HALL-PETCH DEPENDENCE OF J-FRACTURE TOUGHNESS PARAMETERS FOR A RPV STEEL - PART II: QUENCHED AND TEMPERED MICROSTRUCTURES

José Ricardo Tarpani

Materials, Aeronautical and Automotive Engineering Department, Engineering School of São Carlos – University of São Paulo – Brazil

jrpan@sc.usp.br

Waldek Wladimir Bose Filho

Materials, Aeronautical and Automotive Engineering Department, Engineering School of São Carlos – University of São Paulo – Brazil

waldek@sc.usp.br

Dirceu Spinelli

Materials, Aeronautical and Automotive Engineering Department, Engineering School of São Carlos – University of São Paulo – Brazil

dspinell@sc.usp.br

Abstract. The elastic-plastic fracture toughness and crack extension behavior under quasi-static loading regime of several microstructures of a reactor pressure vessel steel were assessed on the basis of microstructural parameters. J-integral fracture toughness testing were conducted at 300° via the unloading elastic compliance technique. Miniaturized compact test specimens, 0.2 and 0.4 T C(T) (5 and 10mm-thick, respectively), were machined from thick forged plates (T/4 position, T-L orientation) of the microalloyed steel in the as-received and the several thermally embrittled conditions. It has been verified that the bainite packet size of single-phase quenched and tempered microstructures controls their J-fracture toughness properties. Results have been found in close agreement to those obtained in a parallel study with dual-phase annealed microstructures derived from the same low alloy steel. Similarly to annealed materials, it has been concluded that a Hall-Petch relationship correlates J-fracture toughness parameters to the grain size. These conclusions have been shown to hold regardless the J-R curve fitting method adopted, namely the widely used power-law, the here proposed logarithmic fit and old-dated linear fitting method.

Keywords. fracture toughness, Hall-Petch relationship, J-R curve, RPV steel, thermal embrittlement.

1. Introduction

In a companion paper, Tarpani et all (2003) submitted a nuclear grade steel to annealing heat treatments devised at obtaining a range of low to intermediate elastic-plastic fracture toughness in the quasi-static loading regime. Thermal cycles were designed to simulate the mechanical behavior of structural steels undergoing high neutron dose damage in radioactive environments, e.g. reactor pressure vessel (RPV) steels.

By means of a simple rule of mixture, it was concluded that the equivalent grain size of the dual-phase annealed microstructures controlled overall J-fracture toughness properties. The procedure was based on the relative percentage of both phases present in the heat treated alloy, similarly to that applied on the inference of mechanical properties of composite materials.

None the less, it became clear that, despite the broad range of fracture toughness achieved, annealing heat treatments did not generate microstructures with hardness and tensile properties, viz. yield and ultimate tensile strengths, closely comparable to neutron damaged low alloy structural steels. For instance, yield strength increases up to 150 MPa have been reported by Onizawa & Suzuki (1997) for RPV steels in the early life stages of commercial nuclear power plants. However, just a fraction of this value was obtained by Tarpani et all (2003) by means of the severest annealing heat treatment. So, alternative quenching and tempering (Q&T) routes were designed and applied to the original RPV steel in order to generate some microstructures that minimally satisfied those mechanical requirements.

2. Base material

The Brazilian ASTM A508 Class 3A steel is a typical RPV material for the nuclear industry, and has been fully described in Part I by Tarpani et all (2003).

3. Experimental and analytical procedures

3.1 Quenching and tempering thermal cycles

Figure (1) shows schematic drawings of the six Q&T embrittling heat treatments, named from I to N, which were individually applied to the original A508 steel.

3.2 Conventional mechanical properties and J-fracture toughness test

Hardness measurements at ambient temperature and tensile tests at 300°C, were carried out for all Q&T embrittled materials.

Likewise performed in Part I by Tarpani et all (2003) for the annealed and as-delivered conditions of the A508 steel, fracture toughness J-R curve testing were conducted at 300°C via the unloading elastic compliance (UEC) technique, using miniaturized 5 mm and 10 mm-thick compact tensile specimens, 0.2 T and 0.4 T C[T], respectively. Procedures were identical as in Part I, concerning the tasks of J- Δa data points determination, data fitting and extrapolation, as well as for deals of deriving the initiation J value, J_i, the ductile tearing instability parameter, J₅₀, which has been previously defined in Part I of this work, and the rate of increase on deformation-J crack growth resistance at 1 mm of crack extension, dJ_D/d $\Delta a_{(1mm)}$.



Figure 1. Schematic drawings of Q&T routes aimed at producing thermally embrittled microstructures (I-N) obtained from the original A508 steel (A). (*Continued*)



Figure 1. (Continued).

4. Results and discussion

4.1. Microstructural characterization

Shown in Fig. (2a) is microstructure I, which is comprised of low carbon martensite, slightly decomposed in ferrite and carbides due to mild tempering. Retained austenite is observed as small acicular areas. Mn and C segregation at grain boundaries caused intergranular fracture mode during J-R curve testing. Microstructure J, Fig. (2b), presents heavily spheroidized carbides, due to hard tempering. Recrystallization, grain homogenization and refinement thermal cycles, sequentially applied to J, favored bainite transformation and prevented austenite retaining. Microstructure K, in Fig. (2c), exhibits a complex arrangement of several phases, that is retained austenite, tempered martensite and bainite, as well as a mix of ferrite and coalesced carbides. Cleavage interceded in both tensile and J-fracture toughness testing of this material, possibly due to large amounts of retained austenite.





Figure 2. Light micrographs of materials tested: (a) I, (b) J (c) K, (d) L, (e) M, (f) N. Etched in Nital 2%.

Microstructure L, Fig. (2d), exhibits some morphological similarity with microstructure J, but a coarser aspect is clearly noticed on it due to its heavier austenitizing conditions, and the absence of grain refinement and homogenization thermal cycles. Microstructure M, Fig. (2e), shows great similarity to microstructure I, presenting too alloy segregation at the grain boundaries and intergranular crack propagation during J-fracture toughness testing. Microstructure N, Fig. (2f), underwent the same Q&T thermal cycles applied during fabrication of the original A508 steel (A), except by the subsequent post-welding heat treatment applied to the latter. Thus, a similar mechanical behavior was obtained for A (Part I, Tarpani at all, 2003) and N. Table (1) supplies final bainite and martensite packet sizes of Q&T materials tested. Hardenability clearly depends on the prior austenite grain size achieved during the thermal cycles, with martensite invariably deriving from the largest ones, whereas bainite originated from intermediate and fine grained austenite phase.

Table 1. Microstructural parameters of Q&T materials. ASTM grain size is provided in brackets.

Quenching and Tempering Route	D _{bainite/martensite} (µm)		
A (as-received)	19 (8.5)		
Ι	775 (00)		
J	105 (3.6)		
K	119 (3.2)		
L	153 (2.5)		
М	589 (00)		
Ν	25 (7.7)		

4.2 Hardness and tensile properties

Table (2) lists conventional mechanical properties of the obtained materials. It can be seen that hardness, yield and ultimate tensile strengths, S_Y and S_U , values of Q&T products are higher than those determined in Part I for the annealed A508 steel. Therefore, it can be concluded that Q&T treatments are more efficient than annealing in simulating hardening and strengthening effects developed in nuclear steels due to neutron irradiation damage.

Table 2. Mechanical properties of as-received (A)	and thermally embrittled materials (I-	-N)).
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Q&T Route	Brinell (100kgf)	S _Y (MPa)	S _U (MPa)	EL (%)	RA (%)
A	175	400	555	11	77
Ι	340	1040	1120	10	75
J	269	725	865	07	61
Κ	360	970	1145	11	77
L	246	700	810	08	44
М	359	920	1195	10	70
Ν	212	535	700	11	73

As mentioned earlier, tempered martensite microstructures I, K e M exhibited brittle behavior, so they were not considered further in this study. Overall mechanical properties confirmed that tempered bainite microstructures J, L and N simulate more properly the as-irradiate state performance of the RPV steel.

Tables (1) and (2) show that microstructures A, J, L and N present an inverted correlation between reduction in area at fracture, RA, and the mean size of the bainite packet, D_{bainite} . Figure (3a) displays the relationship between RA and the inverse of squared root D_{bainite} , where good data correlation is noticed, as denoted by the coefficient *r*. Similar results obtained in Part I for annealed microstructures are provided in Fig. (3a) to allow the comparison between both thermal approaches.

The notation RCS, representative cell size, will denote hereafter the significant cell size of a particular microstructure, namely the equivalent grain size, EGS, adopted in Part I for dual-phase ferrite/bainite annealed products, and D_{bainite} for Q&T ones. Therefore, the RCS concept intends to denote the significant size of the cell controlling the fracture toughness parameters of the materials tested.

Likewise demonstrated in Part I for annealed microstructures, Fig.(3a) shows that RA and RCS values for Q&T materials also obey a Hall-Petch relationship (Hall, 1951 & Petch, 1953), as follows:

$$RA = a + b \cdot RCS^{-0.5}$$
(1)

where *a* and *b* are fitting constants.

As already found in Part I, Eq. (1) indicates that RA is grain-size driven for Q&T microstructures too. The same dependence will be shown regarding their J-fracture toughness properties.

Displayed in Fig. (3b) is the relationship between the true fracture strain, ε_f , of tensile specimens and the RCS value, where ε_f was obtained from RA as follows:

$$\varepsilon_{\rm f} = [1 - (\rm RA/100)]^{-1} \tag{2}$$

A Hall-Petch dependence of ε_f on RCS is clearly noticed in Fig. (3b), corroborating previous findings by Armstrong (1997).



Figure 3. Hall-Petch relationship for the reduction in area (a) and true fracture strain (b) dependence on grain size for the materials tested. Previous results from Tarpani et all (2003) for annealed materials are also supplied.

4.3 J-R curves and fracture toughness parameters

Figure (4) presents typical J- Δa data points for all Q&T material conditions. In general, extensive ductile crack growth is observed, typically in the range 2.5-4 mm, as required for the determination of J_i, J₅₀ and dJ_D/d $\Delta a_{(1mm)}$ parameters, previously defined in Part I. The effect of deeper side-grooving level (33%SG, in Fig. 4b, against 20% SG, in Fig. 4a) in steeping down the J-R curves is clearly noticed.

Observe that the rank established for RA, in terms of bainite packet size (see Fig. 3), is faithfully obeyed for the J-R curves positioning, signalizing that RCS also rules the ductile crack growth resistance of Q&T microstructures.

Besides the conservatism of J-R curves derived from deeper side-grooved test specimens, Fig. (4b) also displays a J-specimen size dependence for microstructure J. A thinner testpiece originated a less conservative J-R curve due to the loss of plastic constraint, i.e. triaxial stress state relaxation along crack leading edge, identically as verified in Part I, related to annealed microstructure G.



Figure 4. Typical J- Δa curves of materials tested. (a) 20% SG, (b) 33% SG testpieces, which are 10 mm-thick unless otherwise indicated. J-R curves of original steel (A) obtained in Part I are drawn as baseline.

In Fig. (5), this dependence is shown according to a Hall-Petch relationship, which has already been proven valid in Part I for duplex annealed microstructures of the RPV steel, given by:

$$J = c + d. RCS^{-0.5}$$

where *c* and *d* are fitting constants.

Figure (5) unequivocally depicts the conservative aspect of the logarithmic fit of $J_{-\Delta a}$ data points in generating lower J_i and J_{50} values, as compared to power-law and mainly linear data fitting. Observe that this conclusion does not apply to the $dJ_D/d\Delta a_{(1mm)}$ criterion. In fact, an inverted behavior is noticed regarding the latter fracture-toughness parameter. The same figure confirms earlier predictions that more conservative J-criteria are determined by testing deeper side-grooved test specimens (Fig. 4). As previously mentioned (see Fig. 4b), a slight specimen-size effect is also observed in Fig. (5), when thinner J-testpiece of microstructure J conducted to less conservative J-criteria results. Note that this effect is almost imperceptible for the $dJ_D/d\Delta a_{(1mm)}$ criterion.



Figure 5. Relationships between J-parameters and equivalent cell size, according to Eq. (3). (a) Crack initiation J_i ; (b) Crack instability J_{50} , and (c) Rate of cracking resistance $dJ/d\Delta a$. Test specimens are 10mm-thick unless otherwise specified.

Correlation between elastic-plastic fracture mechanics properties and grain size has been proposed in the literature by Srinivas et all (1987, 1991), invariably on the basis of the widely accepted Hall-Petch relationship and for single-phase non-structural metallic materials only. The present study has strengthened the validation of such an approach for a broad range of grain sizes in single-phase microstructures of a structural nuclear grade steel. It should be recalled that in a companion paper by Tarpani et all (2003) the same behavior was observed regarding to the equivalent grain size of dual-phase annealed microstructures.

5. Concluding remarks

The elastic-plastic fracture toughness and crack extension behavior under quasi-static loading regime of singlephase Q&T microstructures of a RPV steel were assessed on the basis of microstructural parameters.

It has been concluded that grain size does control the J-fracture properties of the materials tested. The dependence of J-parameters on bainite packet size obeys a Hall-Petch relationship, confirming scarce propositions available in the literature.

The results, which have been shown to hold disregarding the adopted J-R curve fitting method, agree very well with trends observed in a parallel research conducted by Tarpani et all (2003) in annealed microstructures obtained from the same low alloy steel.

Hence, the whole study has enlarged the possibilities of obtaining a broad range of fracture toughness from a nuclear grade steel, as a way to simulate its mechanical behavior, viz. fracture toughness, during in-service neutron exposed conditions. This procedure can lead to considerable simplification, cost and timesavings, as well as risk reduction in periodic inspection programs and related subjects of nuclear power industry.

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