	- Embrittled at 925°F for 48 hours and oil-cooled	- Rc $24\frac{1}{2}$
Stage V	- Retoughened - retempered at 1175°F for 1 hour and oil-cooled	- Rc 24
Stage VI	- Re-embrittled at 925°F for 48 hours and oil-cooled.	- Rc $23\frac{1}{2}$

I - Normalized



II - Hardened

III - Pre-tempered



IV - Embrittled

V - Retoughened

VI - Re-embrittled

(All X250)

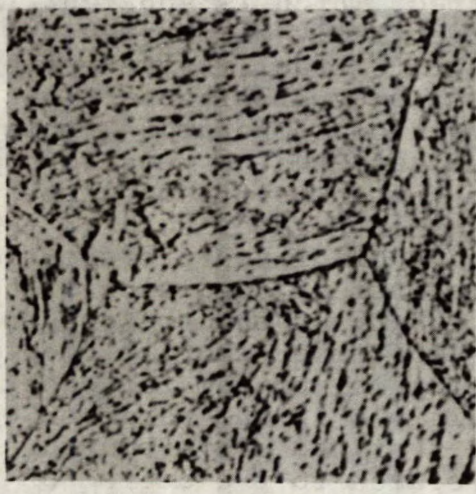
Figure 10. Microstructures after various stages of heat treatment of SAE 3330 sample.
(All samples etched 5 minutes in a saturated solution of picric acid in water.)

From the foregoing it is readily apparent how the inference has been drawn that such grain-boundary etching behaviour is indicative of temper-brittle material. However, current work on a series of molybdenum-bearing steels makes it apparent that some qualification of this viewpoint is called for.

Current Investigations

Metallographic examinations were carried out on the series of experimental steels referred to in the previous section of this report. All samples, in both the "embrittled" and the "retoughened" conditions, were etched in the reagent containing sodium tridecylbenzene sulfonate, described by Dreyer, Austin, and Smith (33). As can be seen from Figure 11, which illustrates the structures of the embrittled and re-toughened Steel 3 samples containing residual molybdenum, the distinct grain-boundary etching effect which is apparent in the embrittled condition is almost entirely eliminated by the retoughening treatment. Since this steel is susceptible to temper embrittlement, as revealed by a displacement of 135 F° in the 35-ft-lb impact transition temperature, this etching behaviour would seem to indicate that a metallographic determination of susceptibility to temper brittleness is possible. However, it can be seen from Figures 12 to 14 that all the steels involved in this study displayed preferentially etched grain boundaries in the "embrittled" condition of heat treatment. This was not apparent in any of the "retoughened" samples. Since the susceptibility to temper embrittlement was shown to decrease with increasing molybdenum for each of the three steels, becoming negligible in the range of 0.2 to 0.3 per cent Mo, it is apparent that the grain-boundary etching characteristic cannot be related to the degree of susceptibility to temper embrittlement.

3AE - (X500)



3AE - (X2000)

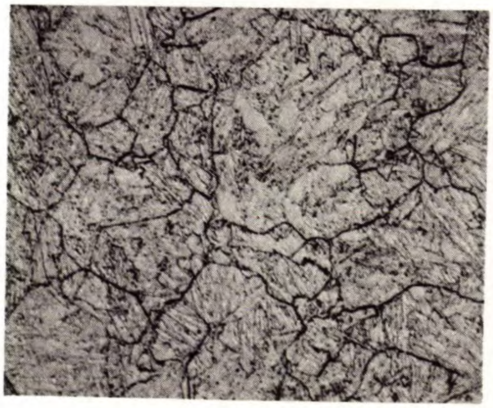
3AT - (X500)

Figure 11. Etched microstructures of Steel 3 samples containing residual molybdenum.

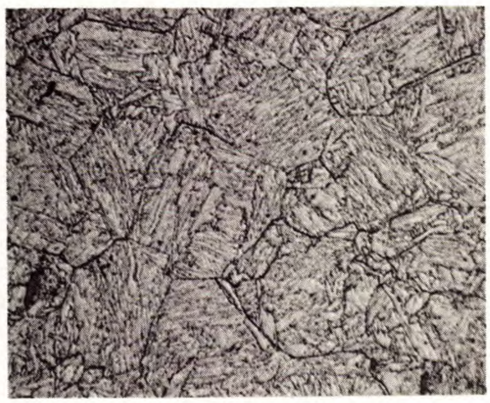
The differential grain-boundary attack seen in the photomicrographs of the embrittled sample (left) is not apparent after the retoughening treatment (right).

0.01% Mo

0.1% Mo



0.2% Mo



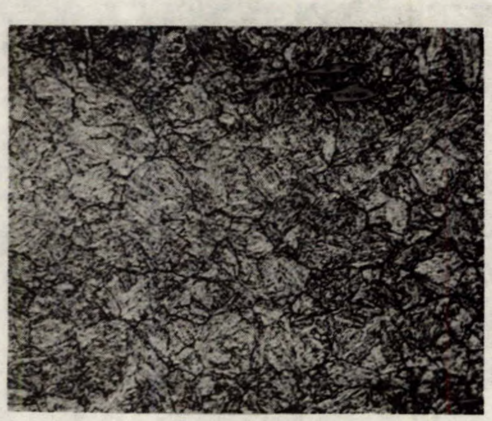
0.3% Mo

(All X500)

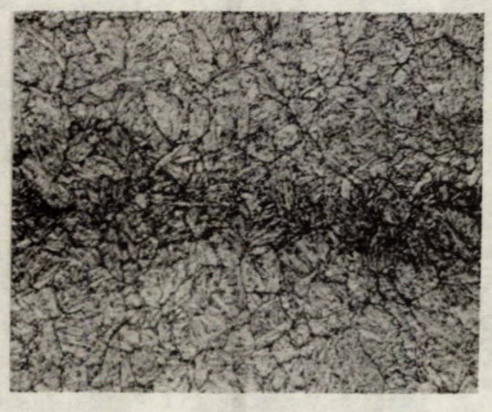
Figure 12. Microstructures of all Steel 1 compositions in the "embrittled" condition.

All "embrittled" samples exhibit a grain-boundary etching effect. All the corresponding "retoughened" samples (not shown) show very little or no grain-boundary etching.

0.01% Mo



0.1% Mo



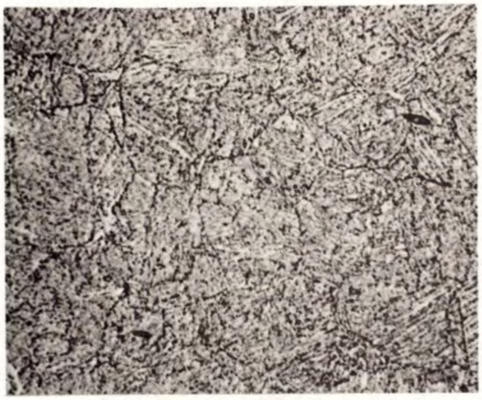
0.2% Mo

0.3% Mo

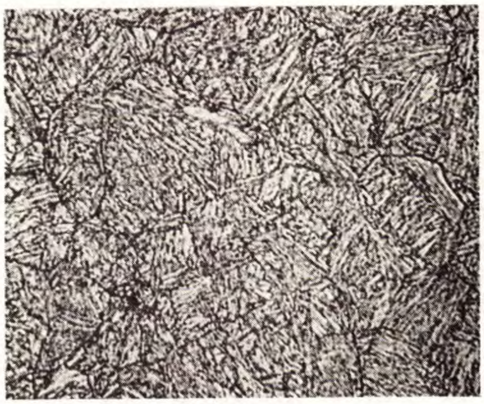
(All X500)

Figure 13. Microstructures of all Steel 2 compositions in the "embrittled" condition.

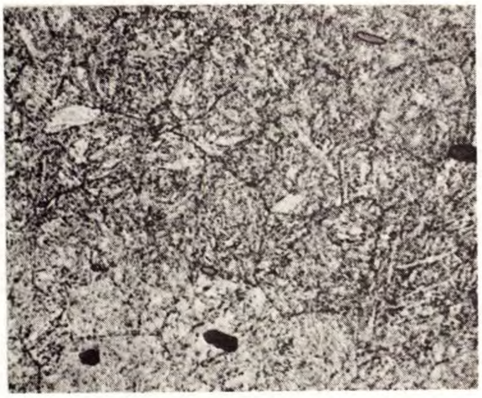
All "embrittled" samples exhibit a grain-boundary etching effect. All the corresponding "retoughened" samples (not shown) show very little or no grain-boundary etching.



0.01% Mo



0.1% Mo



0.2% Mo

0.3% Mo

0.5% Mo

1.0% Mo

(All X500)

Figure 14. Microstructures of all Steel 3 compositions in the "embrittled" condition.

All "embrittled" samples exhibit grain-boundary etching effects. All the corresponding "retoughened" samples (not shown) showed very little or no grain-boundary etch.

Conclusions

Regardless of the degree of susceptibility to temper embrittlement displayed by the experimental steels, they all showed the etching behaviour previously described as being characteristic for temper-brittle materials. Hence, the distinctive grain-boundary etching behaviour does not necessarily identify a material as temper brittle, but rather, it is indicative of its condition of heat treatment. If it is in the "embrittled" condition--that is, heat-treated so that, if susceptible, it will become embrittled--the distinctive grain-boundary etching occurs. Even this is a relative effect, since "retoughened" samples will etch at the grain boundaries if the etching duration is extended.

The mechanism of reversible temper embrittlement has been described as being associated with an accumulation, by physical adsorption, of solute atoms at grain boundaries and other places of lattice imperfection. This would seem to be consistent with the observed differences in the electrode potential of the embrittled grain boundary as compared with that of the unembrittled boundary, as well as with the different etching behaviour in the two conditions. It is apparent that whatever the mechanism may be by which molybdenum decreases the susceptibility to temper embrittlement, there is no interference with the mechanism responsible for the grain-boundary etching behaviour.

4. EFFECT OF EMBRITTLEMENT TREATMENTS ON THE TENSILE PROPERTIES OF EXPERIMENTAL STEELS

Introduction

In the course of conducting the temper-embrittlement investigations reported in earlier sections of this report, a program of tensile testing at room temperature and at reduced temperature was carried out on experimental Steels 2 and 3. The tensile properties of the Series 3 steels were determined in duplicate, using 0.505-inch-diameter specimens. A supplementary evaluation of the properties of this series was also carried out at -70°C (-94°F). For Series 2 steels there was sufficient material for only single tests using 0.313-inch-diameter specimens. Accurate hardness values were assured for all steels in both conditions of heat treatment, by averaging a relatively large number of hardness readings.

Results

The results of room-temperature tensile and hardness testing are listed in Table 4, and are plotted in Figure 15 in relation to the scatter bands of normal expectancy for steels consisting essentially of tempered martensite as described by Hodge and Bain (50). The results of reduced-temperature tensile testing are listed in Table 5.

TABLE 4

Room-Temperature Tensile and Hardness Properties of Experimental Steels

Steel	Average Rc Hardness	UTS (kpsi) Converted From Rc	TENSILE PROPERTIES				
			UTS (kpsi)	YS λ (kpsi)	Y/U	Elong. (%)	R.A. (%)
2AE	23	115.0	114.4	99.8	0.87	22.4	63.6
2AT	22½	113.5	111.8	92.3	0.91	24.8	63.6
2BE	25	120.0	119.4	103.3	0.87	20.8	61.4
2BT	24½	118.5	119.0	105.0	0.88	24.0	64.9
2CE	25½	121.5	125.0	108.3	0.87	21.6	61.0
2CT	26	123.0	121.6	107.0	0.88	23.2	63.6
2DE	26½	125.0	125.7	109.6	0.87	24.0	62.3
2DT	26½	125.0	123.1	111.8	0.91	22.4	60.0
3AE	19½	107.0	114.3	94.8	0.83	22.3	62.4
3AT	21	110.0	111.7	92.5	0.83	23.8	66.1
3BE	25	120.0	123.6	106.2	0.86	21.0	63.8
3BT	23	115.0	117.5	99.5	0.85	22.0	64.1
3CE	26½	125.0	129.8	112.8	0.87	21.0	63.5
3CT	24½	118.5	122.0	104.7	0.86	22.8	64.9
3DE	28½	131.0	133.2	117.2	0.88	21.0	63.4
3DT	26	123.0	126.2	108.8	0.87	21.8	64.3
3XE	30½	138.0	142.3	126.7	0.89	20.5	61.4
3XT	28½	131.0	133.1	117.0	0.88	22.0	63.5
3YE	33½	149.0	150.8	137.2	0.91	18.3	57.5
3YT	30	136.0	138.9	123.9	0.88	20.3	61.4

λ - Yield strength for Series 2 by lower yield point and for Series 3 by 0.2% offset.

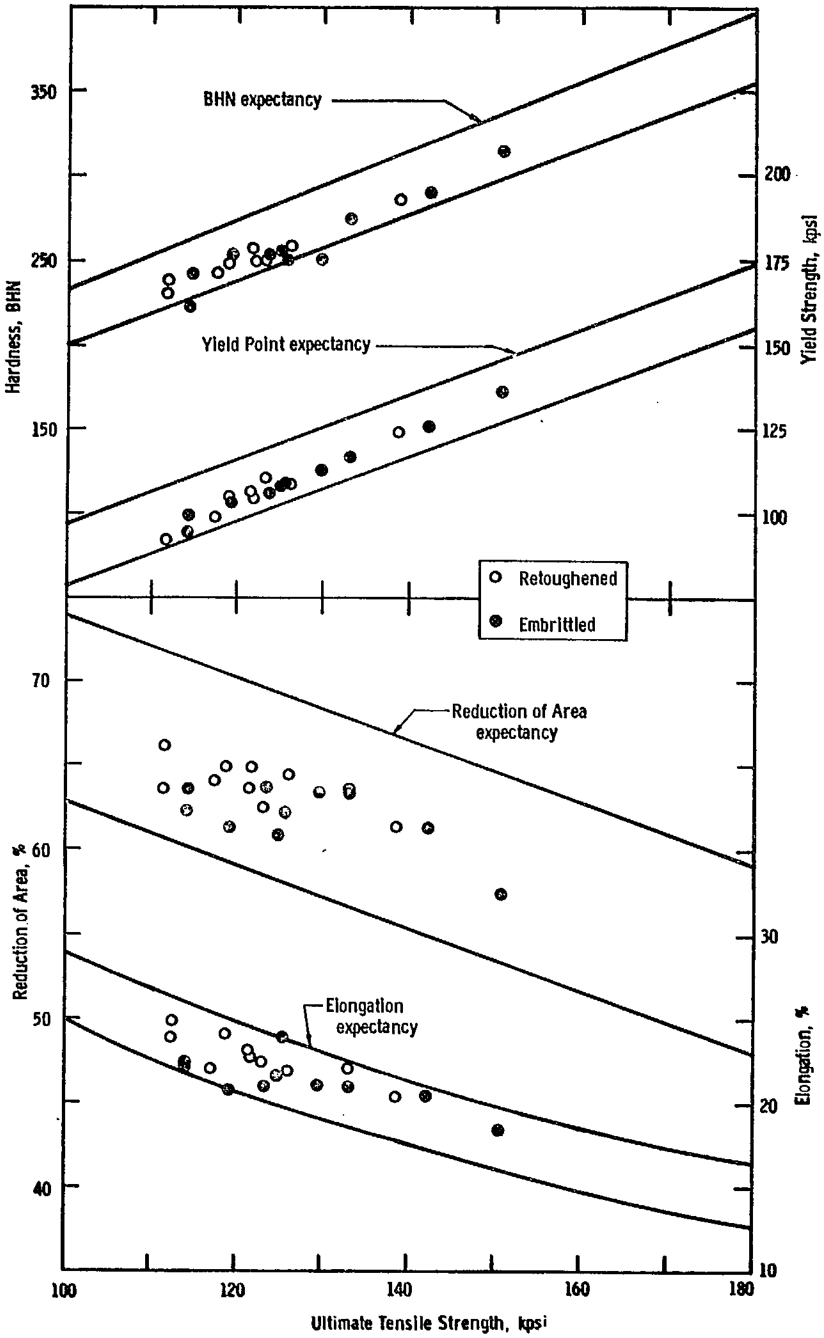


Figure 15. Correlation of strength functions in experimental steels.

Bands enclosed by the solid lines are the normal expectancy for each function for steels consisting essentially of tempered martensite (50).

TABLE 5

Reduced-Temperature* Tensile Properties

Steel	UTS (kpsi)	0.2% Offset YS (kpsi)	Y/U	Elongation (%)	R. A. (%)
3AE	128.8	n.d.	n.d.	21.5	57.1
3AT	128.2	102.7	0.80	26.0	62.2
3BE	139.1	116.0	0.83	25.0	57.3
3BT	132.5	107.5	0.81	25.5	63.3
3CE	142.8	122.0	0.85	23.0	57.0
3CT	136.0	112.6	0.83	24.0	63.0
3DE	147.8	125.6	0.85	23.5	56.0
3DT	140.0	117.2	0.84	24.0	61.0
3XE	156.6	134.2	0.86	21.0	58.0
3XT	148.0	125.0	0.84	23.0	61.6
3YE	166.4	144.1	0.87	22.0	55.2
3YT	152.3	n.d.	n.d.	21.0	58.3

* -70°C (-94°F); n.d. - not determined.

As can be seen from Figure 15, there is a good correlation with the experimental scatter expected for low-alloy quenched and tempered steels, and no striking differences between the "embrittled" and "retoughened" conditions are apparent. The slight improvements observed in the percentage elongation and percentage reduction of area appear to be consistent with the small drop in hardness level caused by the retoughening treatment (in spite of the pre-temper which was carried out to minimize this effect). In any case, there was no effect directly relatable to temper-embrittlement phenomena, since the improved ductility is apparent for all levels of molybdenum, regardless of the degree of susceptibility to temper embrittlement.

The effect of molybdenum on various strength functions, for both the "embrittled" and "retoughened" conditions, is shown in Figure 16.

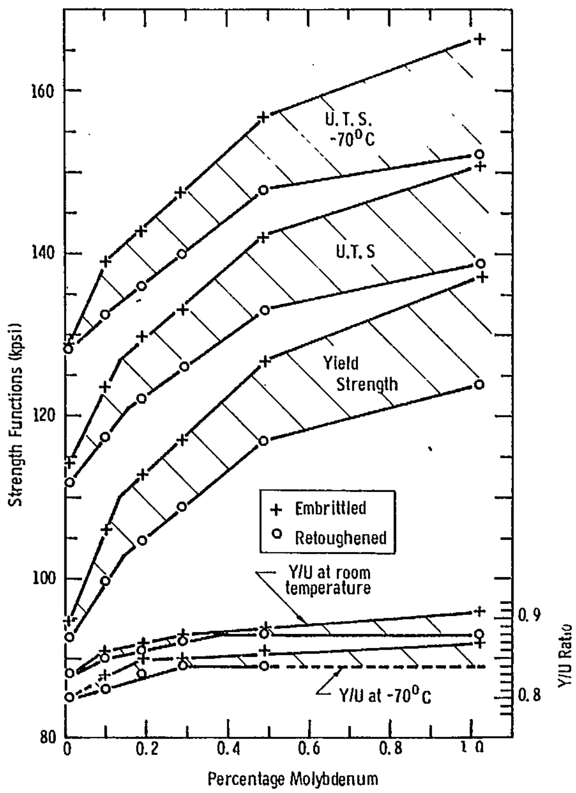


Figure 16. Effect of molybdenum content on various strength functions of experimental steels.

Except for displacement along the strength axis, the curves are essentially identical for:

a - ultimate tensile strength at room temperature;
b - 0.2 per cent offset yield strength at room temperature;

c - ultimate tensile strength at -70°C (-94°F);
d - yield strength at -70°C (-94°F);
e - tensile strength predicted from hardness values.

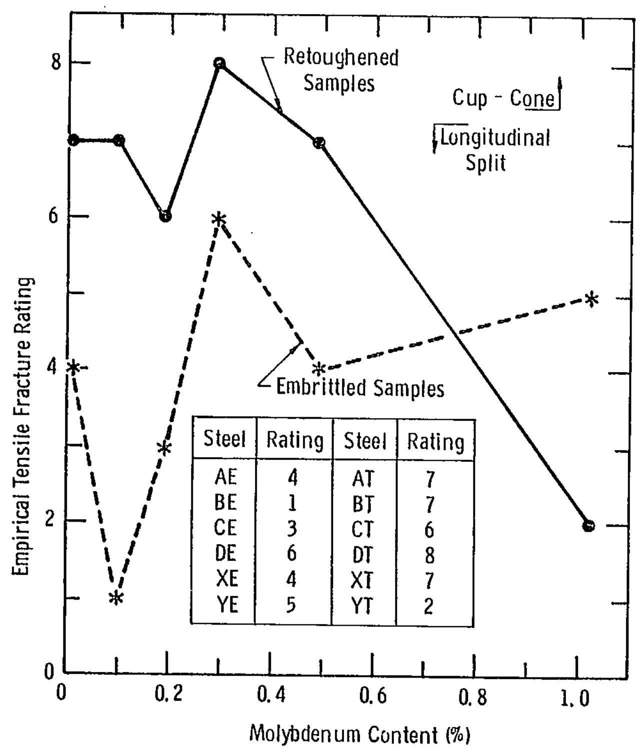
For the sake of clarity the latter two functions are not illustrated in Figure 16, but they would occur at displacements of 9 kpsi and 4 kpsi respectively, below the curves shown for ultimate tensile strength.

Again, there does not appear to be any anomalous behaviour which could be directly related to temper embrittlement. In the "embrittled" condition there is a more rapid strength increase accompanying the initial molybdenum increment than is found for the "retoughened" condition. This is attributable to the effect molybdenum has in retarding the softening due to tempering. For the molybdenum-free steel, the level of hardness resulting from the pre-temper and embrittlement treatments was such that little further softening resulted from the retoughening treatment. The addition of molybdenum up to about 0.15 per cent apparently introduced a progressively greater resistance to tempering, such that the retoughening treatment results in significant further softening. Beyond the initial increment of molybdenum and up to about 0.5 per cent, the resistance to tempering remains constant, so that with increasing molybdenum content the strength level increases at about the same rate (4 kpsi per 0.1 per cent Mo) for both conditions of heat treatment. Beyond 0.5 per cent, the resistance to tempering appears to increase slightly, but the rate of increase in strength with increasing molybdenum decreases.

A comparison of the tensile behaviour at room temperature and at -70°C (-94°F) shows that although there is a marked increase in the ultimate strength at the lower temperature, the yield-to-ultimate ratio actually drops slightly. With the exception of the molybdenum-free steel in the embrittled condition, there was a small but consistent increase in tensile ductility at the reduced temperature as measured by percentage elongation. Recent data presented by Ebert, Krotine and Troiano (51) have shown similar effects on ductility down to an abrupt transition temperature below which the ductility decreases rapidly.

The only reflection of the temper-embrittlement phenomenon that was apparent from tensile testing was the appearance of the fractures. As observed in other work by Lea and Arnold (22) and the author (23), the "embrittled" samples tended to display a higher incidence of longitudinal, radial splitting than did the "retoughened" samples.

On visually comparing the fractures, there seemed to be a minimum degree of splitting associated with the 0.3 per cent molybdenum level, for both the "embrittled" and the "retoughened" conditions. In order to present a graphical representation of this effect, the fractures of the room-temperature samples were empirically rated according to the degree of longitudinal splitting. A rating of "1" was used to indicate the most extensive splitting observed, and the rating "8" indicated the best-developed "cup-cone" fracture. All the samples were rated between these limits by visual comparison, and Figure 17 shows a graphical representation of the results of this empirical fracture-rating exercise.



Steel	Rating	Steel	Rating
AE	4	AT	7
BE	1	BT	7
CE	3	CT	6
DE	6	DT	8
XE	4	XT	7
YE	5	YT	2

Figure 17. Effect of molybdenum content on empirical tensile-fracture rating.

It can be seen that, at least up to 0.8 per cent Mo, the tensile specimens in the retoughened condition are less prone to longitudinal splitting than are the equivalent embrittled specimens. The best-developed cup-cone fracture occurs for both conditions of heat treatment at approximately 0.3 per cent molybdenum.

In both conditions there is an increased degree of splitting associated with the initial increment of molybdenum. It is of interest to note that the minima of the two curves shown in Figure 17 both occur at approximately the same strength level. However, the actual appearances of the fractures at these minima are considerably different, and so it is obvious that some mechanism other than different degrees of hardness is responsible for the different behaviour.

The series of samples tested at -70°C (-94°F) also exhibited an increased degree of splitting associated with the initial increment of molybdenum. It was also readily apparent that any such rating based on fracture appearance is critically dependent on the testing temperature, in that all samples tested at the reduced temperature exhibited a far greater degree of splitting than did the corresponding room-temperature bars.

Conversely, another series of bars tested experimentally at a higher than normal rate of tensile loading (1.0 rather than 0.05 inch per minute crosshead speed), while not indicating any significant differences in tensile properties, all showed a notable decrease in longitudinal splitting. In fact, only the 1 per cent Mo steel in the embrittled condition (i.e., the hardest steel) showed any evidence of splitting.

Summary

The delaying action of molybdenum on the tempering reaction was confirmed by the occurrence of a hardness difference between the two test conditions beyond the initial increment of molybdenum.

From 0.1 to 0.5 per cent molybdenum, all strength functions increased at the rate of 4 kpsi per 0.1 per cent molybdenum. Beyond 0.5 per cent Mo and up to 1.0 per cent, the rate of increase in strength declined somewhat.

There was no difference in the tensile properties, per se, which could be attributed to the temper-embrittlement process. The slight increase in tensile ductility for the retoughened condition as compared with the embrittled condition can be ascribed to the slight decrease in hardness.

In spite of substantial increases in strength shown in tensile tests carried out at reduced temperature, there was a slight improvement in tensile elongation as compared with the room-temperature values.

Comparison of the tensile-fracture appearance provides a tenuous reflection of temper-embrittlement phenomena. After an initial increase in the degree of longitudinal splitting, a further addition of molybdenum up to the 0.3 per cent Mo level, where susceptibility to temper embrittlement is very slight, results in an optimum cup-cone appearance for both conditions of heat treatment.

The tensile-fracture appearance is strongly influenced by testing temperature and test speed. A reduced testing temperature greatly increased the degree of longitudinal splitting, and an increase in tensile-test speed practically eliminated splitting.

5. A NEW APPROACH IN RATING RELATIVE SUSCEPTIBILITY TO TEMPER BRITTLENESS

General

The introductory section of this report reviewed the early use of a "susceptibility ratio" rating based on room-temperature impact values, and the development of the currently accepted practice of measuring the difference in the impact transition temperature of "embrittled" and "retoughened" steels. The viewpoint of most Russian investigators, that the important factor involved in embrittlement is the impairment of the impact strength, and that a shift in the transition temperature does not necessarily reflect the actual degree of impairment at any particular temperature, was pointed out. Again, there is no universal agreement on the proper choice of a transition-temperature criterion, and indeed, while one criterion may be selected as suitable to characterize the embrittlement within one set of experimental conditions, it may not be suitable under another set of conditions. Another problem inherent in the use of transition-temperature data to compare relative susceptibilities of steels is that much of the reported data is a tabular presentation of the absolute displacement of the measured transition temperature and the conclusions are formulated without regard to the temperature at which the transitional behaviour occurs. The view of Mikhailov-Mikheev (7), that no steel should be considered susceptible if the transitional behaviour does not occur down to the particular service temperature of interest, may be somewhat extreme. However, there is certainly validity in the argument that a displacement of the transition at temperatures near ambient temperature is of greater consequence than an equal displacement at very low temperatures which are unlikely to be encountered in service.

A new method for evaluating susceptibility is proposed herein, and is illustrated by applying it to a further examination of various results from the literature, as well as to the current Mines Branch data.

Proposed Method

In view of the aforementioned difficulties encountered in the use of impact transition temperatures to characterize temper embrittlement, it is proposed that a plot of a "susceptibility function" (based on the impact energies) versus testing temperature is an improved criterion for rating the susceptibility of steels. The "susceptibility function" (SF) is a modification of the "susceptibility ratio" used in the early work, and is defined as follows:

$$SF = \frac{Iu}{Ie} (Iu - Ie)$$

The "Iu" and "Ie" values are the energy levels of the "unembrittled" and "embrittled" steels, as determined from the impact-energy versus testing-temperature curves, for various testing temperatures.

Consideration was given to the use of Iu/Ie values, but it can be seen that such a function would not take into account the significance of the absolute drop in impact strength. Hence, a value of 5 would be recorded for a drop from 200 to 40 ft-lb, and for a drop of 10 to 2 ft-lb. On the other hand, the measurement of the actual drop in impact strength (Iu - Ie) would not be acceptable in all cases. For example, a value of 50 would be shown for a drop from 200 to 150 ft-lb as well as for a drop from 55 to 5 ft-lb. In one case the drop would not be of practical significance, in that there would be sufficient impact strength for full ductility in both conditions, whereas in the other case the failure mode would probably be mostly ductile for the tough condition and fully brittle in the embrittled condition.

The use of such a system gives a rating of the maximum degree of embrittlement based on the impact properties themselves, and not on some arbitrarily selected transition-temperature criterion, and allows one to take into account the temperature at which the maximum degree of embrittlement occurs.

Application of Proposed Method

A - Examination of Data from the Literature

None of the commonly used criteria for evaluating susceptibility to temper embrittlement is universally applicable, because of the variety of conditions and materials that have been investigated. Therefore, in order to ensure that a good sampling of conditions is represented in this evaluation of the proposed method, data from six different reports were selected, as shown in Table 6, and the results were re-examined using the proposed method, as shown in Figure 18.

TABLE 6

Identification of Materials and Source of Data

Steel Shown	Chemical Composition					Reference
	% C	% Mn	% Ni	% Cr	% Mo	
A	0.1	0.5	3.5	1.5	-	8
B	0.3	0.75	1.5	0.75	0.25	38
C	0.37	0.65	-	0.57	0.15	3
D	0.39	0.79	1.26	0.77	0.02	21
E	0.35	0.87	1.16	0.50	0.005	52
F	0.18	0.52	-	-	0.93	53

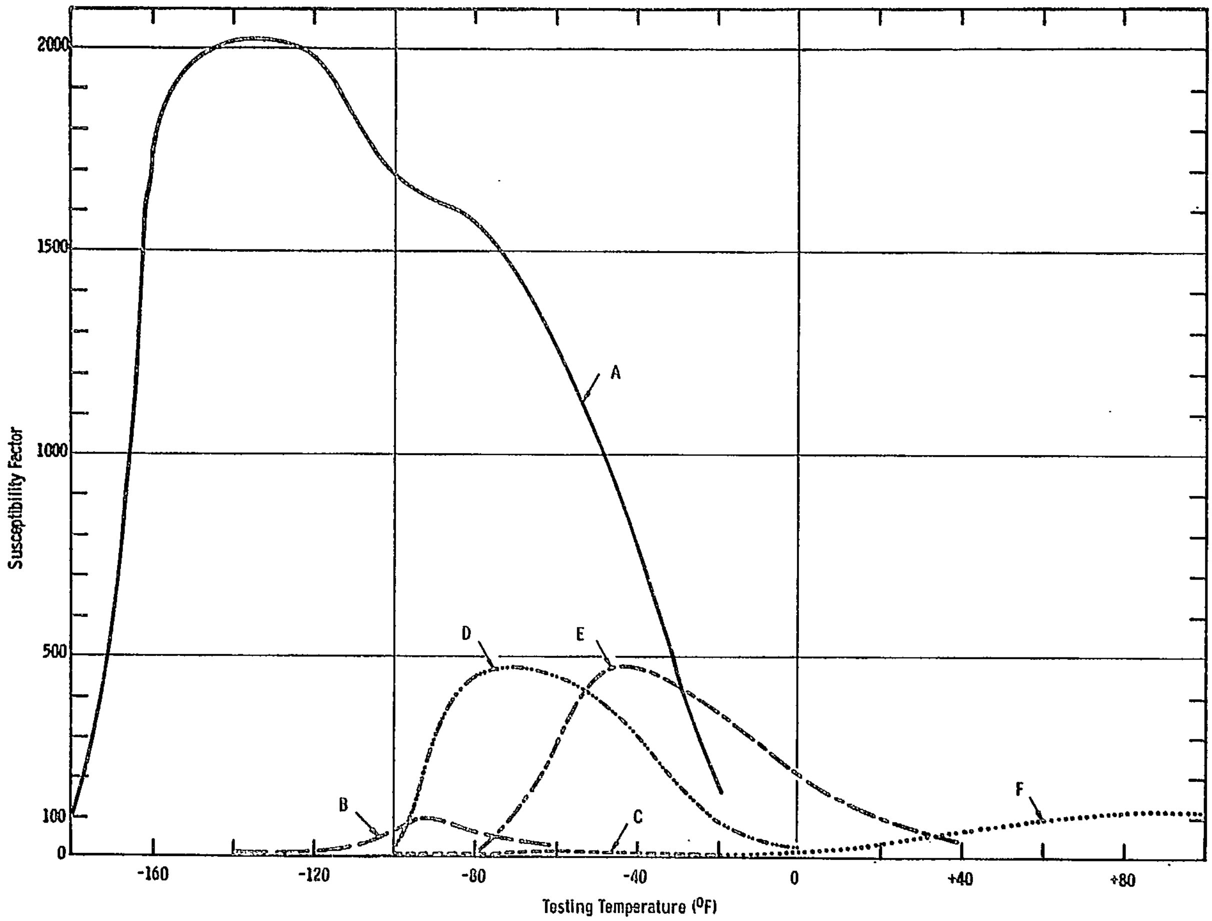


Figure 18. Illustration of application of proposed method for rating susceptibility to temper embrittlement.

Steel A, the susceptibility of which is readily apparent by any transition-temperature criterion, can be seen to have an extremely high maximum-susceptibility factor. Of course, the magnitude of the factor has in this case been enhanced by a comparatively high upper-shelf of impact energy associated with the relatively high alloy content and low carbon content. It can be seen, on the other hand, that the maximum-susceptibility factor occurs at a testing temperature of about $-93°C$ ($-135°F$), and that the rating at what would be rather severe winter temperatures is not much different from that of Steels E and D.

Steels D and E, which appear to have approximately the same susceptibility rating by transition-temperature methods, can be seen to have almost identical maximum-susceptibility factors. However, if one were rating these two steels, the proposed method of presentation would make it immediately apparent that the temperature at which the maximum occurs is approximately 17 centigrade (30 Fahrenheit) degrees higher for Steel E than for Steel D.

The curves for the molybdenum-bearing steels are quite illustrative of the effects documented in the literature. Comparison of Steels B and D which are similar in composition with the exception of the molybdenum content, illustrates the marked improvement due to small quantities of molybdenum. Steel C is seen to be even less susceptible with a smaller quantity of molybdenum. This is undoubtedly a consequence of the smaller tendency to embrittlement to be counteracted in this grade of steel, due to the absence of nickel. The curve for Steel F is illustrative of the reversibility of the beneficial effect of molybdenum, whereby higher levels apparently induce embrittlement. It can be readily seen that the high temperature at which the maximum-susceptibility factor occurs is in this case of paramount importance.

B - Re-evaluation of Current Experimental Data

The data obtained from a re-evaluation of the results reported in an earlier section of this report, using the proposed susceptibility evaluation method, are shown in Figures 19, 20 and 21.

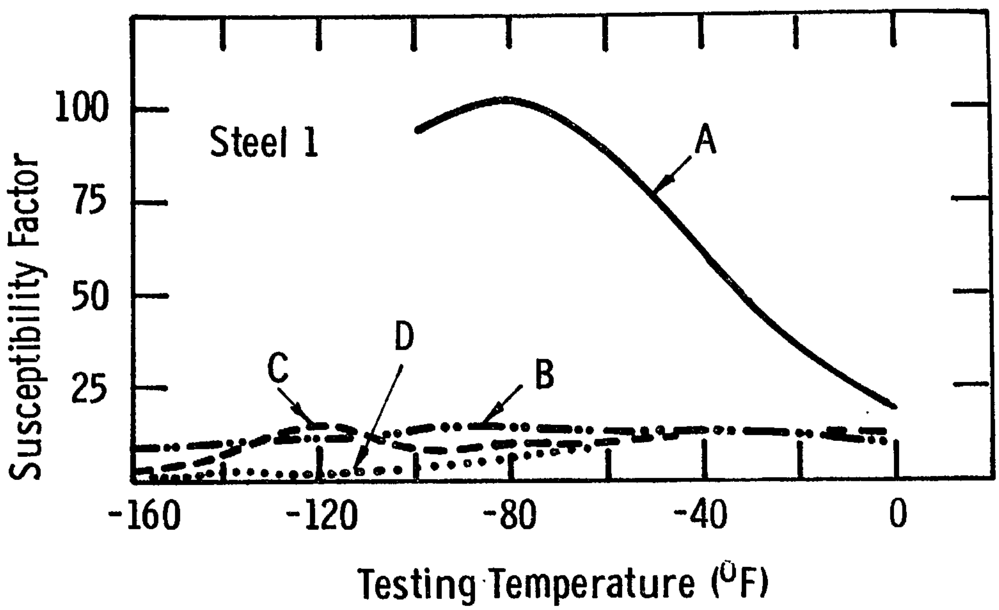


Figure 19. Susceptibility-factor curves for experimental steel Series 1.

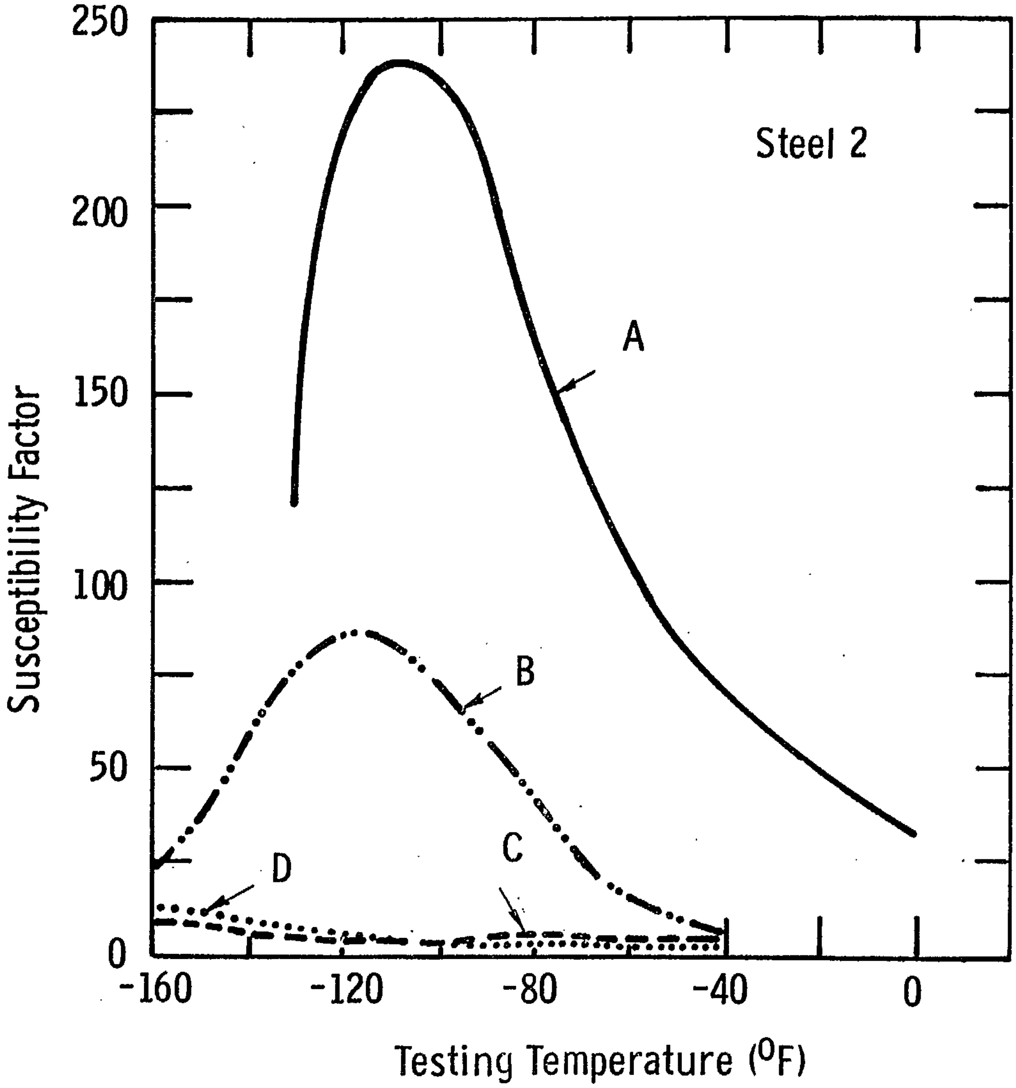


Figure 20. Susceptibility-factor curves for experimental steel Series 2.

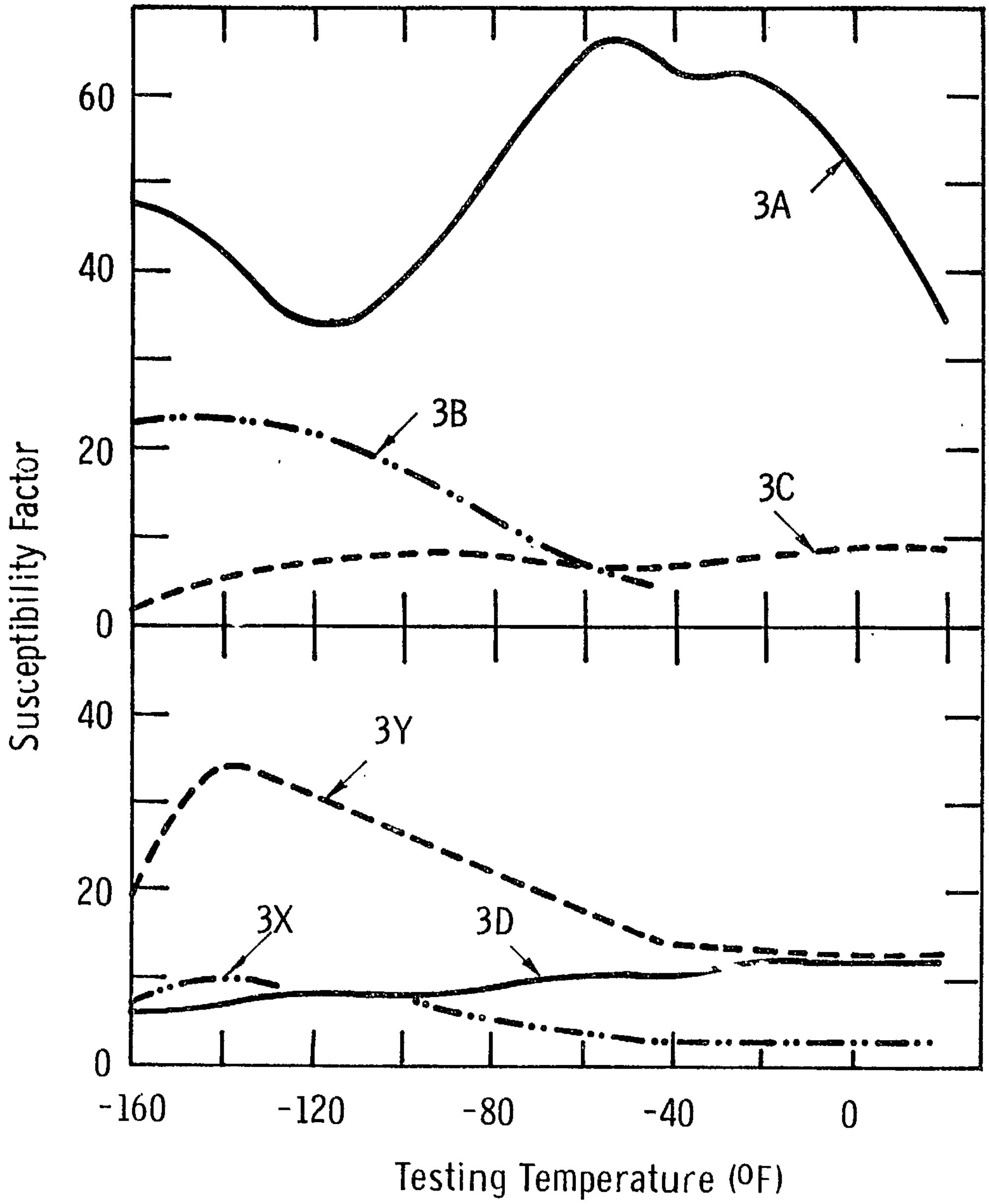


Figure 21. Susceptibility-factor curves for experimental steel Series 3.

For each of these steels the maximum-susceptibility factor (SF_{max}) was obtained from the curves. For each increment of molybdenum content over that of the base steel (residual), the percentage reduction of SF_{max} over that of the base steel was obtained. Figure 22 shows a plot of the average percentage reductions in SF_{max} at the various molybdenum levels.

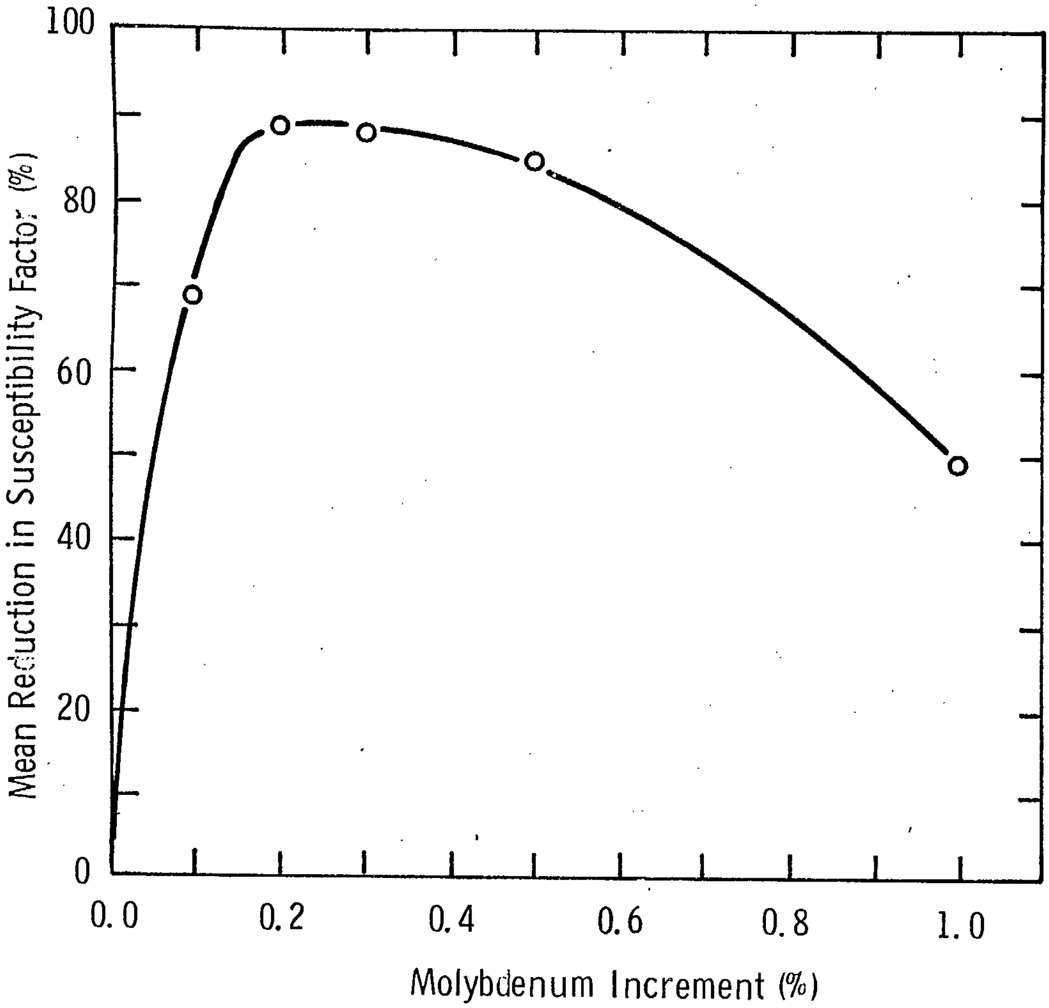


Figure 22. Average percentage reduction of SF_{max} for various increments of molybdenum content.

The higher level of susceptibility apparent for Steel 2A as compared with Steels 1A and 3A would appear to be a reflection of the strong influence of the manganese content, and also to be in accord with Woodfine's (15) observation that aluminum additions to a nickel-chromium steel increase the embrittlement.

It is readily apparent that small additions of molybdenum result in marked improvements in the degree of susceptibility as measured by the susceptibility-factor concept. Considering, as a simplification, the percentage reduction in the SF_{max} value attendant upon the addition of various levels of molybdenum, an optimum content would appear to be of the order of 0.2 per cent, with relatively little variation from 0.15 per cent to 0.50 per cent. Beyond this level the degree of embrittlement appears to increase significantly.

On examination of the curves for Steel 1, it is seen that the initial increment of 0.1 per cent was sufficient to eliminate all but a small proportion of the embrittlement inherent in this composition. A further increment of 0.1 per cent apparently resulted in no further improvement on the basis of the SF_{max} value, but the temperature of occurrence of the SF_{max} was depressed a further 14 centigrade (25 fahrenheit) degrees; the first increment caused a depression of 7 centigrade (13 fahrenheit) degrees of the temperature for SF_{max}. It is apparent that the plateau of low susceptibility-factor values observed before the curves begin to rise to the SF_{max} peak is to some degree attributable to the depression in the maximum impact-energy level due to the embrittlement treatment.

Examination of the curves for Steel 2 reveals that the 0.1 per cent initial increment was not sufficient to overcome the higher degree of susceptibility of this composition. The susceptibility was virtually completely eliminated by the next 0.1 per cent increment. A similar improvement in the temperature of occurrence of the SF_{max} is observed for this steel as for Steel 1.

Steel 3 can be seen to have required 0.2 per cent molybdenum to remove virtually all the susceptibility tendency. An increment of 0.1 per cent did remove a good portion of the susceptibility and resulted in a significant improvement in the temperature at which the SF_{max} peak was observed. It can be seen that at the 1.0 per cent molybdenum level a significant percentage of the initial susceptibility has been restored, although at a lower temperature.

Conclusions

The use of the proposed susceptibility-factor concept has been demonstrated to provide a useful pictorial comparison of the results of different investigators. A number of known temper-embrittlement effects have been shown to be reflected in the appearance of the curves developed.

The re-evaluation of the current laboratory results by means of comparison of peak susceptibility factors led to the same general conclusions as by more standard evaluation techniques.

6. ACKNOWLEDGEMENTS

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