Investigation of the Characterization of Material Inhomogeneity and Its Effect on the Ductile-to-Brittle Transition in Ferritic Steel[1]

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It has become routine to characterize the ductile-to-brittle transition in ferritic structural steels in terms of the Master Curve approach and the T_o reference temperature. A standard procedure has been developed in ASTM E1921 which utilizes as few as six small specimens to develop both the T_o reference temperature and the expected confidence bounds. This procedure implicitly assumes that the material is homogeneous and the six specimens tested are a faithful representation of the material to be utilized in the structural application. In many important applications the structure can be very large and the specimens can be cut from material removed from a single location during fabrication. The question as to whether the specimen results can be used to accurately or conservatively assess structural performance requires additional consideration. Recently an annex to E1921[1] has been proposed with procedures to investigate whether a material is homogeneous or inhomogeneous. The work presented in this paper describes a research program which applied a simple Monte Carlo procedure to address deficiencies in the proposed annex. Idealized input data sets representing both homogeneous and bimodal materials were analyzed using the proposed procedure to examine the likelihood that the proposed criteria for identifying the homogeneity of the material is acceptable. The assumed "test temperatures" and the number of data input to the analysis strongly influences the accuracy of the results of the annex procedure. Based on the results of the analyses, proposals are presented for modifying the proposed homogeneity annex.

1.0 Objective

The ASTM E1921 procedure utilizes a data set as small as six fracture toughness specimens to characterize the ductile-to-brittle transition in ferritic structural steels in terms of the Master Curve approach and the T_o reference temperature. The procedure relies on the implicit assumption that the material is homogeneous and provides confidence bounds consistent with this assumption. The method acknowledges the possibility of an "occasional outlier" and suggests that it is possible to reduce the influence of the outlier by testing additional specimens. While the outlier cannot be discarded, the method does not provide guidance if additional test results fall outside of the expected confidence bounds.

In reality many applications require defining the ductile-to-brittle transition in structural materials that are known from the outset to be inhomogeneous. To investigate the homogeneity

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of an individual data set an annex has been proposed for E1921 based on the work in Wallin et al.[2]. In this proposed annex, the data set is fit to either a bimodal Weibull distribution or a more general multi-modal distribution. If the data fits either of these cases the material is presumed to be inhomogeneous and revised confidence bounds are developed to more accurately account for the material inhomogeneity. The procedure has been evaluated in an informal round robin and the participants consistently determined the material properties for a given set of data. However, there was more variability in the calculations pf the significance criterion, MLNH, and participants were not able to consistently determine whether a data set represented a homogeneous, bimodal, or multi-modal material. There is also no justified requirements for the number of specimens that should be tested to achieve a desired level of confidence that the material is inhomogeneous. Additionally, participants identified that the proposed testing regime of $(T_b - 50^oC) \leq T_o \leq (T_a + 50^oC)$ could be much wider than currently allowed for a homogeneous sample.

In this paper a research program is described which utilizes a Monte Carlo (MC) method to determine the number of specimens necessary to accurately define whether a bimodal or homogeneous material model more accurately represents a data set. The multi-model option of the proposed annex is not investigated here. This work also investigates the allowable temperature range that is appropriate for testing a bimodal material.

2.0 Analysis

The bimodal model in the proposed annex to E1921 assumes that the material is described by two independent Weibull distributions. Each distribution also adheres to the E1921 master curve relationship between material toughness and temperature. The combined distribution is fully defined by three parameters; the reference temperature of the population represented by distribution A, T_a, the reference temperature of the population represented by distribution B, T_b, and the probability that an individual specimen is from population A, p_a.

T_a, T_b, and p_a are determined using an appropriate solver that maximizes the logarithm of the likelihood given by:

$$\ln L = \sum_{i=1}^{N} \delta_i \ln f + (1 - \delta_i) \ln S_i \tag{1}$$

where:
N = the number of specimens tested,
δ_i = 1.0 if the datum is valid or zero if the datum is a dummy substitute value,
f_i = the datum failure density given below,
S_i = the datum cumulative survival probability given below.

$$f_i = 4 p_a \frac{\left(K_{Jc(i)} - K_{\min}\right)^3}{\left(K_a(T_{(i)}) - K_{\min}\right)^4} \exp\left[-\left(\frac{K_{Jc(i)} - K_{\min}}{K_a(T_{(i)}) - K_{\min}}\right)^4\right] + 4(1 - p_a) \frac{\left(K_{Jc(i)} - K_{\min}\right)^3}{\left(K_b(T_{(i)}) - K_{\min}\right)^4} \exp\left[-\left(\frac{K_{Jc(i)} - K_{\min}}{K_b(T_{(i)}) - K_{\min}}\right)^4\right] \tag{2}$$

$$S_i = p_a \exp\left[-\left(\frac{K_{Jc(i)} - K_{\min}}{K_a(T_{(i)}) - K_{\min}}\right)^4\right] + \left[(1 - p_a) \exp\left[-\left(\frac{K_{Jc(i)} - K_{\min}}{K_b(T_{(i)}) - K_{\min}}\right)^4\right]\right] \tag{3}$$

where: K_{min} = 20 MPa√m,

$K_{Jc(i)}$ = either a valid K_{Jc} datum or a dummy value substituted for an invalid datum using the E1921 censoring procedure,

$K_a(T_{(i)})$=characteristic fracture toughness values (K_o) of population a at the test temperature $T_{(i)}$,

$K_b(T_{(i)})$=characteristic fracture toughness values (K_o) of population b at the test temperature $T_{(i)}$.

The uncertainty in the estimated parameters depends on the number of data available and is given by

$$\sigma_{T_a} = \frac{22°C}{\sqrt{N \cdot p_a - 2}} \tag{4}$$

$$\sigma_{T_b} = \frac{16°C}{\sqrt{r - N \cdot p_a - 2}} \tag{5}$$

$$\sigma_{p_a} = \frac{0.35}{\sqrt{N \cdot p_a - 2}} \tag{6}$$

where *r* is the number of valid data. The criterion proposed to determine if a material is bimodal is given by

$$MLNH = \frac{|T_a - T_b|}{\sqrt{\sigma_{T_a}^2 + \sigma_{T_b}^2 + \sigma_{\exp}^2}} > 2 \tag{7}$$

where σ_{exp} is the experimental uncertainty and is a constant 4°C.

Distributions A and B are ordered so that $T_b \leq T_a$. Because three parameters are estimated, the proposed annex suggests that 12 to 16 specimens be tested to determine if the material is inhomogeneous. This is essentially twice the minimum number of tests required in E1921 to determine T_o for a homogeneous material. The valid temperature range suggested for the bimodal distribution is [T_b -50°C, T_a +50°C] which can be much wider than E1921 presently allows, [T_o -50°C, T_o +50°C], if the difference between T_a and T_b is large.

In the method applied in this study T_a , T_b , p_a were assumed for the idealized material using values that are representative of thick-walled steel plate materials. For each analysis a "test temperature" (T) and number of specimens for the data sample (N) were chosen. A MC analysis was then performed using the following steps: 1) a set of N K_{Jc} test values were randomly sampled from this idealized material, and 2) values of T_a, T_b, and p_a were estimated for this set of data using Eqs. 1-6 and MLNH was evaluated using Eq. 7. Steps 1) and 2) were repeated 1000 times and the number of times MLNH was greater than 2.0 was determined. This analysis was repeated for three different assumed bimodal distributions with T_a - T_b = 20, 30, and 40°C respectively, and p_a = 0.75. Different simulations were also conducted to sample data using a single temperature or at multiple temperatures as allowed by E1921 and the proposed inhomogeneity annex.

A similar MC analysis was also conducted for an idealized material which is represented by a single Weibull distribution such that $T_a = T_b = T_o$ and p_a = 0 or 1. Steps 1) and 2) above were repeated 1000 times for this material and the number of times that MLNH was greater than 2.0 was again determined.

3.0 Bimodal Weibull Results

Figure 1 shows the results of applying the Monte Carlo analysis to the Annex single temperature procedure for cases of 12 and 32 specimens, using and input bimodal distribution with T_a = -75°C, T_b = -105°C, and p_a = 0.75, and with the data sets chosen at bimodal distribution T = T_a and T = T_b temperatures, and at the center temperature between the two bimodal reference temperatures, here referred to as T_c. This plot shows the number of times in the 1000 sample MC that the MLNH value on the x-axis was calculated. Most of the data cells clustered at MLNH=0 correspond to the case where the argument of the square root was less than zero in one of the denominators in Eqs. 4 and 5. The proposed annex provides no guidance what to do in such a case, but since this typically corresponds to a small or large p , it is reasonable to assume that the procedure implies that the material cannot be shown to be bimodal.

Clearly from this result, 12 data samples are inadequate to correctly determine with any reasonable confidence that the data sets are chosen from a bimodal Weibull distribution since these data sets meet the requirement that MLNH > 2.0 only about 30% of the time.

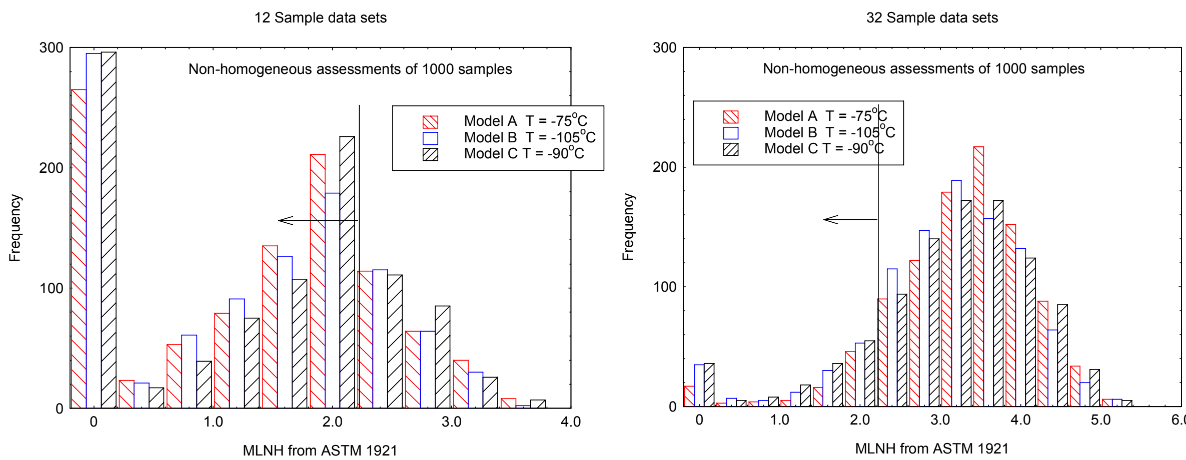
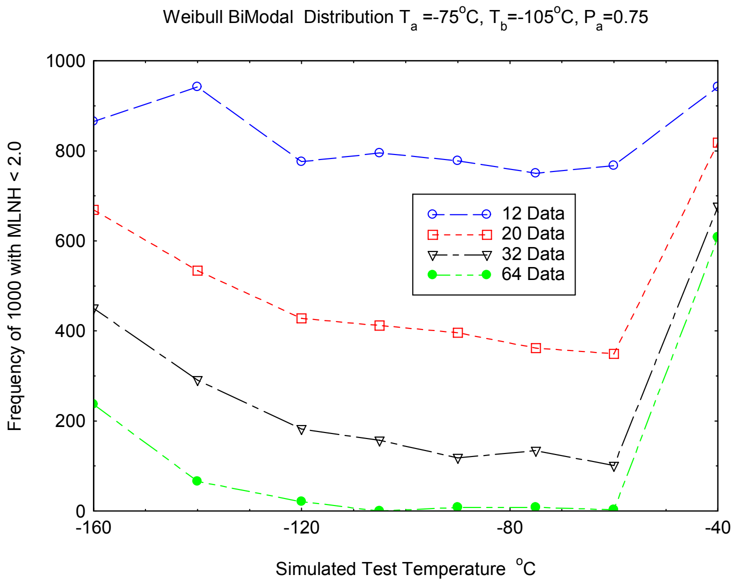


Figure 1 Results showing the observed frequency distribution of the MLNH parameter for the 12 and 32 data sample analyses, respectively.

Increasing the sample size to 32 data gives a much more satisfactory result, as is shown in Figure 1. Here, the analysis correctly determines that the input data corresponds to a bimodal distribution 85 to 90% of the time at the three simulated input temperatures.

A wider range of sample set temperatures and N values were evaluated for the same idealized bimodal material as shown in Figure 2. This plot depicts the number of instances in the MC analyses where MLNH < 2.0 as a function of the simulated test temperature. Therefore, these are the number of times that the procedure cannot identify the material as bimodal with statistical confidence. These results show that sample set sizes of 64 specimens are required to accurately predict the presence of the bimodal distribution with an error of less than 10% over a range of temperatures from -160°C to -40°C.

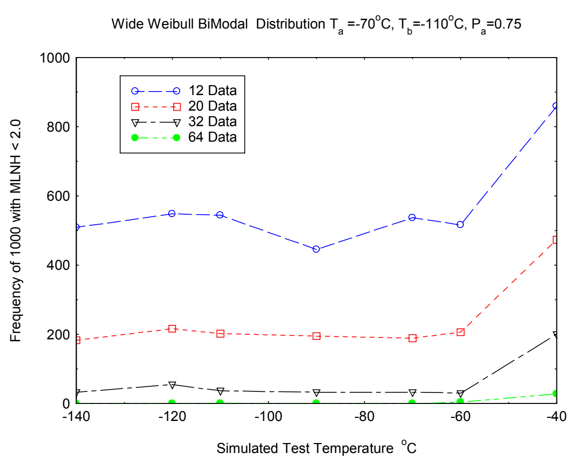


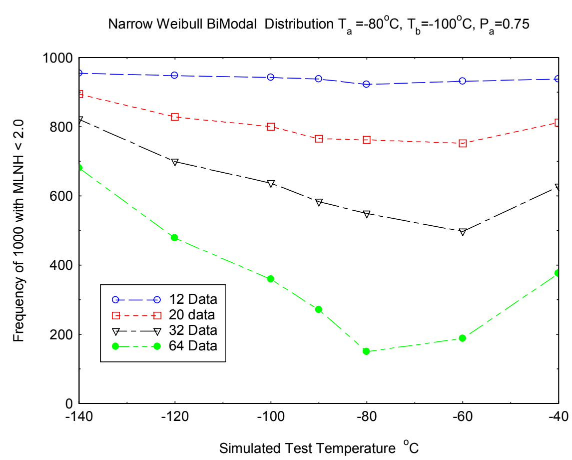
At the -40°C temperature, many of the highest toughness data corresponding to the T_b distribution are being censoring because the sampled values exceed the E1921 limiting size-independent toughness value $K_{Jclim.}$ These simulations assumed that data was obtained on a 1T, W = 50.8 mm, a/W = 0.5 specimen with yield stress = 538 MPa. This effect dramatically reduces the ability of the annex procedure to distinguish the bimodal nature of the input data.

Figure 2 Frequency of inhomogeneity versus simulated test temperature and the size of the simulated data set.

The analysis was repeated for a wider, idealized bimodal distribution having T_a = -70°C , T_b = -110°C, and p_a = 0.75 (Figure 3). For this case, a 32 sample data set accurately predicts that the material is bimodal 90% of the time. As in Figure 2, censoring becomes important at -40°C, and the procedure much less accurately distinguishes the bimodal distribution. A narrower, idealized bimodal distribution having T_a = -80°C, T_b = -100°C, and p_a = 0.75 was also evaluated (Figure 3). For this distribution at least 64 specimen sample sets are required to correctly assess that the material is bimodal with a confidence of 80%.

Figure 3 Results for wider and narrower bimodal distributions.





There fore, the accuracy of the procedure decreases as the difference between T_a and T_b decreases. An equivalent implication is that more specimens are needed to determine that a material is bimodal as the difference between T_a and T_b decreases.

The idealized distribution having T_a = -80°C, T_b = -100°C, and p_a = 0.75 was also evaluated at T = T_a = -75°C as a function of the size of the sample data sets. Figure 4 depicts the T_a and T_b results for each single MC sample in these analyses. As the size of the sample data set is increased the variability of the estimated reference temperatures is reduced and the magnitude of the MLNH parameter is systematically increased. The 1000 sample data sets demonstrate that the MC procedure used in the analysis does converge such that the procedure always correctly identifies that the material is bimodal and that the idealized T_a and T_b results are obtained. Additionally, the average MLNH value properly approaches $|T_a - T_b|/4$ as would be expected from Eq. 7 for large data sets.

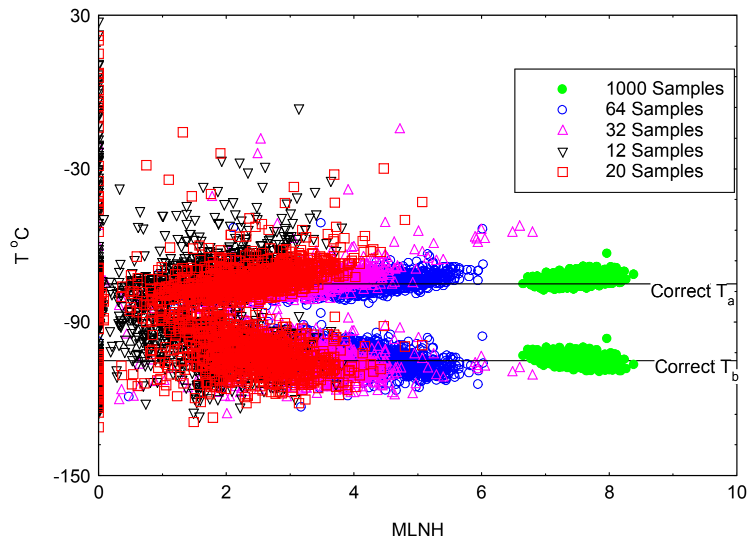


Figure 4 View of bimodal reference temperatures versus input values.

There are an infinite number of possibilities for evaluating idealized distributions by sampling data at more than one "test" temperature. Therefore, this study only evaluated the initial (Figures 1 and 2) idealized bimodal distribution and assumed that half of the samples were obtained at each of two temperatures. Figure 5 shows MLNH versus the number of data samples for several choices of data sample temperature pairs. The choice of the test temperature clearly affects the accuracy of the evaluation. This figure also illustrates that more than 32 total specimens are needed to ensure that the method accurately predicts that the material is bimodal at least 80% of the time. Further, as in the single temperature analysis, temperatures above T_a should be avoided because censoring then starts to dramatically affect the reliability of the procedure.

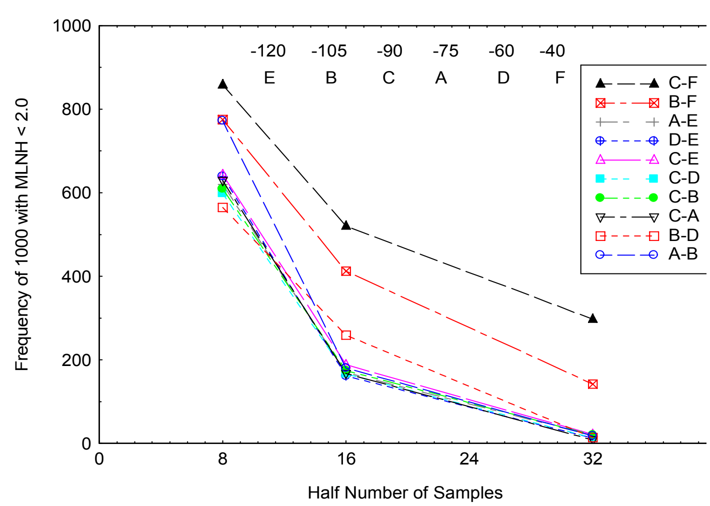
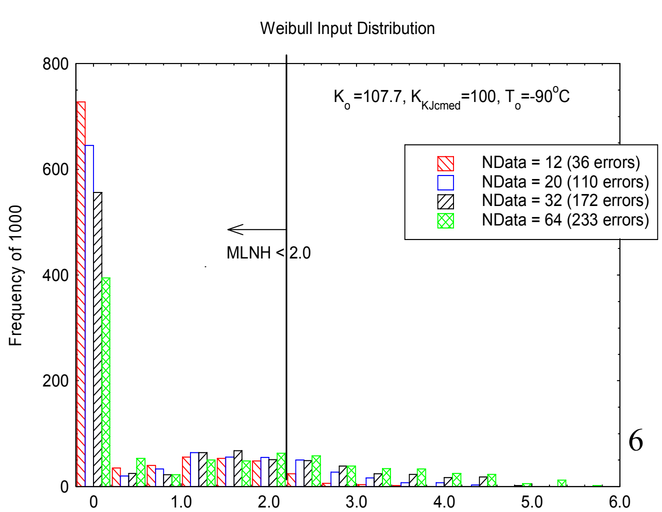


Figure 5 Results of the multi-temperature analysis.



4.0 Analysis of Idealized Homogeneous Material

Application of the proposed bimodal analysis also provides the possibility that a data set that is truly homogeneous can be interpreted to be bimodal. To investigate this possibility, the MC procedure described previously was used to evaluate an idealized homogeneous material to determine how often MLNH > 2.0. This outcome describes the number of times that this material is incorrectly identified as bimodal. The assumed homogeneous distribution was defined by T_o = -90°C. Once again, data sets with N = 12, 20, 32, and 64 were sampled at single temperatures. Steps 1) and 2) in Section 2.0 were followed to estimate T_a, T_b, and p_a for the data set at each of 1000 MC runs. The MLNH value was also determined as previously described. Typical results are shown in Figure 6. In this analysis, small data sets (i.e. N = 12) predicted that the material was bimodal less than 5% of the time. Rather surprisingly, as the data set size increased, the instances that the bimodal procedure incorrectly identified the material as bimodal also increased. For the 64 sample data set, the data was

Figure 6 Results of single modal input.

incorrectly classified over 20% of the time. This trend must be related to the higher likelihood that samples are obtained from both the upper and lower tails of the homogeneous distribution as the sample size increases. The bimodal procedure has no way to distinguish that these are from the tails and instead presumes that they are drawn from separate distributions.

5.0 Comparison with Experimental Results

Application of the bimodal method[3-4] to the European Round Robin Data[5] shows a wider range of T estimations than would be expected for a homogeneous material as defined by E1921. In previous work[4] the proposed bimodal analysis was applied to data reported in[5] as part of an extensive round robin evaluating the master curve approach on a forging steel. These results of the bimodal analysis are summarized for the 1T and 1/2T data sets that were part of this round robin in Tables 1 and 2 respectively.

When single temperature data sets composed of approximately 30 specimens were analyzed using the bimodal procedure, the data sets were found to be inhomogeneous only occasionally. However, when data sets were combined to include approximately 64 specimens, the results of the multi-temperature analyses consistently showed that the data sets were bimodal. The previous work [4] took a further scrutinized the Euro round robin material by testing additional specimens from adjacent slices so that the variation in toughness through the forging thickness could be assessed. These specimens were precracked Charpy specimens tested 10°C below the T_o temperature anticipated based on the previous Euro round robin specimen results. One result from [4] is reproduced here as Figure 7, and the E1921 calculations are presented in Table 3. Figure 7 shows that a large percentage of the results obtained on these specimens fall outside of the E1921 ±95% confidence bounds specified in E1921, even when accounting for the fact that the censoring limit (K_{Jclim}) is 142 MPa√m. The proposed bimodal analysis technique does not predict that either slice, individually, is bimodal, but the combined data set is clearly predicted to be bimodal. This result occurs because the material properties vary between slices N and P more than they vary within each slice. Hence, when the data from both slices are combined, the material inhomogeneity becomes clearer.

Table 1 Homogeneity Results, 1T Euro Forging Specimens

Temp. °C	N	r	T_o °C	T_a °C	T_b °C	p_a	MLNH	SX Blocks
-91	34	34	-97.2	-74.2	-98.6	0.23	2.35	8
-60	34	34	-87.5	-87.5	--	1	--	10
-40	32	31	-91.	-46.2	-91.2	0.01	--	12
Multi-Temp	68*	68	-91.	-83.9	-97.9	0.61	2.25	8, 10

* -40 °C data not included since $T - T_o > 50$ °C.

Table 2 Homogeneity Results, 1/2T Euro Forging Specimens

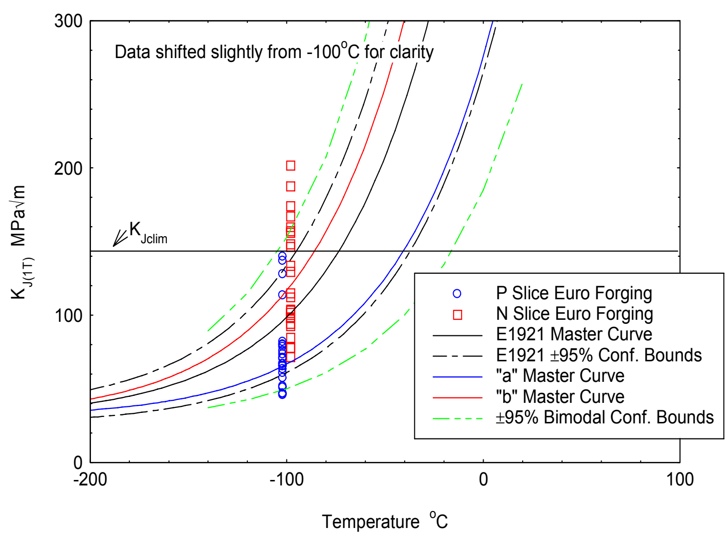
Temp. °C	N	r	T_o °C	T_a °C	T_b °C	p_a	MLNH	SX Blocks
-110	55	55	-85.0	-71.3	-86.9	0.20	1.78	1, 2, 4
-91	31	31	-90.9	-90.2	-91.	0.17	0.07	8
-60	31	29	-79.1	-73.3	-96.0	0.82	1.61	10
-60	31	22	-92.7	-74.2	-97.1	0.26	2.12	2, 9 from 2T C(T)
-60	62	51	-86.6	-76.4	-103.8	0.69	3.32	2,9,10
Multi-Temp	148	137	-86.4	-71.5	-90.2	0.29	3.38	1, 2, 4, 8, 9, 10

Table 3 Homogeneity Results, Precracked Charpy Euro Forging Specimens

Temp. °C	N	r	T_o °C	T_a °C	T_b °C	p_a	MLNH	Layers	Slices
-100	48	34	-99.0	-66.2	-111.4	0.42	5.63	1-6	N, P
-100	24	13	-109.2	-109.3	-109.2	1.0	--	1-6	N
-100	24	21	-84.5	-62.8	-121.1	0.82	--	1-6	P

This result is corroborated by the individual data for each slice (Figure 7). Clearly, the larger, bimodal-material confidence bounds correspond much better to the observed data than do the homogeneous-material, E1921 confidence bounds.

Basically, slice N is homogeneous because 11 of the 24 specimens are censored, and these contain all the "b" distribution data, while the P slice is found to be inhomogeneous because it has too few of the "b" distribution data, causing $(r - N^*p_a - 2) < 0$ and thus neither σ_{Tb} or MLNH can be evaluated for this case. When the data sets are combined, the larger size of the data set allows the evaluation of MLNH and the data is then found to be strongly inhomogeneous, a result that compares with intuition when viewing Figure 7. The larger confidence bounds, including the effect of the bimodal distribution,



correspond much better to the observed data, as shown in Figure 7.

Figure 7 Master curve plot of precracked Charpy data

7.0 Discussion

This analysis raises important questions about how to analyze the homogeneity of test specimen data with the Master Curve approach and use this data in structural analysis. One possible approach could be to always assume that a material is inhomogeneous, or bimodal, unless enough data is available to confidently demonstrate otherwise. With this approach, applicable T_a and T_b values could be determined and structures could be conservatively analyzed by assuming that T_b describes the material performance. Conservatism could be relaxed as the knowledge and confidence in the materials properties and the stress throughout the structure are known.

A more urgent concern is that the current E1921 presumes that a material is homogeneous and only requires a minimum of six specimens in the analysis. This work clearly demonstrates that this is an inadequate number of specimens to assess homogeneity strictly by data analysis. Currently, E1921 provides little guidance or requirements for the user to assess homogeneity. Further, the apriori assumption that a material is homogeneous could lead to an unconservative estimation of material performance if the material is actually inhomogeneous.

Therefore, the E1921 standard should be revised to provide clear criteria and guidance for evaluating material inhomogeneity with a high degree of certainly. Future work that extends the analysis in this paper is needed to options for this criteria and guidance. Then, the broad community of E1921 users will need to assess these options choose the appropriate strategy which satisfies practical evaluation constraints yet still provide sufficient confidence in application of the data. One possible option would be to couple knowledge of the microstructural, alloying, and processing differences throughout the material with the Master Curve analysis to more rigorously assess material homogeneity.

8.0 Conclusions

Based on the analysis conducted in this study, the following conclusions can be drawn:

1) Relatively large data sets are required to determine with reasonable accuracy whether a data set represents a bimodal material. The proposed number of 12 to 16 is inadequate and a number between 30 and 60 is needed to ensure that the MLNH > 2 criterion is accurate at least 80% of the time.

2) The use of multi-temperature data does not decrease the need for testing between 30 and 60 specimens.

3) The allowed temperature range suggested by the proposed bimodal procedure is too wide. This study suggests that the specimen test temperature range should correspond to [T_b +50°C, T_a -50°C], where $T_b \leq T_a$.

4) The characterization of idealized bimodal material is less accurate as the amount of censored data increases. Therefore, testing temperatures and/or specimen sizes should be appropriately chosen for the material being evaluated.

5) When an idealized homogeneous material is analyzed using the bimodal procedure, the material is typically accurately characterized as homogenous using small data sets (i.e., N = 12). However, the accuracy decreases as the data set size increases due to the increased likelihood

that the upper and lower tails of the idealized homogeneous distribution are sampled in the analysis.

6) The large number of specimens required to assess material inhomogeneity predicted by this MC analysis general agrees with experimental results reported earlier on the Euro round robin forging steel.

7) Future work is necessary to develop criteria and guidance for assessing material inhomogeneity with a high degree of confidence within the E1921 standard. Options for revising the standard need to be developed and assessed by a broad community of E1921 users to determine which option satisfies practical evaluation constraints yet still provides sufficient confidence in application of the data.

9.0 References

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