The creep and temper embrittlement of Hy-100 steel.

Courtney Jay Hill

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THE CREEP AND TEMPER EMBRITTLEMENT OF HY-100 STEEL

by

Courtney Jay Hill

A Thesis
Presented to the Graduate Committee
of Lehigh University
in Candidacy for the Degree of
Master of Science

in

Metallurgy and Materials Science

Lehigh University
1975
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This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science.

21 May 1975
(Date)

Professor in Charge

Chairman of Department
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ABSTRACT

The creep and temper embrittlement susceptibility of medium section (4 inch wall thickness) HY-100 quenched and tempered to 110 and 135 ksi yield strength was examined by simulating heat treatments with plates 1 1/2 inches thick. Embrittlement was produced by step cooling, isothermal aging at 750°F, and creep aging at 750°F. The severity of embrittlement was evaluated by measuring the shift of the midrange brittle to ductile transition temperature of Charpy V-notch specimens, as determined by the absorbed energy and the lateral expansion criteria. Uniaxial tension tests, performed on unnotched specimens at room temperature and notched specimens at -58°F, were also employed in an attempt to assess the influence of embrittlement on tensile properties. Optical and scanning electron microscopy were utilized to examine the microstructural and fracture characteristics of creep and temper embrittlement.

The overall impact transition temperature of the steel as quenched and tempered was quite low on the order of -30°F to -110°F, however the shelf toughness of the alloys was also low, about 35-45 ft-lb. Embrittlement of the quenched and tempered steel was revealed to be severe, on the order of 300°F for step cooling and 165°F as measured by isothermal aging for 5000 hours. However, the specific transition temperature change was influenced
by Charpy impact specimen size, the impact resistance criterion employed and the steel strength level. While step cooling provided an estimate of the maximum extent of embrittlement possible in this steel, it does not exactly reproduce actual service at 750°F, thus it probably is an overestimate of embrittlement in service. Step cooling also produced a deterioration in the shelf toughness that was not observed in the isothermally aged steel. De-embrittlement at 1050°F for 1 hour of the embrittled steels demonstrated the reversibility of temper embrittlement.

Creep embrittlement, characterized by a degeneration of impact properties at ambient temperatures and grain boundary void formation, was not observed. Rather, a delay of temper embrittlement, particularly at the lower strength level, was recorded.
INTRODUCTION

BACKGROUND

Low alloy high strength steels have been used in a variety of industrial applications involving elevated temperature exposure for over 50 years. In spite of their generally good service record, the long time application of these steels for this type of service has been of increasing concern in recent years since the possibility that metallurgical aging effects can change the mechanical properties of the material in service has become a demonstrated reality. In many instances, these changes are induced by the effects of temperature alone, but in others, plastic strain and/or environmental influences may be significant. Examples of such effects are decreased creep ductility or stress corrosion cracking in Cr-Mo oil refinery equipment, increases in strength and decreases in ductility in austenitic stainless alloys, and, most commonly, decreased impact toughness in heat treated steels due to temper embrittlement. This latter effect has been identified as a significant cause for concern in both the electric power generation and petroleum industries. The American Society for Testing and Materials, The American Petroleum Institute, and The Electric Power Research Institute have initiated important research programs in this area in recent years. Work sponsored by A.P.I. at Lehigh University established that, during the first year of service at temperatures of 800 to 900°F, the impact transition temperature
of 2 1/4 Cr - 1 Mo steel pressure vessels (ASTM A542) may increase as much as 150°F. As a result, the 70°F Charpy impact toughness may be in the range of 10 ft-lbs or less. This low toughness in the ambient temperature range has resulted in at least two cases of major cracking, necessitating expensive repairs in refinery vessels in both the United States and Japan. In addition, there is at least one instance of a major failure in power generation equipment in Great Britain that was, in part, attributable to the low toughness induced by temper embrittlement. It is prudent, therefore, that users of low alloy steels which operate in the elevated temperature range consider the effects of service environments on the static ambient and elevated temperature properties of these steels.

It is the purpose of this research to develop precisely such information on the effects of a moderate service environment on the toughness of HY-100 type (Ni-Cr-Mo) steel after an extended period of service. This type of steel is used as plates (HY-80, HY-100, ASTM A543), forgings (A508 Class 4), and other product forms in a number of pressure vessels including nuclear pressure vessels. The particular application of interest in this program is service in intermediate thickness (4 inch wall thickness) forged vessels used to grow quartz single crystals in caustic solution. The temperature of service in this case is up to approximately 750°F, and pressures at this temperature are relatively
high. Although pressures in the ambient temperature range are low, the relatively high pressures in service dictate that these steels, for design purposes, be heat treated to high strength levels. While, indeed, the service environment is only moderate with respect to those which produce normal temper and creep embrittlement effects, the long times of service (greater than 50,000 hours) make such effects a possible source of embrittlement. For this reason, a three phase study of HY-100 Ni-Cr-Mo steel at 110 and 135 ksi yield strength levels to isothermal embrittlement was performed. The three phases include step cooling and isothermal aging and creep testing at 750°F.

Along with this experimental program, a literature review of this topic was also undertaken. This literature review is presented in the next sections of the Introduction to this report.

TEMPER EMBRITTLEMENT LITERATURE REVIEW

Although long recognized to be of significant metallurgical importance—the first published reference appeared in 1917(1)—the theory of temper embrittlement has eluded complete development. The problem approached major proportions during the concerted effort to utilize alloy steels in World War I. In particular Krupp of Germany produced a Ni-Cr armor steel which, in heavy sections, was extremely susceptible to brittle failure. (2) The manifestations of temper embrittlement were further demonstrated in 1954 when several large steam turbine generator rotors, within
a period of ten months, experienced catastrophic failure.\(^{(3,4)}\)

In 1969, yet another steam turbine rotor failed catastrophically\(^{(5)}\).

Each of these failures, to some extent, was attributable to temper embrittlement. Failure caused by temper embrittlement has also been experienced in the petro-chemical industry and is of vital concern to operators of any large, heavy section chemical pressure vessels.

The long history of failures, in spite of extended and extensive metallurgical research, indicates the complex and perplexing nature of the temper embrittlement phenomenon. In general, it is a phenomenon that, although well characterized, is not well understood. The extent of the present understanding can best be presented by listing the prominent characteristics of temper embrittlement. This list represents a modification and extension of that developed by Emmer, Clauser and Low.\(^{(6)}\)

**Mechanical Properties**

1. Temper embrittlement is most commonly characterized by a reduction of impact resistance, and is usually described by the shift in the ductile to brittle transition temperature of the steel.\(^{(7,8,9)}\)

2. Embrittled steels are sufficiently tough that an impact test is usually required to detect the phenomenon.\(^{(10)}\)

3. Shelf energies are not affected by isothermal embrittlement. However, a step-cooling assess-
4. Embrittlement does not affect those mechanical properties of the material usually determined by the tension test. It may, however, affect flow strength. (11)

5. Temper embrittlement does not affect the endurance limit of the material, although it does cause a reduction of fatigue strength above it. At high stress levels this effect is not pronounced. (12, 13, 14)

6. The rate of fatigue crack propagation increases with the degree of embrittlement. (14)

7. Materials that are susceptible to temper embrittlement are also susceptible to hydrogen embrittlement. (15)

**Kinetics**

1. Temper embrittlement occurs in the temperature range of 700-1000°F (2, 16, 17, 18) and exhibits a typical C-curve time-temperature relationship within this range.

2. At low temperatures within the embrittling range, the initial rate of embrittlement is slow, while the maximum embrittlement and the time to achieve it are increased. (19)
3. After an extended period of time, the rate of embrittlement is reduced. \((7,8,20,21)\)

4. Overaging appears to occur at temperatures within the embrittlement range somewhat above the nose of the C-curve. However, this apparent overaging must also take into account the tempering of the material that occurs. \((8,16,20-24)\)

Alloy effects

1. The susceptibility of a steel is influenced by its alloy content.

2. Plain carbon steels are not observed to embrittle when subjected to an embrittling treatment. \((7,22,25)\)

3. Cr, Ni and Mn increase susceptibility to temper embrittlement. However, specific effects are determined by the interaction between these and other alloy or impurity elements present in the steel. \((8,22,25,26)\)

4. Mn and Cr individually must be present in a minimum concentration in order to induce temper embrittlement. This minimum concentration is influenced by the specific impurity element concentration of the alloy. \((2,22,26)\)

5. Susceptibility of a steel is related to hardenability with respect to Ni, Cr and Mn. This
effect is amplified by the fact that these elements tend to strengthen the grain relative to its boundary, thereby shifting the fracture transition temperature to higher temperatures. (7)

6. In alloy steels, lowering the carbon content decreases the degree of embrittlement. This effect is obscured by the fact that reducing the carbon level of the steel decreases the transition temperature in the unembrittled condition. A complete absence of carbon, however, does not prevent embrittlement. (22, 25)

7. Embrittlement occurs only in the presence of specific impurity elements. These are Sb, P, Sn, and As in order of decreasing severity on a weight basis. Bi, Se, Ge, V, and Te have also been demonstrated to promote temper embrittlement. (22, 25, 27-30)

8. In addition to promoting temper embrittlement, the addition of P and Sb to alloy steels causes an increase in the transition temperature of the unembrittled material. (30, 31)

9. Controlled amounts of Mo, W, and Ti suppress both the rate and the extent of temper embrittlement. (20, 28, 52)

10. Mo retards both embrittlement and de-embrittlement. (20)
11. Mo and W must be present in a minimum concentration in order to suppress embrittlement. A deviation from this amount will reduce their effectiveness and may even result in increased brittleness. (20)

12. Alloy and impurity interactions have a substantial effect on temper embrittlement. V and Mo alone, for example at a concentration of 1%, have been shown to induce embrittlement. However, a 1% Mo - 1% V addition decreases susceptibility. (20,55)

Segregation

1. Both impurity elements and alloying elements are concentrated along prior austenite grain boundaries during embrittlement. (32-37) However, segregation has been shown to occur along all high angle grain boundaries of an embrittled steel. This segregation is significant when the angle of grain boundary mismatch is greater than 15 degrees. (19,37,38)

2. Cr, Ni, and Mn segregate to an appreciable extent only in the presence of minor impurity elements. (53,54)

3. The depth of impurity segregation in an embrittled steel is on the order of 5 to 10 A.
The segregation profile of alloy elements tends to be more diffuse, approximately 50 Å.\(^{(31)}\) The concentration of the segregated species is reported to be at least five times the bulk concentration.\(^{(31,33,40)}\)

4. Segregation does not occur in the austenite field but in the ferrite plus carbide field. Segregation in the ferrite phase is also reversible in the ferrite phase.\(^{(19,31)}\)

5. There is no immediate correlation between the amount of segregation and the degree of decohesion along grain boundaries.\(^{(19)}\)

**Reversibility**

1. Temper embrittlement may be reversed by heating above the embrittling temperature range.\(^{(20,32)}\)

2. The kinetics of the de-embrittlement process are influenced by alloy composition and temperature. The addition of Mo, for example, suppresses the rate of reversibility as well as the rate of embrittlement.\(^{(20,32,52)}\)

3. Higher temperatures permit shorter times for de-embrittlement.\(^{(32)}\)

**Surface effects**

1. In general, embrittlement is characterized by a change in fracture mode from trans- to intercrystalline.\(^{(8,37)}\) This does not require
an associated large shift in the ductile to brittle transition temperature of the steel.\(^{25}\) However, both the percent intergranular fracture and the percent grain boundary decohesion, as well as the numerical shift in transition temperature, have been employed to assess the extent of embrittlement.

2. The grain boundary facets which characterize the fracture surfaces of embrittled steels are indistinguishable from those observed on the unembrittled material. However, their chemistry is quite different.\(^{32}\)

3. In low alloy steels, fracture is observed to occur along prior austenite grain boundaries. In carbon free alloys fracture proceeds along ferrite-ferrite grain boundaries.\(^{37}\)

4. Temper embrittlement may sometimes be deduced in tensile specimens by a radial crack pattern along the fracture surface.\(^{2,30}\)

5. In steels containing chromium and phosphorous, an etheral picric acid etch will preferentially attack prior austenite grain boundaries.\(^{41}\)

**Heat treatment**

1. The upper shelf fracture mode of some temper embrittled steels is sensitive to prior heat treatment. In some 3.5% Ni - 1.7% Cr based steels,
a step austenitizing treatment prior to embrittlement results in a reduction of the upper shelf and a change from intergranular fracture to intergranular microvoid coalescence. The transition temperature, however, is unaffected by this treatment. (42)

2. Prolonged heating at a temperature above the embrittling range inhibits subsequent temper embrittlement by reducing the embrittling rate. (8,30,32)

3. Intercritical heat treatment has been observed to reduce susceptibility to temper embrittlement by manipulation of the microstructure such that the interacting alloying and impurity elements are morphologically separated. (43)

Microstructural effects

1. Susceptibility to temper embrittlement is influenced by hardness but these effects are not entirely clear. Steels possessing higher yield and tensile strengths, (i.e., in the quenched and tempered condition rather than a softer one), will more severely embrittle. However, for steels with approximately the same microstructure, the higher strength steel appears to be embrittled less than the lower strength one.

2. Martensitic steels undergo a larger shift in transition temperature than do bainitic steels.
Bainitic steels, in turn, are more susceptible than pearlitic steels. The embrittled transition temperature, however, may be independent of structure. The different shifts result from differing initial unembrittled conditions. (8,26,56)

3. Increased austenitic grain size results in increased embrittlement. (7,44,45,46)

4. Plastic deformation prior to embrittlement reduces the susceptibility of quenched and tempered alloy steels. Plastic deformation during embrittlement appears to delay the development of temper embrittlement. The extent of embrittlement, however, may be increased by the initiation of creep embrittlement. Plastic deformation subsequent to embrittlement lowers the transition temperature of the steel. (47,48,49)

From examination of this list it is understandable that, at present, no well formulated model for the temper embrittling process exists. While it is clear that impurity and alloy segregation play a major role in the process, it is not clear to what extent each of these is responsible or in what manner these species become segregated in the steel.

While recent results indicate that segregation occurs during embrittlement in the ferrite phase, and does not proceed by any mechanism requiring pre-segregation during austenitizing, the
question of how these elements concentrate along high angle grain boundaries remains unanswered. It is currently believed that this occurs by either equilibrium segregation or by alloy rejection during precipitation at the grain boundaries. The difficulty in justifying both of these mechanisms is that those characteristics of temper embrittlement which are exploited to defend one model may also be suitably tailored to support the other. Specifically, the kinetics, the reversibility, the segregation and alloy effects may be the consequence of either an equilibrium segregation or a precipitation mechanism.

CREEP EMBRITTLEMENT LITERATURE REVIEW

To present, creep embrittlement has not received the metallurgical attention reserved for temper embrittlement. However, it, too, has long been recognized to have profound effects upon ultimate material performance.

Creep embrittlement became recognized as a phenomenon of consequence when boiler flange bolts manufactured of heat resisting steel experienced failure within several years of service. Specifically, it had been found that vessels operating in a temperature range of 850 to 950°F experienced catastrophic failure at the bolts within a service period of 3000 to 12,000 hours, depending upon the composition of the steel and the loading stresses.

Creep embrittlement must also be considered in those applications for which temper embrittlement may be significant. For, in general, those materials which are susceptible to temper embrit-
tlement in service are, under particular conditions of temperature and stress, likely to exhibit creep embrittlement. Hence, manufacturers of large pressure vessels and petro-chemical equipment such as hydrocracking towers and reformers must also consider creep embrittlement during design and material evaluation.

Presented below are the most prominent characteristics of creep embrittlement.

**Mechanical properties**

1. Creep embrittlement is accompanied by the reduction, and subsequent partial recovery of stress rupture ductility. \(^{(63,64)}\)

2. Associated with creep embrittlement is the loss of notch ductility and an increase in the brittle to ductile transition temperature of the steel. \(^{(61,62)}\)

3. A reduction of the upper shelf energy has also been reported to be a consequence of creep embrittlement. Hence embrittlement may be characterized by a reduction in the ambient temperature impact resistance of the steel. \(^{(62)}\)

**Kinetics**

1. In ferritic steels, creep embrittlement occurs in the temperature range of 800 to 1100°F. \(^{(65-69,73)}\)

2. At lower temperatures within the embrittling range, the rate of creep embrittlement diminishes.
However, susceptibility increases and embrittlement maximizes after longer exposures. (65,70-72)

3. The most severe conditions for a given material are those which produce a small amount of creep after an extended period of time. (65,67,70,72)

Alloy effects

1. There is some evidence that the temper embrittlement inducing elements may also contribute to the creep embrittlement of steels. (57,73)

2. In general, those materials susceptible to temper embrittlement are also likely to exhibit creep embrittlement under the appropriate service conditions. (60-62)

Reversibility

1. Creep embrittlement, to distinguish it from temper embrittlement, is irreversible by subsequent heat treatment. However, some improvement of the transition temperature may be produced by tempering effects. In effect, the void formation associated with creep embrittlement is irreversible. (62)

Surface effects

1. Creep embrittlement is accompanied by a transition from a transgranular to intergranular mode of fracture. (73)
Microstructure effects

1. Creep embrittlement is characterized by the formation of small voids and microcracks along prior austenite grain boundaries. These are commonly observed to be formed transverse to the tensile direction.\(^{(62,64)}\)

2. For a tempered martensitic structure, susceptibility is observed to be greater at higher strength levels.\(^{(65,67)}\)

3. Coarse grain materials are more susceptible than fine grain ones.\(^{(65,67)}\)

The mechanism by which creep embrittlement proceeds is intimately associated with the specific carbide precipitation processes and kinetics that occur under the specific conditions of stress and temperature. During exposure to elevated temperatures, carbides are precipitated at austenite grain boundaries. Simultaneously, alloy carbides are formed within each grain and tend to dispersively strengthen it. However, it is observed that those carbides which are precipitated near grain boundaries tend to dissolve and reprecipitate on the complex grain boundary carbides. As a result a thin, weak denuded zone is formed. The combination of a strong grain bulk, a weak grain boundary region, and a stress concentrating grain boundary precipitate promotes intergranular void formation under an applied stress which eventually leads to failure. Consequently, the susceptibility of a
steel to creep embrittlement is determined by the manner and extent
to which the strength of the grain interior is increased by the
dispersion of incoherent carbide precipitates relative to the
weakening of the grain boundary region by denudation and stress
concentrating grain boundary precipitation. When the strength of
the individual grains is enhanced to such an extent that bulk
deformation is preceded by grain boundary sliding and void for-
mation, embrittlement results and the fracture mode shifts from
trans- to intergranular. The precipitation process during creep
embrittlement not only acts to promote intergranular failure but
also prohibits the normal accommodation of strain under creep
conditions, such as bulk deformation, climb, and grain boundary
migration.

Roper\(^{(66)}\) summarizes these individual contributions as
follows:

1. The fine dispersion of carbide precipitates within the
grain interiors produces significant resistance to bulk
deformation of the individual grains during creep.
2. The increasing width of the denuded zone with time at
temperature lowers creep strength near the grain bound-
aries.
3. The formation of grain boundary carbides reduces the
cohesive strength of the boundaries and the combination
of hard particles in a soft matrix, as stress concen-
trators, initiate voids.
EXPERIMENTAL PROCEDURE

INTRODUCTION

In order to ascertain the susceptibility to temper and creep embrittlement of heavy section commercial grade HY-100, it was necessary to simulate the microstructure of a heavy plate given an industrial heat treatment, using a smaller plate thickness that could be treated in the laboratory. In this case, the heat treatment of a 4-inch thick water quenched plate was simulated with one 1.5 inches thick to obtain 1) the desired center half-time-temperature cooling rate upon quenching from the austenitizing temperature, and 2) the desired yield strength levels in the tempered condition, 110 and 135 ksi. Heat treated specimens were subsequently embrittled isothermally at 750°F and by a standard step cooling process. Standard Charpy impact, uniaxial room temperature tensile, and low temperature notched tensile tests were performed on the aged material to assess the extent of embrittlement. Reversibility of the temper embrittlement was emphasized by de-embrittlement of some isothermally aged and step cooled specimens.

To investigate the effect of an applied stress on the embrittlement process, creep specimens were loaded to one half their quenched and tempered yield strength at 750°F. Subsequently, Charpy data was accumulated by utilizing friction welded subsize (5 by 5 by 55 mm) V-notch impact specimens.
Finally, to further characterize the microstructural ramifications of temper and creep embrittlement in HY-100, standard metallography and scanning electron metallography were performed.

MATERIAL AND HEAT TREATMENT

The material used in this investigation was HY-100 quenched and tempered steel in the form of a 23\" dia. forging cut into 1 1/2 inch slices. The material was obtained from Earle M. Jorgensen Co. and the composition is listed in Table 1. To reproduce the water quenching of a 4-inch plate steel in a 1.5-inch plate, a simulation procedure based on cooling rates was employed. Although previously used at Lehigh in several programs, the effect of various quench media on the cooling rate of 6 in. by 10 in. by 1.5 in. HY-100 plate was again explored to verify previous work and to establish the exact procedures necessary to heat treat the steel in this program. The cooling rates were determined by inserting three shielded chromel-alumel thermocouples into the center thickness of the test plate, austenitizing for 1 hour at 1575°F, and independently recording the subsequent cooling process with three strip chart recorders (see Figure 1). Variations of cooling rate, as measured at each couple position, were essentially negligible. This procedure was repeated for a series of coolants to determine which would reproduce the 4 inch cooling rate in the 1.5-inch plate most closely. The results of this study are discussed later.
To obtain the desired strength levels, 110 and 135 ksi quenched and tempered yield strengths, it was necessary to identify the tempering dependence of the mechanical properties of HY-100. This time-temperature relationship was accomplished by tempering 1 in. by 10 in. by 1.5 in. sections for 1, 2, 5, 10 and 30 hours at 1100°F and performing an identical treatment at 1200°F. Following the temper, Brinell hardness tests, utilizing a 3000 kg. load and tensile tests, were performed. As is described later, from these tempering relationships, the appropriate heat treating times and temperatures were determined. This determination was substantiated by mechanical testing of material receiving these treatments.

STEP COOLING

An immediate assessment of the susceptibility of HY-100 to temper embrittlement was obtained by utilizing the following step cooling treatment on 6 in. by 5 in. by 1.5 in. plates at each strength level:

Holding 1 hour at 1100°F followed immediately by

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<tr>
<td>15 hours</td>
<td>1000°F</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
<tr>
<td>24 hours</td>
<td>975°F</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
<tr>
<td>48 hours</td>
<td>925°F</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
<tr>
<td>72 hours</td>
<td>875°F</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
</tbody>
</table>

air cooling to room temperature.

Treatment was done in a forced air furnace and temperature measurements performed during the treatment demonstrated that the
time between each cooling step was a small fraction of the total time at any given temperature as outlined by the required treatment.

ISOTHERMAL AGING

Isothermal aging was accomplished by subjecting 6 in. by 5 in. by 1.5 in. plates at each strength level to 750°F for 1, 10, 30, 100, 300, 1000, 2000 and 5000 hours in a forced air furnace. Specimens were immediately water quenched from the aging temperature.

CREEP EMBRITTLEMENT

To evaluate the influence of the addition of stress to the susceptibility of HY-100 to embrittlement, 0.375 inch diameter, 6 inch gage length creep specimens were subjected to a stress level equal to one half the yield strength for 1000 hours at 750°F. This procedure was repeated for each strength level.

Subsequently, impact specimens were prepared by cutting 1 inch sections from the gage and friction welding each of these between two 1-inch lengths of 1020 steel bar stock (see Figure 2). These welded assemblies were then ground and milled into 5 by 5 by 55 mm Charpy V-notch impact specimens (these are subsize specimens machined according to ASTM E23 specification). This procedure was followed in order to conserve test material and to reduce the time necessary to obtain a sufficient number of Charpy specimens from the crept material to determine a full impact curve. Since only the material in the vicinity of the notch is actually
tested, the sectioning and welding procedure remains valid as long as the welding process does not alter the material near the notch. For this reason, friction welding, characterized by a small heat affected zone, was employed to fabricate the assemblies. This procedure has been used in previous studies at Lehigh University.60

Because such a small notch (0.39 inch deep) is required in the subsize Charpy specimens that were used, notch uniformity is essential. This was verified by comparing the profile of optically magnified notches, and measuring the width of each notch at the specimen surface to determine variability.

Since specimen size effects are important in impact tests, subsize Charpy specimens of the quenched and tempered and of the step cooled material were also prepared. In this manner, the impact data accumulated from creep embrittled specimens could be directly compared to unembrittled and step cool embrittled material of the same specimen size.

MECHANICAL TESTING

Mechanical test specimens were cut from the center thickness of the test plates, and surface layers were discarded. A schematic diagram of the orientation of the test specimens is found in Figure 3.

Tensile Tests

Tensile testing was performed at room temperature on 0.252 inch diameter specimens. All tests were performed at a
strain rate of 0.02 in/min. Elongation prior to yielding was measured with an extensometer. A second set of specimens was prepared with a 0.05 inch deep, .005 root radius notch at the center of the gage length. These were tested at -58°F also at a strain rate of 0.02 in/min. Data obtained from these tests included yield load, as determined by a 0.02% offset, tensile load, fracture load, reduction in area, and percent elongation. Yield, tensile and fracture loads were subsequently converted to the appropriate values of engineering stress.

**Impact tests**

Chapry V-notch impact curves were employed to determine the effect of embrittlement on the upper shelf energy and the transition temperature of the quenched and tempered material. To develop full curves, both high and low temperature testing was required. Low temperatures were obtained by utilizing liquid nitrogen to cool a 2-methylbutane bath. Heated water was used to obtain temperatures from room temperature to 150°F. Boiling methanol-water solutions were found to produce stable temperatures from 150 to 212°F. For higher temperatures, heated oil was employed. All specimens were immersed in the appropriate bath for a minimum of 5 minutes before testing. Subsequently, fractured specimens were immediately submerged in acetone to prevent frost formation on low temperature specimens and to remove the oil from the very high temperature specimens. Fracture surfaces were then dried in air and immediately sprayed with an
acrylic coating to protect them from contamination. For each set of specimens, the characteristic transition temperature and shelf energies were determined from curves fitted with the aid of a special computer program developed and on file at Lehigh University.

**Metallography and Surface Preparation**

To examine the microstructural effects of creep embrittlement on HY-100, samples of the quenched and tempered and creep embrittled material were given a standard metallographic polish. Creep specimens for metallographic examination were sectioned longitudinally prior to examination.

The particular metallographic procedure employed required the mounting of specimens in bakelite before polishing. Initially, mounted samples were ground on an 80 grit wet belt sander to develop a flat surface. To produce a final polish acceptable for examination at high magnifications, surface preparation was completed with 6 micron and 1 micron diamond paste and a quick buff on the Linde B (0.06 micron alumina) polishing wheel. Etching was performed by swabbing each polished specimen with 2% nital for approximately fifteen seconds. Typically, the surface was lightly repolished with Linde B and re-etched. This procedure was repeated until a satisfactory surface could be obtained.

Examination of the prepared specimens in the optical microscope was not entirely satisfactory because the fine structure could not be resolved. Consequently, microstructural analysis was completed with the aid of the scanning electron microscope.
Fracture surfaces did not require any special surface treatment prior to examination with the scanning electron microscope. However, stripping of the protective coating, degreasing, and cleaning were performed by immersing the fracture surfaces in acetone and ultrasonically cleaning them for 15 minutes.

**Scanning Electron Microscopy**

Scanning electron microscopy was utilized to examine fracture surface appearance of Charpy impact and tensile specimens. Specifically, each of the following were inspected under the 20 KV electron beam and photographed on Polaroid PN Type 55 film.

1. Unnotched tensiles - unembrittled and temper embrittled
2. Notched tensiles - unembrittled and temper embrittled
3. Upper shelf Charpy - unembrittled and progressively embrittled
4. Lower shelf Charpy - embrittled
5. Lower shelf Charpy - de-embrittled
6. Upper shelf Charpy - creep embrittled

In addition, microstructural information was acquired from polished and etched specimens of HY-100 in the unembrittled and creep embrittled conditions.
RESULTS AND DISCUSSION

HEAT TREATMENT

An examination of Figure 4 illustrates that to simulate the cooling characteristics of a water quenched 4 inch plate, a center cooling rate in a 1.5 inch plate between approximately 1.8 and 3.1°F/sec is required. To produce this rate, several coolants were tested. Presented in Table 2 are the center thickness half-time-temperature cooling rates derived from the various quench media employed. It is evident from these data that the most satisfactory results were achieved by forcing room temperature air over the plate faces with high capacity rotary fans. During the course of experimentation, it was determined that while the desired simulated cooling rate could be approached by placing one fan perpendicular to each side of the austenitized plate at a distance of 8 inches from the surface, it was possible to obtain slightly faster cooling by employing two additional fans. Utilizing this procedure, a consistent, reproducible value of 1.8°F/sec could be achieved for the half-time-temperature cooling rate.

One of the interesting conclusions reached from examination of the possible quenchants was that only slight variations in cooling rate could be obtained by specific modifications to the quench medium. Substantial changes could only be produced by replacing the medium itself. For example, by employing heated rather than room temperature oil as the coolant, only a 1.74°F/sec
change in cooling rate could be obtained. By increasing the flow rate of air over the plate surfaces from normal circulation a maximum cooling rate increase of only 0.88°F/sec was possible. This may be compared to the 8.22°F/sec increase achievable by substituting an oil quench for air cooling.

The further heat treatment required to obtain the base (un-embrittled) condition of the HY-100 was established by examining the tempering characteristics of the mechanical properties. This dependence is summarized in Figure 5 for hardness and Figure 6 for yield and tensile strength. In Figure 7, a widely used tempering parameter is used to summarize the hardness data. In Figure 8 the correlation between the various mechanical properties produced by these treatments is illustrated. With the aid of this information, it was concluded that the desired yield strengths of 110 and 135 ksi could be obtained by tempering the quenched material for 6 1/2 hours at 1200°F and 1100°F respectively. Tensile tests performed on material receiving this treatment demonstrated that, indeed, this temper was satisfactory. The tensile properties of the unembrittled steel are presented in Table 3, and the toughness properties of the steel are shown in Table 4.

While examining these data, it should be noted that this material, while it has a low as-tempered mid-range transition temperature, also has very low ambient shelf toughness. The 1200°F tempered material has a shelf toughness of 48 ft-lb, while
the 135 ksi yield strength material has a shelf toughness of only 33 ft-lb. These values are low for materials in normal pressure vessel service. On the basis of typical fracture toughness characterizations, this material would not be expected to tolerate cracks larger than about 6 in. long and 1.5 in. deep (i.e., not through wall cracks) without high potential for fast fracture. Thus 4 inch thick pressure vessels of this steel operated at one-third of the tensile strength would not meet leak-before-break fracture criteria.

TEMPER EMBRITTLEMENT

A summary of the information extracted from the impact testing of the unembrittled and step cooled HY-100 is furnished in Table 4. The specific impact curves involved, computer generated from the energy absorbed and lateral expansion data, are displayed in Appendix A, Figures A1-A4. From an examination of these results, two important conclusions may be drawn. It is apparent that the higher strength material, though exhibiting a higher transition temperature in the unembrittled condition, is less susceptible to temper embrittlement as measured by the shift in the ductile to brittle transition temperature. This finding has been reinforced by investigations of other low alloy steels performed at Lehigh. (60)

Secondly, during the step cool embrittlement that is required to obtain an immediate evaluation of the susceptibility of the
material, a discernible drop in the upper shelf toughness as measured by both the energy absorbed and lateral expansion is evident. The deterioration of high temperature impact resistance, was also perceivable from the evaluation of subsize Charpy tests.

The influence of the kinetics of temper embrittlement on impact properties is depicted in Figures 9 and 10 and in Table 5. The accompanying impact curves are reproduced in Appendix A, Figures A5 through A20. It is apparent from this information that, at 750°F, the degeneration of impact resistance, characteristic of temper embrittlement, is slow to develop. Only a slight embrittlement, a 30°F shift or less, is produced after 1000 hours at 750°F at both strength levels. In addition, consistent with the step cooling results, isothermal aging demonstrates that the 135 ksi yield strength material is less susceptible to temper embrittlement than the 110 ksi yield strength material.

Figures 9 and 10 illustrate that not only is embrittlement a slow process at 750°F, it is preceded by a modest decrease in the transition temperature. This characteristic, also recognized in earlier studies,\(^{(61)}\) suggests that while embrittlement is initially sluggish, other processes resulting in slight toughening will occur. However, at the higher embrittling rates which accompany exposure to higher temperatures, on the order of 900°F, or after prolonged subjection to lower temperatures, greater than 1000 hours at 750°F, this toughening is overwhelmed by temper embrittlement.

Comparison of the step cool and isothermally embrittled
impact data demonstrates several important characteristics of these particular aging treatments. Although step cooling permits an immediate evaluation of the severity of embrittlement in a specific steel, it does not provide information concerning the rate of embrittlement at a particular temperature, in this case 750°F. What it does provide is an identification of the relative maximum degree of embrittlement likely to be encountered in a material, regardless of the service temperature or exposure time. Consequently, isothermal data, in conjunction with step cooling information, illustrates that at the higher strength level, not only is the susceptibility (maximum embrittlement) of the steel reduced, the rate at which that maximum is reached is also reduced.

While step cooling accelerates the rate and degree of embrittlement, it also influences the upper shelf of the Charpy impact curve. Although the step cooling tests would indicate a decrease in the toughness of HY-100 in the ductile temperature regime, isothermally treated material exhibits no distinct trend (see Figures 11A and B). Hence, utilization of a step cooling treatment to assess susceptibility may also produce an artificially poor value of the upper shelf. The cause for the loss in shelf toughness on step cooling is not entirely clear. It is known that this steel can undergo several types of aging phenomena in the 900 to 1000°F range included in the step cool thermal cycle but not encountered in 750°F aging. Thus step cooling is apparently not a good predictor of 750°F embrittlement phenomena for the steel.
De-embrittlement at 1050°F for 1 hour of the embrittled steel demonstrated the reversibility of temper embrittlement. As Table 6 indicates, not only is the extent of embrittlement less at a strength level of 135 ksi, the extent of the recovery of impact resistance is greater for a given time at temperature. Nevertheless, de-embrittlement was considerable for both the 110 and 135 ksi yield strength material.

Confirmation of temper embrittlement by the evaluation of tensile properties was also attempted. It was determined, however, that the properties of unnotched specimens tested at room temperature were unaffected by embrittlement (see Table 7). Hence, the conventional tension test cannot be utilized to assess susceptibility. Similarly, it was found that the mechanical behavior of notched specimens subjected to -58°F was not affected (see Table 6). What was observed was a distinct change in the appearance of the fracture as embrittlement became more pronounced. Scanning electron microscopy provided corroboration of this change.

CREEP EMBRITTLEMENT

Before an evaluation of the influence of an applied load on the embrittlement at 750°F could be performed, it was necessary to reproduce the impact data of the unembrittled and step cooled material. With these data, it was not only possible to compare the impact toughness of the strained HY-100 directly to the base condition by utilizing subsize specimens, but the effect of speci-
men size on the apparent shift in transition temperature could be determined.

For subsize specimens, the small range of energy absorbed on impact (0 - 10 ft-lb) did not facilitate an accurate determination of the mid-range transition temperature. Consequently, to evaluate the impact data accumulated by testing smaller Charpy specimens, only lateral expansion data were employed.

The mid-range lateral expansion transition temperature changes that are associated with embrittlement are presented in Table 8. Corresponding impact curves are displayed in Appendix B, Figures B1 through B6. It is evident from this information, that while the strength dependence of embrittlement is again emphasized, the extent of temper embrittlement was found to be substantially less than that obtained from the standard size specimens. In particular, less than one half the degree of embrittlement seen in the larger specimens was evidenced.

Creep embrittlement, characterized by a decrease in the upper shelf and the associated increase of the brittle to ductile transition temperature, was not observed. While creep strain may induce embrittlement, it will also defer the initiation of temper embrittlement. This retardation is particularly evident in the HY-100 treated to 110 ksi yield strength in the unembrittled condition. Rather than a delay of embrittlement,
there was an actual improvement of the material as characterized by a substantial decrease in the transition temperature.

OPTICAL AND ELECTRON MICROSCOPY

**Metallography**

To reinforce the interpretation of the mechanical tests, optical metallography was performed. A typical optical micrograph of quenched and tempered HY-100 is presented in Figure 12A. Its scanning electron counterpart is shown in Figure 12B. This tempered bainitic structure is not only representative of unembrittled HY-100, but is also illustrative of the temper embrittled and creep strained steel. Grain boundary void formation at prior austenite grain boundaries, indicative of creep embrittled steels, was not observed. Thus, the conclusion that creep embrittlement, characterized by a reduction of the upper shelf and increase in transition temperature, did not occur was supported metallographically.

**Electron Fractography**

Figure 13 displays a fracture surface common to HY-100 in the brittle condition. Intergranular fracture and secondary cracking are both evident. The effect of the de-embrittling treatment is illustrated in Figure 14. As a result of heat treatment at 1050°F for 1 hour, the faceted fracture appearance is replaced by microvoid formation and a substantial increase in impact resistance.
In an attempt to understand the upper shelf drop that was characteristic of the step cooling treatment, several fracture surfaces of Charpy specimens broken in the ductile temperature region were examined in the unembrittled, step cooled, and isothermally aged conditions. However, by observation with the scanning electron microscope, no dissimilarities in fracture appearance, typically dimpled, were discerned.

Fractographs of temper embrittled notched and unnotched tensile specimens are presented in Figure 15. While the unnotched specimens possessed a dimpled rupture surface after significant plastic deformation, the notched specimens tested at a low temperature showed evidence of secondary grain boundary fracture superimposed upon the dimples. At the strain rates employed in these tests, however, no significant difference in fracture strength was observed between these two samples.
CONCLUSIONS

1. By step cooling, severe embrittlement, on the order of a 300°F shift in the mid-range transition temperature, was produced in the HY-100 quenched and tempered to 110 ksi yield strength. This shift was determined to be approximately 150°F in the 135 ksi yield strength material.

2. After isothermal aging at 750°F for 5000 hours, shifts in the transition temperature of approximately 165°F and 100°F were observed in the 110 and 135 ksi yield strength material, respectively.

3. The superposition of creep stress equal to one-half of the quenched and tempered yield strength upon isothermal aging at 750°F did not induce creep embrittlement after 1000 hrs. It did delay the initiation of temper embrittlement.

4. HY-100 quenched and tempered to a higher yield strength, while possessing a larger unembrittled transition temperature, was less susceptible to temper embrittlement as characterized by the shift in transition temperature.

5. While step cooling provides an assessment of the maximum embrittlement likely to be encountered in a steel, regardless of temperature, it does not provide a determination of the degree or rate of embrittlement that will develop at the service temperature.

6. By step cooling, a perceivable drop in the upper shelf
was observed. This decrease of high temperature toughness was not observed during isothermal embrittlement. Examination of Charpy fracture surface with the scanning electron microscope could not distinguish this behavior.

7. The embrittlement that evolves during isothermal aging at 750°F was slow to develop and was preceded by a slight decrease in transition temperature.

8. Step cooled and isothermally aged HY-100 could be significantly de-embrittled by heat treatment at 1050°F for 1 hour.

9. By de-embrittlement of step cooled and isothermally embrittled Charpy specimens, the predominantly intergranular fracture at low temperatures was replaced by microvoid coalescence and a substantial increase in toughness.

10. Optical and scanning electron microscopy revealed that the structure produced by heat treatment was bainite.

11. Metallography substantiated that creep embrittlement, characterized by intergranular void formation, did not occur.
Table 1
CHEMICAL ANALYSIS OF HY-100

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>Material</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Co</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>15164 HY-100</td>
<td>.18</td>
<td>.35</td>
<td>.010</td>
<td>.025</td>
<td>.29</td>
<td>3.16</td>
<td>1.68</td>
<td>.53</td>
<td>.023</td>
<td>.21</td>
<td>.005</td>
<td></td>
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</tbody>
</table>

supplied by Earle M. Jorgensen Co., Seattle, Washington

Table 2
EFFECT OF QUENCH MEDIA ON CENTER HALF-
TIME-TEMPERATURE COOLING RATE

<table>
<thead>
<tr>
<th>Medium</th>
<th>no. of tests</th>
<th>ave. cooling rate (°F/sec.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Still air</td>
<td>2</td>
<td>0.96</td>
</tr>
<tr>
<td>Forced air</td>
<td>4</td>
<td>1.65</td>
</tr>
<tr>
<td>High capacity forced air</td>
<td>2</td>
<td>1.84</td>
</tr>
<tr>
<td>Still oil</td>
<td>3</td>
<td>9.18</td>
</tr>
<tr>
<td>Heated oil</td>
<td>2</td>
<td>10.92</td>
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Table 3
MECHANICAL PROPERTIES OF HY-100
TEMPERED AT 1100 AND 1200°F

<table>
<thead>
<tr>
<th>Temperature(°F)</th>
<th>Time(hr)</th>
<th>Y.S.(ksi)</th>
<th>T.S.(ksi)</th>
<th>% Elong.</th>
<th>% RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>1100</td>
<td>6 1/2</td>
<td>129.7</td>
<td>152.3</td>
<td>17.2</td>
<td>34.7</td>
</tr>
<tr>
<td>1200</td>
<td>6 1/2</td>
<td>108.0</td>
<td>123.1</td>
<td>18.1</td>
<td>52.7</td>
</tr>
</tbody>
</table>

39
Table 4

EFFECT OF STEP COOL EMBRITTLEMENT ON IMPACT PROPERTIES

<table>
<thead>
<tr>
<th>Condition</th>
<th>Midrange Trans. Temp.(°F)</th>
<th></th>
<th>Upper Shelf</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Energy Absorbed (ft-lbs)</td>
<td>Lateral Expansion (mils)</td>
<td>Energy Absorbed (ft-lbs)</td>
<td>Lateral Expansion (mils)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unembrittled</td>
<td>-110</td>
<td>-100</td>
<td>48</td>
<td>36</td>
</tr>
<tr>
<td>Step Cooled</td>
<td>166</td>
<td>218</td>
<td>31</td>
<td>33</td>
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<tr>
<td>ΔTT</td>
<td>276</td>
<td>317</td>
<td>ΔU.S. 17</td>
<td>3</td>
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<tr>
<td></td>
<td>110 ksi yield strength</td>
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<td></td>
<td></td>
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<tr>
<td>Unembrittled</td>
<td>-69</td>
<td>-32</td>
<td>33</td>
<td>23</td>
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<tr>
<td>Step Cooled</td>
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<td>111</td>
<td>24</td>
<td>18</td>
</tr>
<tr>
<td>ΔTT</td>
<td>152</td>
<td>143</td>
<td>ΔU.S. 9</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>135 ksi yield strength</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Condition</td>
<td>Midrange Transition Temperature (°F)</td>
<td>110 ksi</td>
<td>135 ksi</td>
<td></td>
</tr>
<tr>
<td>--------------------</td>
<td>-------------------------------------</td>
<td>---------</td>
<td>---------</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Energy Absorbed (ft-lbs)</td>
<td>Lateral Expansion (mils)</td>
<td>Energy Absorbed (ft-lbs)</td>
<td>Lateral Expansion (mils)</td>
</tr>
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<td>-100</td>
<td>-69</td>
<td>-32</td>
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<tr>
<td>1 hour</td>
<td>-147</td>
<td>-131</td>
<td>-84</td>
<td>-48</td>
</tr>
<tr>
<td>10 hours</td>
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<td>-73</td>
<td>-58</td>
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<tr>
<td>30 hours</td>
<td>-81</td>
<td>-75</td>
<td>-82</td>
<td>-62</td>
</tr>
<tr>
<td>100 hours</td>
<td>-102</td>
<td>-83</td>
<td>-102</td>
<td>-92</td>
</tr>
<tr>
<td>300 hours</td>
<td>-94</td>
<td>-101</td>
<td>-70</td>
<td>-38</td>
</tr>
<tr>
<td>1000 hours</td>
<td>-80</td>
<td>-80</td>
<td>-42</td>
<td>-53</td>
</tr>
<tr>
<td>2000 hours</td>
<td>-37</td>
<td>-25</td>
<td>+9</td>
<td>+33</td>
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<tr>
<td>5000 hours</td>
<td>+53</td>
<td>+64</td>
<td>+51</td>
<td>+43</td>
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</table>
Table 6
DE-EMBRITTLEMENT OF HY100 AT 1050°F FOR 1 HOUR

<table>
<thead>
<tr>
<th>Heat Treatment</th>
<th>Test Temperature(°F)</th>
<th>% Embrittlement</th>
<th>% Recovery</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>E.A.</td>
<td>L.E.</td>
</tr>
<tr>
<td>110 ksi yield strength</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Step cooled</td>
<td>-25</td>
<td>94</td>
<td>97</td>
</tr>
<tr>
<td>1000 hours</td>
<td>-50</td>
<td>29</td>
<td>7</td>
</tr>
<tr>
<td>2000 hours</td>
<td>-50</td>
<td>44</td>
<td>33</td>
</tr>
<tr>
<td>5000 hours</td>
<td>-40</td>
<td>89</td>
<td>91</td>
</tr>
<tr>
<td>135 ksi yield strength</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Step cooled</td>
<td>-25</td>
<td>69</td>
<td>67</td>
</tr>
<tr>
<td>1000 hours</td>
<td>-25</td>
<td>27</td>
<td>8</td>
</tr>
<tr>
<td>2000 hours</td>
<td>-25</td>
<td>50</td>
<td>42</td>
</tr>
<tr>
<td>5000 hours</td>
<td>-4</td>
<td>46</td>
<td>40</td>
</tr>
</tbody>
</table>

% Embrittlement = \((1 - \frac{\text{embrittled}}{\text{unembrittled}}) \times 100\) (at testing temperature)

% Recovery = \((\frac{\text{de-embrittled}}{\text{unembrittled} - \text{embrittled}}) \times 100\) (at testing temperature)

E.A. = energy absorbed (ft-lbs)
L.E. = lateral expansion (mils)
<table>
<thead>
<tr>
<th>Condition</th>
<th>Unnotched at R.T.</th>
<th>Notched at -58°F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Y.S.(ksi)</td>
<td>T.S.(ksi)</td>
</tr>
<tr>
<td><strong>110 ksi yield strength</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unembrittled</td>
<td>108.0</td>
<td>123.1</td>
</tr>
<tr>
<td>Step Cooled</td>
<td>114.7</td>
<td>131.0</td>
</tr>
<tr>
<td>1000 hrs. at 750°F</td>
<td>109.3</td>
<td>124.5</td>
</tr>
<tr>
<td>2000 hrs. at 750°F</td>
<td>106.3</td>
<td>121.1</td>
</tr>
<tr>
<td>5000 hrs. at 750°F</td>
<td>108.7</td>
<td>122.9</td>
</tr>
<tr>
<td><strong>135 ksi yield strength</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unembrittled</td>
<td>129.7</td>
<td>152.3</td>
</tr>
<tr>
<td>Step Cooled</td>
<td>137.7</td>
<td>152.7</td>
</tr>
<tr>
<td>1000 hrs. at 750°F</td>
<td>132.3</td>
<td>145.8</td>
</tr>
<tr>
<td>2000 hrs. at 750°F</td>
<td>137.9</td>
<td>151.7</td>
</tr>
<tr>
<td>5000 hrs. at 750°F</td>
<td>134.4</td>
<td>148.9</td>
</tr>
</tbody>
</table>
### Table 8

**EFFECT OF APPLIED STRESS ON EMBRITTLEMENT**

<table>
<thead>
<tr>
<th>Condition</th>
<th>Shift in lateral expansion trans. temp(°F)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>110 ksi yield strength</strong></td>
<td></td>
</tr>
<tr>
<td>Step Cooled</td>
<td>98</td>
</tr>
<tr>
<td>Aged 1000 hr.-no stress</td>
<td>20</td>
</tr>
<tr>
<td>Aged 1000 hr.-55 ksi</td>
<td>-80</td>
</tr>
<tr>
<td><strong>135 ksi yield strength</strong></td>
<td></td>
</tr>
<tr>
<td>Step Cooled</td>
<td>76</td>
</tr>
<tr>
<td>Aged 1000 hr.-no stress</td>
<td>20</td>
</tr>
<tr>
<td>Aged 1000 hr.-67.5 ksi</td>
<td>-80</td>
</tr>
</tbody>
</table>
Figure 1: Cooling Study - Plate Specifications
CREEP SPECIMEN

FABRICATION OF CHARPY IMPACT SPECIMEN BY FRICTION WELDING

ORIENTATION OF IMPACT SPECIMEN TO BE REMOVED

Figure 2: Preparation of Impact Specimens from Creep Specimen
Figure 3: Specimen Orientation
Figure 4: Effect of Plate Thickness on Center Cooling Rate
Figure 5: Tempering Dependence of Hardness
Figure 6: Tempering Dependence of Strength
Figure 7: Tempering Parameter

\[ T_p(10^4) = T(\text{oK}) \left[ 20 + \log_t(\text{hr}) \right] \]
Figure 8: Correlation of Mechanical Properties Produced on Tempering
Figure 9B: Kinetics of Isothermal Embrittlement - 110 ksi Yield Strength
Figure 10A: Kinetics of Isothermal Embrittlement - 135 ksi Yield Strength
Figure 10B: Kinetics of Isothermal Embrittlement - 135 ksi Yield Strength
Figure 11A: Effect of Embrittlement on the Upper Shelf - 110 ksi Yield Strength
Figure 11B: Effect of Embrittlement on the Upper Shelf - 135 ksi Yield Strength
Figure 12A: Optical Micrograph of Quenched and Tempered HY-100. Tempered bainite structure revealed by 2% Nital etch. 500X.

Figure 12B: SEM Micrograph of Quenched and Tempered HY-100. Tempered bainite structure revealed by 2% Nital etch. 1700X.
Figure 13: SEM Fractograph - Step Cool Temper Embrittled. Note that the intergranular fracture and secondary cracking of the Charpy specimen are predominant. 300X.

Figure 14: SEM Fractograph - Step Cool Temper Embrittled - Subsequently De-embrittled. Note the ductile fracture surface of the Charpy specimen. 300X.
Figure 15: SEM Fractographs of Temper Embrittled HY-100. Dimpled fracture surface of the unnotched tensile specimen (a) is replaced by dimples with evidence of secondary grain boundary cracking in the notched specimen (b). Both at 400X.
REFERENCES


70. Roper, C., Lukens Steel Co., RDR 68-12, June 1968.


Figure A1: 110 ksi yield strength - Unembrittled
Figure A-2: 110 ksi yield strength - Step Cooled

A-2
Figure A-3: 135 ksi yield strength - Unembrittled
HY100 - STD. CHARPY
QUENCHED + TEMPERED
135 KSI YIELD ST.
STEP COOLED

Figure A-4: 135 ksi yield strength - Step Cooled
Figure A-5: 110 ksi yield strength - Aged 1 hour
Figure A-6: 110 ksi yield strength - Aged 10 hours
Figure A-7: 110 ksi yield strength - Aged 30 hours
Figure A-8: 110 ksi yield strength - Aged 100 hours
Figure A-9: 110 ksi yield strength - Aged 300 hours
Figure A-10: 110 ksi yield strength - Aged 1000 hours
Figure A-11: 110 ksi yield strength - Aged 2000 hours
Figure A-12: 110 ksi yield strength - Aged 5000 hours
Figure A-13: 135 ksi yield strength - Aged 1 hour
Figure A-14: 135 ksi yield strength - Aged 10 hours
Figure A-15: 135 ksi yield strength - Aged 30 hours
HY100 - STD. CHARPY
QUENCHED + TEMPERED
to
135 ksi YIELD ST.
AGED 100 HOURS
AT 750 F

Figure A-16: 135 ksi yield strength - Aged 100 hours
Figure A-17: 135 ksi yield strength - Aged 300 hours
Figure A-18: 135 ksi yield strength - Aged 1000 hours
Figure A-19: 135 ksi yield strength - Aged 2000 hours
Figure A-20: 135 ksi yield strength - Aged 5000 hours

A-20
APPENDIX B: CHARPY CURVES - CREEP EMBRITTLED HY-100

Figure B-1: 110 ksi yield strength - Unembrittled
Figure B-2: 110 ksi yield strength - Step Cooled
Figure B-3: 110 ksi yield strength - Crept 1000 hours

A-23
Figure B-4: 135 ksi yield strength - Unembrittled

A-24
HY100-SUBSIZE CHARPY
QUENCHED + TEMPERED
TO
135 KSI YIELD ST.
STEP COOLED

Figure B-5: 135 ksi yield strength - Step Cooled

A-25
Figure B-6: 135 ksi yield strength - Crept 1000 hours
Courtney Jay Hill, son of Raymond J. and Jean Hill, was born in Baltimore, Maryland, on August 5, 1952. A graduate of McDonogh School, Mr. Hill entered Lehigh University in September, 1970. Upon graduation in May, 1974, he was awarded the degree of Bachelor of Science in Metallurgy and Materials Science with highest academic and interdepartmental honors. In the summer of 1974, he was admitted to the graduate school of Lehigh University where he served as a research and teaching assistant in the Department of Metallurgy and Materials Science.

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