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DETERMINATION OF FRACTURE BEHAVIOR OF FERRITIC STEELS USING MINIATURIZED SPECIMENS

M.P. MANAHAN

Battelle Columbus Division, 505 King Avenue, Columbus, OH 43201-2693, USA

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Miniaturized Specimen Technology (MST) permits mechanical behavior to be determined using a minimum volume of material. A method for obtaining the ductile–brittle transition temperature (DBTT) of ferritic steels was developed using a miniaturized notched bar test. Comparisons between conventional and miniaturized specimen DBTTs are encouraging. Fracture toughness values were calculated for the miniaturized notched specimens and compared with large-specimen data.

1. Introduction

Because Miniaturized Specimen Technology (MST) permits characterization of mechanical behavior using a minimum volume of material, it has many applications, such as nuclear pressure vessel surveillance, failure analysis, and postirradiation testing *. The goal of the present study was to examine the feasibility of a fracture mechanics test making use of miniaturized three-point-bend (TPB) specimens. In particular, a test was developed to determine the ductile–brittle transition temperature (DBTT) of ferritic steel. As a secondary objective, an attempt was made to determine plane-strain fracture toughness (K_{Ic}) from the miniaturized notched specimens.

Partly as a result of the high incidence of brittle fracture failures in the steel of the Liberty merchant ships of the US Navy during World War II, a great interest arose in the development of a laboratory test capable of evaluating the susceptibility of steels to brittle fracture. An extensive body of literature has developed over the past 40 years that addresses size and geometry effects on DBTT determination. Many different test methods such as impact tests, drop-weight tests, and dynamic tear tests have been developed to simulate service conditions. Current fracture behavior tests that grew out of this large body of data are discussed in detail in the ASTM Standards [1–12].

The Charpy V-Notch (CVN) impact test, one of the simplest fracture behavior tests, yields a variety of data, including impact energy, lateral expansion, and percentage of ductile fracture area as a function of test temperature. The test is usually conducted over a range of temperatures, within which the transition from brittle to ductile fracture occurs. Previous studies have attempted to isolate the factors that influence DBTT [13–39].

Recent work, most notably that of Corwin and Houghland [40], Lucas et al. [41], and McConnell et al. [42], has indicated that CVN tests, including those for miniaturized specimens, provide only empirical or qualitative data. These authors concluded that the mini-CVN tests have been found valuable for determining material considerations such as fundamental fracture and flow parameters, the validity of interpolative fracture criteria, valid fracture toughness fibrous crack growth resistance curves, and qualitative data for tracking irradiation behavior and monitoring relative differences of materials. ASTM Standard A370-86a, "Mechanical Testing of Steel Products," suggests that the energy values determined by these tests are "qualitative comparisons ... that cannot be converted into energy figures that would serve for engineering calculations" [1]. Further, Lucas et al. state that:

> "Thus while the behavior of mini-CVN specimens has been found to be qualitatively similar to that of standard CVN's, quantitative comparisons have been found to be only in approximate agreement at best. This is not particularly surprising, since parameters

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* The processes described are explained in part in US Patent No. 4567774 dated February 4, 1986. A patent application on further improvements of the methodology is pending.

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such as transition temperature or ΔTT are functions of an arbitrarily chosen reference energy and are thus sensitive to test conditions and particularly specimen geometry. Thus it would seem that the greater potential for success in using mini-CVNs is to use them to extract more fundamental property information" [41].

The present study advances and extends the current theory of mini-CVN testing to obtain, from miniaturized specimens, quantitative DBTT data that are nearly as accurate as those obtained using conventional ASTM E23 specimens. Work to date has focused on $\frac{1}{3}$ or $\frac{1}{4}$ size mini-CVN specimens [40–42]. In the present study, the specimen size is chosen close to the continuum limit of the material. For the ASTM A508 steel investigated, this results in a miniaturized specimen volume that is about $\frac{1}{16}$ the volume of a conventional specimen.

2. Experimental considerations

The experimental design of the miniaturized fracture mechanics test was based on the material microstructure, current testing practice, amount of material available, and desired stress state. These design considerations are discussed in turn below.

2.1. *Material microstructure*

Material for this work was taken from a special heat of ASTM A508 steel provided by Oak Ridge National Laboratory (ORNL) for crack arrest research as part of the Heavy Section Steel Technology (HSST) Program. The steel has been extensively characterized at ORNL as well as Battelle Columbus Division (BCD) and the University of Maryland (UMD) [43]. Conventional mechanical property and microstructural data are available for three different heat treatments, designated 6, 5A, and 6R, in order of increasing toughness and increasing tempering temperature.

In order to confirm the data reported in ref. [43], samples from the three heats were mounted and etched to enhance the ferritic grain structure. Micrographs were made, and the grain size was observed to be between 7.5 and 8.0 for ASTM designation G.

An important limitation in miniaturizing any specimen is the extent of the material's microstructural inhomogeneities. The usual guideline dictates that the specimen be at least five to ten times as large as the characteristic heterogeneity dimension. Microscopy analysis at UMD [43] (using Heat Treatment 5A) indicated that carbon segregates in slender bands about 0.25 mm wide.

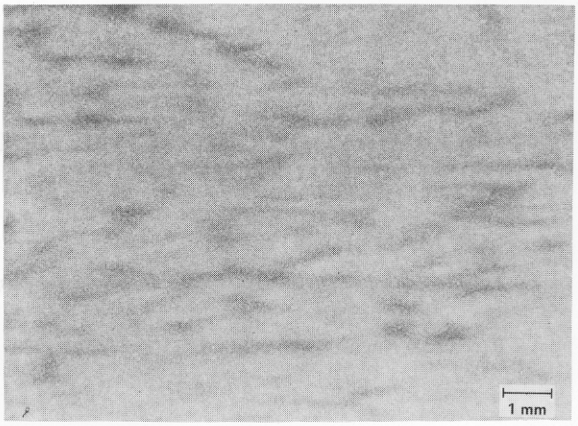

Fig. 1. Low magnification micrograph of Heat Treatment 6R showing overview of carbon segregation.

Examination by the BCD metallography laboratory partly confirmed the existence of segregation, although the morphology was slightly different. Fig. 1 is a low magnification photomicrograph of the steel with Heat Treatment 6R. The dark regions are believed to be carbon-rich, reflecting an enhanced local density of carbide precipitates. Typical bands are shown at higher magnification in fig. 2 for Heat Treatment 5A (intermediate temperature). Steel with the lowest tempering temperature (Heat Treatment 6) exhibited only faint indications of segregation. The reason for this difference is not clear. As a result of these findings, the minimum specimen dimension should be in the range of 3 to 5 mm. For a tensile specimen, this minimum size limits the diameter or thickness; for a fracture behavior

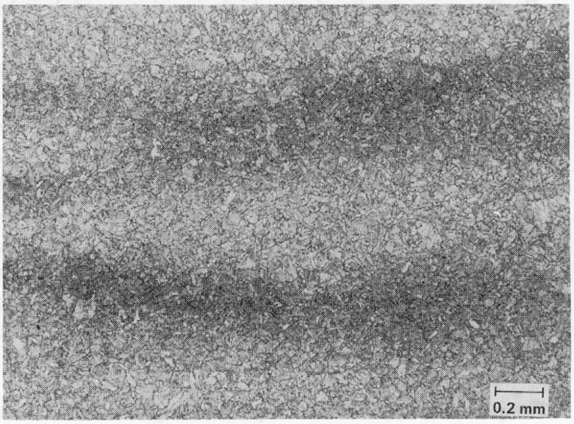

Fig. 2. Higher magnification micrograph of segregation bands in Heat Treatment 5A.

Table 1
Comparison of key experimental parameters

	ASTM conventional Charpy (mm)	Miniature fracture mechanics (mm)
Punch radius	8.00	8.00
Punch tip width	3.99	2.29
Anvil radius	1.00	1.00
Anvil spacing	40.00	11.43

specimen, this minimum limits the dimensions of the crack plane.

2.2. Current testing practice

A number of ASTM standards relate to fracture mechanics testing of steel products [1–12]. Because of its relevance to the nuclear industry and compatibility with current in-service cutting techniques, the miniaturized notched bar specimen was chosen for examination. The specimen geometry was modified so that fracture transition behavior could be characterized using very small specimens.

The anvils were machined in accordance with ref. [2]. All miniaturized specimen testing was performed statically. Conventional Charpy specimens were tested both statically and dynamically using a punch and anvil spacing as prescribed in ref. [2]. For miniaturized specimens, it was necessary to decrease the punch thickness and anvil spacing. Key experimental parameters are compared in table 1. Other procedures and specifications regarding the test temperature, alignment accuracy, and machining tolerances were also in accordance with ref. [2] specifications.

Table 2
Comparison of key specimen dimensions

	ASTM Charpy specimen (mm)	Miniature specimen (mm)
Thickness (B) (crack plane)	10.01	4.83
Depth (H) (direction of crack propagation)	10.01	4.83
Length (L)	54.99	12.70
Reduced side thickness (B_n)	*n/a*	3.86
Notch depth (a)	2.01	0.97
Notch-root radius (r)	0.25	0.25

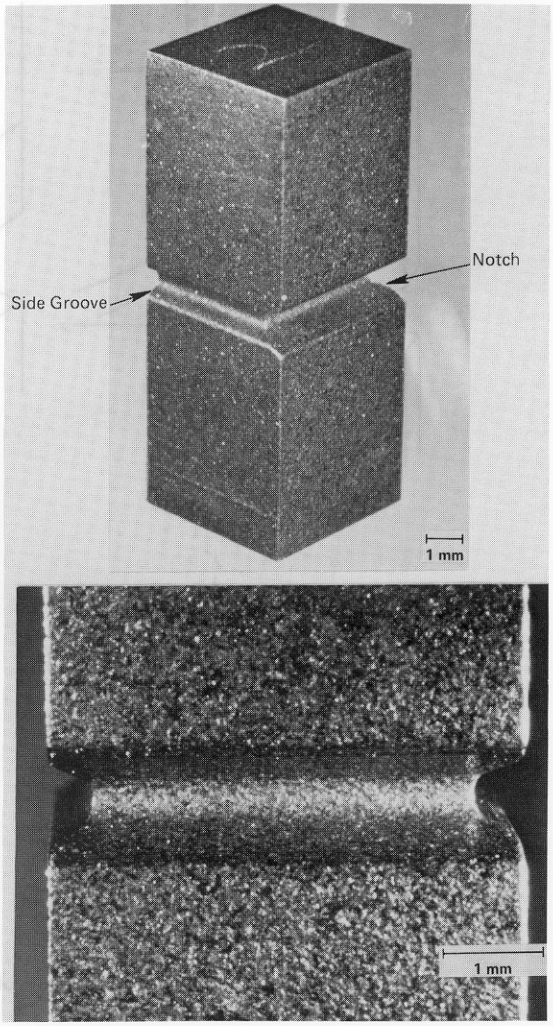

Fig. 3. Miniaturized specimen showing side grooves and notch.

2.3. Material availability

The constraints on material volume in some future applications of the technology were considered in the design. The ratio of the volume of the ASTM standard compact-tension (CT) specimens to the volume of conventional TPB specimens is approximately 0.4. Therefore, the CT specimen is desirable based on a reduced volume criterion. However, the ratios of the thickness (crack plane), depth (direction of crack propagation), and length of the TPB specimen to those of the CT specimen are 1.0, 0.80, and 3.25, respectively. The TPB

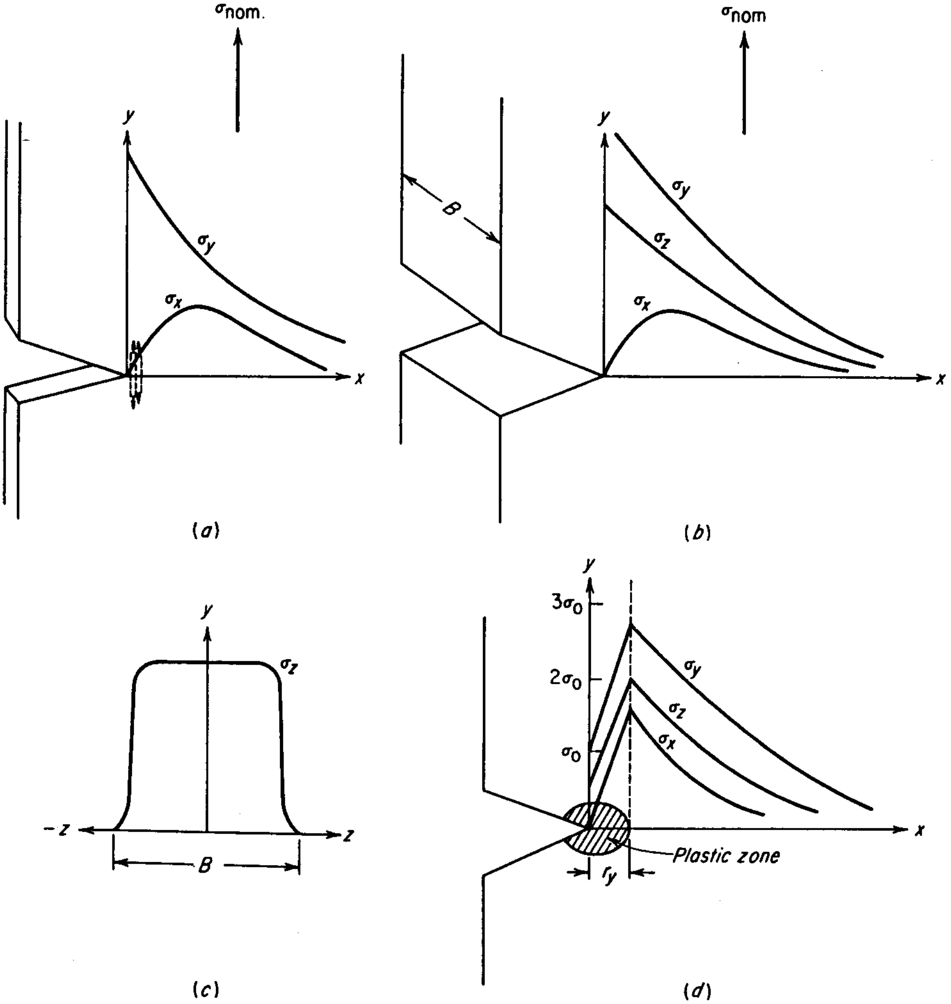

Fig. 4. Schematic of crack tip stress fields [44].

specimen may be more useful for semidestructive sampling, since it is difficult to obtain a through-thickness crack propagation specimen without cutting large, deeply penetrating pieces of material from the surface of a component for CT specimens. Therefore, miniaturized TPB specimens were chosen.

The miniaturized TPB specimens were sized so that eight specimens could be machined from each half of a conventional broken Charpy specimen. This permits the maximum number of specimens to be produced from a broken Charpy specimen from a nuclear surveillance capsule and still satisfy the material-based size requirements for the steel being investigated.

2.4. Desired stress state

An important aspect of a fracture mechanics specimen design is the size effect. The stress field near the crack tip goes from an essentially triaxial (plane strain) to a biaxial (plane stress) field as the specimen thickness decreases. Since K_{Ic} is a function of only the material microstructure, it is the field parameter of interest. Plane stress and mixed mode fracture toughness (K_{c}) depend on both geometrical and metallurgical factors.

The approach adopted in miniaturized specimen testing was to modify the specimen design to increase plastic constraint. In the current program, side grooves were machined into the specimen in addition to the notch, as shown in fig. 3. The key miniaturized specimen design parameters are compared with conventional specimen parameters in table 2.

The effect of the side grooves is illustrated in fig. 4. The σ_x stress component from the side groove in the thin sample is intended to offset the lack of the through-thickness stress σ_z that is present in the thick specimens. Although the σ_x stress in the miniaturized specimen is not, in general, uniformly distributed and is not of the same magnitude as the σ_z stress in the thick samples, sufficient constraint can be induced to enable measurement of the DBTT using miniaturized specimens. Historically, side grooving has been used to obtain more uniform crack fronts in fracture toughness testing and to constrain the fracture plane in certain materials. In the present study, the side grooves are brought into close proximity so that their σ_x stress components overlap; the result is a fairly uniform through-thickness stress field.

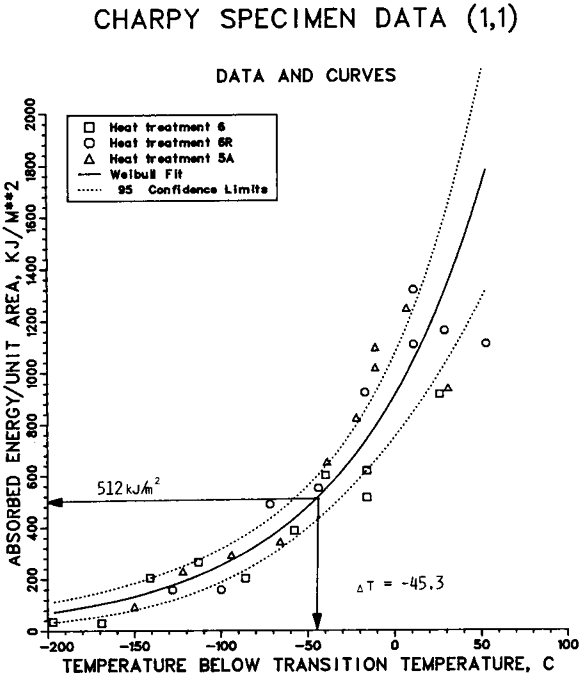

Fig. 5. Energy absorbed in slow-bend fracture of standard Charpy specimens within the ductile/brittle transition region.

3. Data analysis and results

3.1. Transition temperature shift

Several DBTT criteria are used in different industries. Since nuclear-grade steel was used in this study, the 41-J (30-ft-lb) energy absorption level was used as a reference. For the three heat treatments, the Charpy DBTTs are as follows: Heat Treatment 6, 40 °C; Heat Treatment 5A, −7 °C; and Heat Treatment 6R, −29 °C. To allow a heat-to-heat comparison, all the test temperatures for the slow-bend specimens were adjusted by subtracting the appropriate impact-transition temperature. In addition, the absorbed energy was divided by the area of the crack plane in an attempt to place the standard Charpy and miniaturized bend specimen data on a common basis. Fig. 5 shows the conventional Charpy specimen data normalized in this way. The normalizations provide good superposition of the data, as expected. The 41-J (512-kJ/m^2) level occurs at about −45.3 °C. This factor accounts for the downward shift in the 41-J index due to testing statically. The data were fit using the statistical analysis methodology reported in refs. [45,46]. Ref. [47] presents a correlation for the temperature shift between slow-bend and impact loading. Using this correlation, the expected shift would be about 36 °C. This is in reasonable agreement considering that the ref. [47] correlation is based on the assumption that the onset of the dynamic transition temperature is defined by the intersection of tangent lines drawn from the lower shelf level and the transition region.

Fig. 6 shows the results for the miniaturized specimens. Two specimens at higher temperatures are off the scale of interest and consequently are not plotted. The superposition is again good at lower temperatures. The use of the miniaturized specimens results in a further downward shift of 76.7 °C in temperature, producing a total shift of −122 °C between standard impact-Charpy tests and slow-bend miniaturized specimen tests. This result provides encouragement that miniaturized specimen procedures can yield DBTT data of comparable accuracy to those obtained using standard ASTM E23 procedures.

The miniaturized specimen data in fig. 6 are scattered at higher temperatures due to experimental difficulties. At higher temperatures, where there is stable crack growth and substantial plastic deformation, the specimens were observed to slip off the flat portion of the anvil and were supported by the curved portion. In future work, the radius of curvature of the anvil and the punch should be scaled down. These changes are essential to measure high transition and upper-shelf data. Consideration should also be given to increasing the span-to-width ratio.

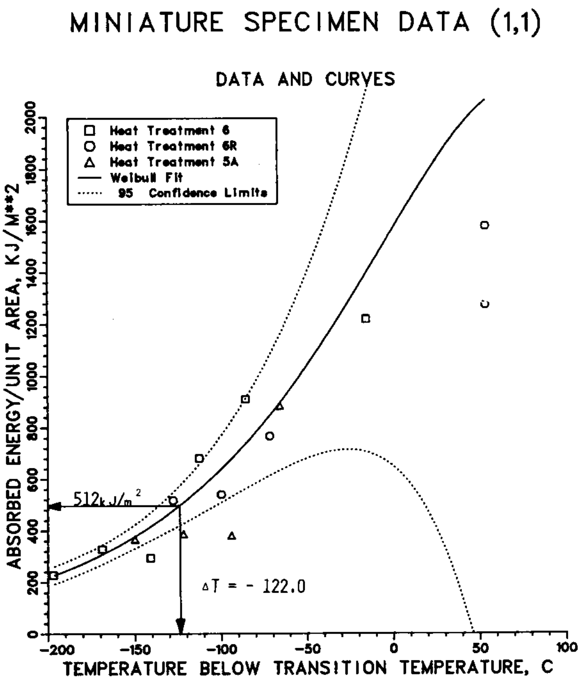

Fig. 6. Energy absorbed in slow-bend fracture of miniaturized specimens within the ductile/brittle transition region.

Tests were run without side grooving the specimen, and the data in the transition region could not be analyzed due to severe plastic deformation. Therefore, although the experimental hurdle of DBTT testing near the material continuum limit has been overcome, future studies should focus on the mode of fracture. The use of the normalized energy parameter and the 512-kJ/m^2 index must be investigated to ensure consistency in fracture behavior.

3.2. *Fracture toughness determination*

A secondary objective of this work was to determine whether the miniature notched specimens could be used to measure plane strain fracture toughness. Therefore, the load versus load point displacement data were recorded and analyzed. At high test temperatures, the load–displacement curve (Path 0ABC, fig. 7) passes through a maximum and then slowly declines (upper-shelf behavior). The behavior is a result of slow crack growth. At lower temperatures, a similar curve is observed, except that unstable cleavage fracture intervenes, resulting in curves 0A1 and 0AB2. The instability point shifts to lower displacements as the test temperature is lowered. This behavior has been arbitrarily divided into two classes:

(1) Failure before the load peak in fig. 7 (denoted by lower shelf).
(2) Failure after the peak in fig. 7 (denoted by upper shelf and transition).

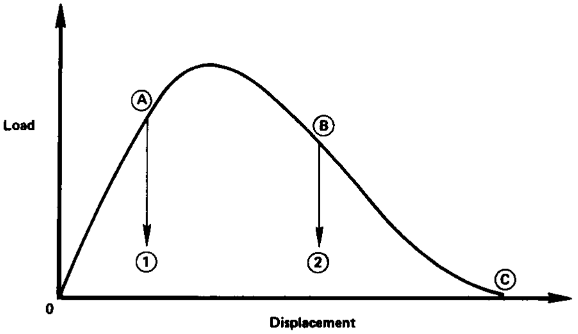

Fig. 7. Schematic load–displacement curve.

Published fracture toughness data [43] are available only for Heat Treatments 6 and 5A. This information was obtained principally at ORNL using two specimen types as follows:

(1) Large cylinders (~ 1.2-m length × 1.0-m outer diameter × 150-mm wall thickness) subjected to thermal shock).
(2) Standard compact specimens (~ 25 mm thick × 60 mm square). K_{Ic} values for these specimens were reported using a thickness correction developed by Irwin [48].

Table 3
Plastic zone size estimates based on LEFM for Heat Treatment 6

Temperature (°C)	Plastic zone size (r_y) (mm)	Ratio of r_y to miniature specimen reduced thickness (B_n)	Ratio of r_y to Charpy specimen thickness (B)
−73	0.31	0.08	0.03
−46	0.38	0.10	0.04
−32	0.58	0.15	0.06
−18	0.67	0.17	0.07
10	0.84	0.22	0.08
32	1.30	0.34	0.13
38	0.77	0.20	0.08
66	1.73	0.45	0.17
82	1.93	0.50	0.19
135	3.45	0.89	0.34

The fracture toughness data obtained from the current program, which uses even smaller specimens, were corrected for side groove geometry and plasticity.

In fracture mechanics testing, it is necessary to keep the plastic zone size small compared to the thickness, the uncracked ligament, and the crack length. Once the plastic zone becomes too large, the currently accepted fracture mechanics field parameters may not be appropriate. Also, the plastic zone size for plane strain is typically one-third of that for plane stress. Table 3 contains estimates of the plastic zone size for plane-strain conditions based on linear elastic fracture mechanics (LEFM). Table 3 also lists the ratio of the plastic zone size (r_y) to the specimen thickness (B) for the miniaturized specimens and Charpy specimens. The adjusted fracture toughness values reported in ref. [49] were averaged at each temperature for Heat Treatment 6. ASTM E399 requires that $r_y/V \leq 0.02$ to obtain valid K_{Ic} data [3]. As seen in table 3, this criterion is not met, and therefore a mixed mode condition likely exists over the temperature range tested.

In an attempt to obtain valid K_{Ic} data, K_{Ic} relations and correction factors not yet recommended by ASTM were used. The general approach to testing methodology is to adhere to the guidance outlined in ASTM standards as much as possible [3,4].

The basic elements of the data treatment are as follows:

(1) Use Srawley's [50] wide range stress intensity factor expression to calculate the driving force for fracture.
(2) Apply a geometry correction to account for the side grooves.
(3) Assume that the elastic stored energy is the only energy available to drive a cleavage crack.
(4) Adjust the apparent fracture toughness values obtained by applying a size correction.

Elements (1) and (2) above are well-known and established within the fracture mechanics community. Elements (3) and (4) are among those being considered by ASTM Standard subcommittees and must be characterized as controversial. For the side grooved miniaturized specimens, the correction factor B/B_{n} was applied to account for the geometry change. In the present study, element (4) was not used.

Rosenfield and Shetty's [51] energy correction method was used. The argument is that the energy expended in stable growth is not available to drive the unstable crack. Therefore, the influence of this energy

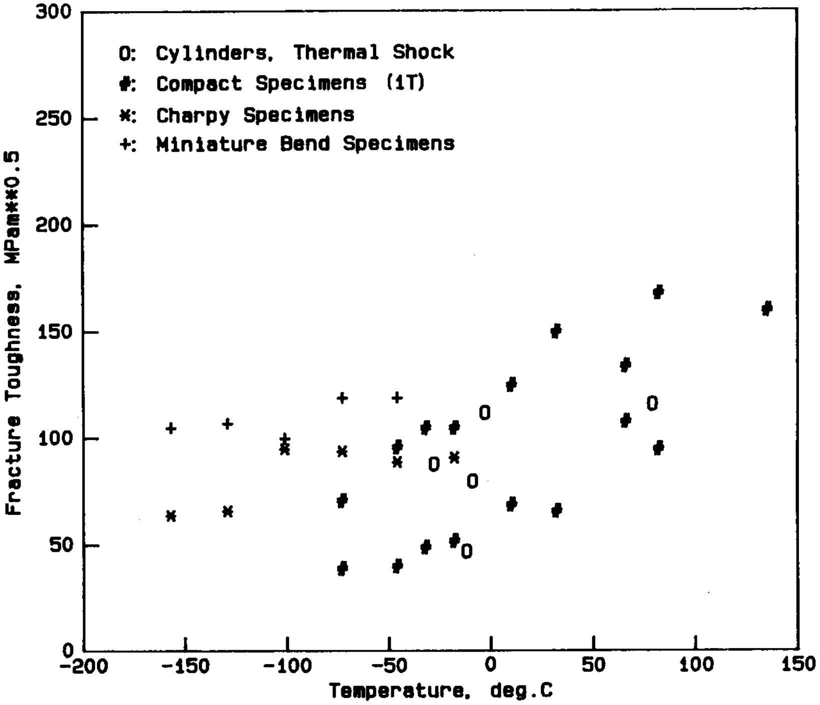

Fig. 8. Fracture toughness; Heat Treatment 6.

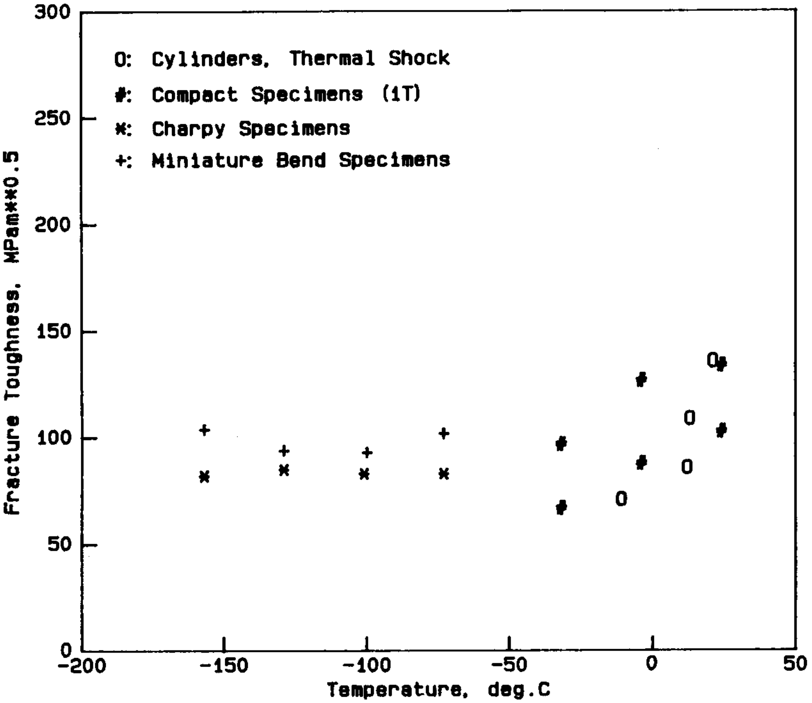

Fig. 9. Fracture toughness; Heat Treatment 5A.

must be eliminated from the experimental record if the data are to be representative of a large vessel where stable growth is unlikely to occur because of the geometrical constraint.

Only the lower transition specimens are included in the comparison because of uncertainties in analyzing the other data. Figs. 8 and 9 show BCD's toughness data compared to the ORNL larger-specimen data. Both figures indicate the extent to which the Charpy and miniaturized-specimen procedures succeeded. Fig. 8 shows that cleavage failure was achieved in the Charpy specimen at a temperature which overlaps both the 1T and cylinder data for Heat Treatment 6. The miniaturized data overlapped only the 1T results. Even so, both designs produced data that were on the high (nonconservative) edge of the simulated vessel scatter band. In fig. 9, for Heat Treatment 5A, temperature overlap was not achieved and the data also appear to be high.

Therefore, based on these observations, we have concluded that the technique of side grooving for stress field modification cannot, in general, produce sufficient through thickness stress to yield plane strain conditions in static testing of miniature A508 steel specimens. Possibilities for approaching plane-strain conditions further using miniaturized specimens include:

1. Fatigue precracking the specimens in accordance with standard procedures.
2. Side grooving the Charpy specimen as the miniaturized specimen has been. This alternative has the advantage of economy at the sacrifice of size. Some savings in material (a factor of three) could be achieved if a reconstitution procedure is used whereby arms are welded onto the center sections of Charpy specimens.
3. Loading at higher rates, to obtain some decrease in toughness to approach lower bound values.

4. Summary

Contrary to beliefs expressed in the literature, it has been possible to develop a miniature test that is capable of yielding quantitative DBTT data. The miniaturized specimen design required only 6% of the volume of a standard Charpy specimen. These specimens have proven satisfactory for estimating transition temperature shifts due to heat treatment of a reactor-grade pressure vessel steel. The key to the approach is the modification of the stress field in the vicinity of the crack plane to ensure that fracture mode transition behavior is exhibited. Without this experimental modification, the deformation for most engineering materials is too severe to yield useful data. In the future, the research should be extended to include several irradiated structural steels and weldments. Of key importance to complete validation of the method is careful investigation of fracture modes in the miniaturized specimens.

High fracture toughness values were obtained using the miniaturized specimens, even after appropriate adjustments were applied. This indicated that, although the stress field modification is sufficient to yield fracture mode transition data, it is not sufficient to yield plane strain conditions in a static test for the A508 steel. Specimen and test procedure modifications may lead to characteristically low toughness values.

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