Study of Ductile-Brittle Transition Temperature of 9Cr-1Mo Steels

EXECUTIVE SUMMARY

Defining the ductile to brittle transition temperature (DBTT) below which catastrophic failures occurring in structures made of ferritic steels is a major technological challenge for designers. Generally practiced empirical approaches (ASME Kc Curve) to predict the fracture toughness over the DBTT regime fail to appreciate the scatter, originating from the random distribution of cleavage crack initiation sites, and often lead to ultra-conservative predictions. Recently a ‘weakest link’ based statistical approach to evaluate DBTT via a reference temperature (T0) prediction (ASTM E 1921) and an associated Master Curve description claims to be more appropriate, however its applicability at high loading rates (dynamic) is yet to be resolved. This aspect has been studied with 9Cr-1Mo and Modified 9Cr-1Mo steels, the candidate FBR wrapper materials, using dynamic fracture toughness results obtained from instrumented impact tests.

OUTLINE

The 9Cr-1Mo and the modified 9Cr-1Mo steels are candidate wrapper materials for the Fast Reactors in next generation. However, the irradiation induced rise in the Ductile-Brittle Transition Temperature (DBTT) is a major technological challenge. Towards the DBTT evaluation, the recently developed Weibull statistics based ASTM E-1921 reference temperature (T0) approach has been applied but under dynamic loading condition, as it is understood that the DBTT is sensitive to loading rate for this type of materials. The dynamic loading rate would provide a more conservative prediction of the DBTT as compared to the quasi-static loading rate, important for the material's selection for this critical component.

Dynamic fracture toughness of these materials has been determined from instrumented impact testing with pre-crackled Charpy specimens. The dynamic loading gives rise to inertial oscillations in the specimen originating from interactions of forward and reflected stress waves. Towards getting a load signal with minimum oscillation effects, testing has been conducted at reduced hammer (~1.12 m/s). Even this loading rate provides a stress intensity factor rate ~106 MPa.m1/2/s, which is well within the dynamic regime. It has been observed that a full velocity of Charpy machine (5.12 m/s) affects the stress intensity factor rate only marginally.

The applicability of the quasi-static shape of Master Curve, as provided in ASTM E-1921, under this high strain rate has been a matter of speculation. This aspect has been experimentally verified with a large number of PCVN testing for 9Cr-1Mo steel at a temperature range well within the ductile-brittle transition regime. At temperatures of ~60, ~55, ~52.5, ~50, ~47.5, ~45 and ~40 °C, reference temperature determined from dynamic fracture toughness datasets (termed as T0%) have been ~47.15, ~45.75, ~62.09, ~64.13 and ~63.47 °C respectively. Taking into account the experimental uncertainties involved in a dynamic fracture toughness evaluation procedure, it is reasonable to say that the T0% remains practically unchanged (±10 °C) within the DBTT regime. Thus, it is proposed that the same shape of the Master Curve describing the fracture toughness variation under quasi-static condition also can be applied in the high strain rate loading. The T0% of the 9Cr-1Mo steel is conservatively predicted as ~45 °C and the same has been chosen to index the Master Curve, as shown in Fig. 1. Except a few, the individual dynamic fracture toughness data has been seen to lie well within the 98% and 2% confidence limit. The ASME-Kc curve for this steel shows much lower fracture toughness as compared to the 2% confidence limit.

Compared to the 9Cr-1Mo steel, the modified 9Cr-1Mo steel yields a higher T0% (15 °C). It is attributed to the change in crack initiation mechanism from carbide-matrix decohesion (for the 9Cr-1Mo steel) to cracking of prior austenitic grain boundary (for the modified 9Cr-1Mo steel). The corresponding fractographs are shown in Figure 2. The change in mechanism is attributed to grain boundary embrittlement phenomenon in the latter, originated from Phosphorus segregation at the prior austenitic grain boundaries. The Phosphorus segregation at the prior austenitic grain boundaries has been confirmed by the Secondary Ion Mass Spectrometer (SIMS) study.
The fracture of ferritic steels in the DBTT regime is generally predominated by initiation controlled cleavage cracking. As the cleavage initiation is often dictated by availability of a weakest link (brittle precipitates, inclusions, weak lath boundaries etc.) in the region of highest stress buildup, the ‘local approach’ is proved to be more meaningful and thus the micro-cleavage fracture stress ($\sigma_f^*$) is the controlling material parameter in this regime.

In pursuit of a model to explain relationship between the microscopic cleavage fracture and the macroscopic fracture toughness, Ritchie, Knott and Rice have suggested (known as RKR Model) that the applied tensile stress has to exceed the critical cleavage fracture stress, $\sigma_f^*$, over a critical microstructural distance, $l^*$. $l^*$ can be estimated from the separation between the crack tip and appropriately configured micro-crack nucleation sites under SEM. A schematic representation of the cleavage cracking is shown in Fig. 3.

Based on the finite element based assessment of stress distribution ahead of a crack, the following equation may be used to estimate $\sigma_f^*$:

$$\sigma_f^* = \sigma_Y A_i (B_i l^*) / K$$

where, $K$ is the static fracture toughness and $\sigma_Y$ is the static yield stress. $A_i$ is a unit less constant and $B_i$ is a constant having unit of stress (MPa) depending on the strain hardening exponent, $n$.

### ADDITIONAL INFORMATION ABOUT $T_{\text{c}}$ AND THE MASTER CURVE

The ASTM E-1921 requires the $T_{\text{c}}$ to be determined from fatigue pre-cracked specimens. Under dynamic loading condition this demands a precise temperature control and strict testing protocol to generate valid fracture toughness from generally small size (10x10x55 for PCVN) specimens. Thus a parallel testing and analysis methodology is under development which would enable determination of a conservative $T_{\text{c}}$ by conducting instrumented Charpy-V notch tests only. This involves predicting dynamic fracture toughness from Charpy energy via semi-empirical correlations over a temperature range within the DBTT regime and then using multi-temperature approach to determine the $T_{\text{c}}$ as given in ASTM E 1921. For 9Cr-1Mo steel, this aspect has been successfully verified.

### GENERAL EXPLANATION RELATED TO THE DESCRIPTION

The fracture of ferritic steels in the DBTT regime is generally predominated by initiation controlled cleavage cracking. As the cleavage initiation is often dictated by availability of a weakest link (brittle precipitates, inclusions, weak lath boundaries etc.) in the region of highest stress buildup, the ‘local approach’ is proved to be more meaningful and thus the micro-cleavage fracture stress ($\sigma_f^*$) is the controlling material parameter in this regime.

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### BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The brittle fracture originates at the weakest link ahead of a sharp crack. A 3-parameter Weibull distribution: $p_i = 1 - \exp\{-[((K_i - K_{\text{min}})/(K_i - K_{\text{min}}))]^{n_i}\}$, is used to model scatter in fracture toughness in DBTT regime and the temperature at which the median fracture toughness (where cumulative probability of failure is 0.5) for 1 inch thick specimen is 100 MPa.m$^{0.5}$, is defined as the reference temperature ($T_i$). $T_i$ is a material specific parameter and used to index the Master Curve, claiming to describe the fracture toughness variation of all the ferritic steels in the DBTT regime. The Master Curve equation is $K_f^*(\text{median}) = 30 + 70\exp(0.019(T-T_i))$.

### ACHIEVEMENT

This study extends the scope of ASTM E-1921 Master Curve description to high strain rate regime, thus enabling a conservative prediction of the DBTT of ferritic steels by taking into account the strain rate effect. The effects of trace elements like Phosphorus on the crack initiation mechanism vis à vis on DBTT has been understood.

### PUBLICATIONS ARISING OUT OF THIS STUDY AND RELATED WORK


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