Communication

Effect of Temperature on the Fracture Toughness of Hot Isostatically Pressed 304L Stainless Steel

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Herein, we have performed J-*Resistance* multi-specimen fracture toughness testing of hot isostatically pressed (HIP'd) and forged 304L austenitic stainless steel, tested at elevated (300 °C) and cryogenic (− 140 °C) temperatures. The work highlights that although both materials fail in a pure ductile fashion, stainless steel manufactured by HIP displays a marked reduction in fracture toughness, defined using $J_{0.2BL}$, when compared to equivalently graded forged 304L, which is relatively constant across the tested temperature range.

Hot isostatic pressing (HIP) is a component manufacturing technique, which employs the use of high temperature, and isostatically controlled pressure to consolidate metal alloy power of desired chemistry into bulk metal under an inert (usually argon) atmosphere.[1] The advantages of HIP are well documented,[1–4] the most significant being within HIP's ability to produce near-net shape components; components with exceedingly complex geometries thus eliminating the need for subsequent machining/welding procedures on the manufactured component. This may not only reduced the costs associated with the overall manufacture process, but through the elimination of welded joints, produces components of homogenous metallurgy; omitting common issues associated with welding of components; hot cracking, different metallurgical zones, induced residual stresses, *etc*. This is clearly an advantage for components which will be subjected to high stress conditions throughout their lifetime. The degree of metallurgical homogeneity which HIP produces results in no grain directionality, like that commonly seen in forgings, due to the isostatically controlled pressure and temperature and therefore HIP materials display isotropic mechanical properties. Finally, HIP produces material with a comparatively smaller grain size than that of forgings and castings, which not only improves the yield strength and ultimate tensile strength, also lends itself to easier inspection view non-destructive examination techniques.

Because of HIP's ability to increase design freedom, there have been increased efforts to demonstrate that components produced by HIP have equivalent or better material properties than those of equivalently graded forged materials. However, the authors have recently shown that the fracture behavior is subtly different between equivalently graded HIP and forged austenitic stainless steel, with HIP 304L and 316L exhibiting a reduction in impact toughness[5,6] as well as HIP 304L exhibiting a reduction in J-integral fracture toughness at ambient temperature.[7,8] This difference in fracture behavior was attributed to the presence of a comparatively large volume fraction of non-metallic oxide inclusions in the HIP microstructure, which lower the energy required to cause fracture *via* an unzipping effect, whereby ductile void growth is unable to occur on the same scale as in forged stainless steel, resulting in premature microvoid coalescence with neighboring voids. Thus, it was shown that the impact toughness was governed by the concentration of oxygen remaining in the austenite matrix.

The authors' previously reported work has demonstrated that stainless steel manufactured by HIP can display a clear and significant reduction in Charpy impact toughness over a temperature range of − 196 °C to + 300 °C when the oxygen concentration is not properly controlled.[5–7] This Charpy impact toughness behavior was also found to extend to J-*R* fracture toughness at ambient temperature[7,8]; for completeness, these data have also been included here. The mechanism by which Charpy impact and fracture toughness is reduced, is governed by the volume fraction and distribution of non-metallic oxide inclusions that are present in the austenite microstructure. These oxide inclusions act as initiation sites for the nucleation, growth, and coalescence of voids during plastic deformation, and are believed to originate in such concentrations as a result of spontaneous surface oxidation of the metal powder during handling and storage.

We have completed the studies by performing J-*R* fracture toughness testing at elevated (300 °C) and cryogenic (− 140 °C) temperatures, to determine whether the Charpy impact toughness phenomena translate to high-constraint fracture toughness testing across a comparable temperature range. For additional reference, tensile testing has also been performed at the same test temperatures.

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The present study is based on industrially supplied HIP'd 304L material from AREVA, and industrially supplied 304 L stainless steel from Creusot Forge et Creusot Mécanique, AREVA, France. The mean grain size of the HIP304L material was measured using the linear intercept method, as per ASTM E112-96,[9] and was determined to be 27 μm, with a standard deviation of 9. The F304L material had a mean grain size of 94 μm, with a standard deviation of 14.

Table I shows the elemental compositions of the forged and HIP materials, as well as the elemental composition of the respective powders from which the HIP materials were manufactured. All materials are within specification. The HIP materials display higher oxygen content by nearly one order of magnitude. For HIP304L, 304L powder was heated from ambient temperature to 1150 °C at a rate of 360 °C h^{-1}, and held at 1150 °C and 104 Mpa for a period of 180 minutes. Cooling was performed at a rate of 240 °C h^{-1}. Post-HIP heat treatment of HIP304L was performed by heating from room temperature to 1070 °C at 360 °C h^{-1}, held for 280 minutes, and water quenched. Forged 304L pipe was subjected to similar heat treatment as the HIP materials (1070 °C, for ca. 250 minutes) and water quenched.

Compact tension (C(T)) specimens were machined in accordance with ASTM A370 recommended[9] dimensions (60 × 60 × 25 mm), and the notch was machined by Electric Discharge Machining (EDM). F304L C(T) specimens were extracted from a pipe section and machined with C-R orientation, where C = circumferential direction and R = radial direction. HIP C(T) specimens were extracted from square blocks and machined with L-T orientation, where L = longitudinal direction and T = transverse direction, though the isotopic nature of the HIP grain structure means that the orientation of HIP specimens is not important. Fatigue pre-cracking of C(T) specimens was performed at Amec Foster Wheeler (Warrington, UK), using five pre-cracking steps and maintaining a low stress intensity (< 20 MPa m$^{1/2}$) during final crack growth to a final a/W of ca. 0.55, in accordance with ASTM E1820,[10] and side grooved using a 90-deg angle. Precise values of total crack length were measured metallographically, after fracture via fatigue cracking (labeled fatigue-post crack on Figure 1). Fracture toughness data were measured using a multi-specimen approach (5 to 7 specimens), and a J-R curve obtained as a power law regression trend through the valid data points, in accordance with ASTM E1820.[10] J_{max} values were calculated in accordance with ASTM E1820.[10] Fracture toughness was conducted in displacement control, with a load rate of 1 mm min^{-1} until sufficient clip

gauge opening was observed. Specimens were heat tinted at 640 °C for 1 hour and opened by fatigue cracking, after which final crack lengths were measured metallographically.

Fracture toughness was conducted in displacement control, with a load rate of 1 mm min^{-1} until sufficient clip gauge opening was observed. Specimens were heat tinted at 640 °C for ca. 1 hours and opened by fatigue cycling, after which final crack lengths were measured metallographically.

F304L tensile test specimens were extracted in the longitudinal (rolling) direction of a forged plate. HIP304L tensile test specimens were extracted in the axial direction of the HIP cylinder. Round bar tension test specimens were machined in accordance with ASTM E8/E8M recommended dimensions, with a gauge length of 50 mm, gauge diameter of 6 mm, and M12 thread.[11] Tensile testing was conducted in displacement control at a strain rate of 0.5 min^{-1}. Three tensile tests were performed for each material and condition; the results shown are representative of the three tests.

Figure 1 shows the engineering stress vs strain data obtained from the tensile tests. The data recorded at − 140 °C exhibit a transition in the strain hardening behavior, and this is associated with strain-induced martensitic transformation during loading. This is consistent with previous work,[5] in which martensitic transformation was observed at the tip of the V-notch in failed 304L Charpy test specimens tested at − 196 °C. The well-known effect of temperature on yield strength[12] and UTS[13,14] is also observed, whereby strength increases with decreasing temperature. The effect of test temperature on elongation is also notable; the tensile data are consistent with other reported data.[15] In all cases, HIP304L exhibits a slight improvement in strength, but a slight reduction in ductility, as can be seen in the overall recorded elongation. The improved strength is associated with HIP's finer grain size (27 μm cf. 94 μm).

Figures 1, 2, and 3 show the fracture toughness test data for ambient, − 140 °C and 300 °C, respectively. In all cases, HIP304L displays a marked reduction in both the crack initiation toughness, determined via the intersection of the power law regression fit with a 0.2 mm blunting line, constructed in accordance with ASTM E1820.[10] HIP304L also displays a clear reduction in the resistance to crack propagation, indicated via the smaller gradient of the powder law regression fits. Table II shows the measured values of initiation toughness ($J_{0.2BL}$) for HIP304L and F304L at each test temperature. The reduction in toughness is expressed as a ratio between F304L and HIP304L at each respective test temperature.

Table I. Elemental Composition

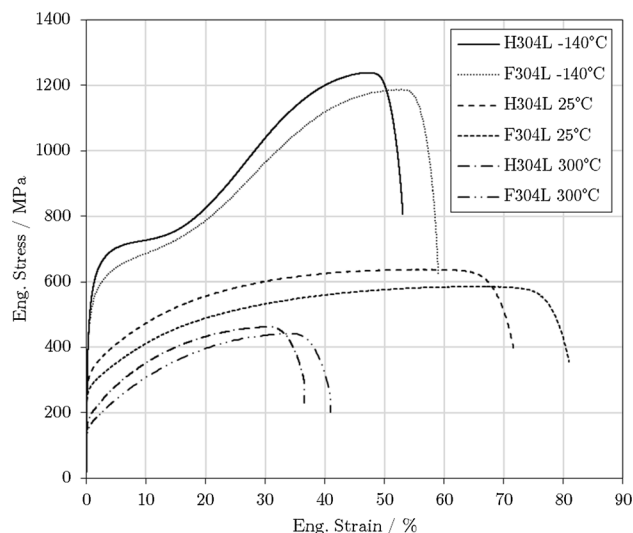| 304L	Grain size	Cr	Ni	Mo	Mn	Si	C	O/ppm	N/ppm
Spec. (Weight Percent)	—	18.5 to 20.00	9.00 to 10.00	—	< 2.00	< 1.00	< 0.035	200	—
Forged	94 μm	19.40	9.65	0.345	1.65	0.57	0.027	15	817
Powder (Weight Percent)	—	19.2	9.44	—	1.37	0.74	0.022	110	—
HIP (Weight Percent)	27 μm	19.5	9.45	0.01	1.33	0.72	0.022	120	840

Fig. 1—Tensile test data for HIP and forged 304L at three test temperatures.

The data herein show that previous observations of Charpy impact toughness behavior[5–8] are translatable to J-R fracture toughness testing. HIP304L, containing 120 ppm oxygen in the bulk material, exhibits a reduction in fracture toughness ($J_{0.2BL}$) by approximately 40 pct at ambient and 300 °C. This reduction in fracture toughness is more pronounced at − 140 °C, where a reduction of approximately 50 pct is observed. The mechanism by which fracture toughness is reduced is the same mechanism by which oxygen concentration affects Charpy impact toughness of HIP stainless steel[5–7]: non-metallic oxide inclusions act as initiation sites for the nucleation, growth, and coalescence of voids during failure. It is interesting to note here that previous studies on Charpy behavior were unable to explicitly reveal quantitative data at elevated temperature, because Charpy test pieces did not fail completely as a result of excessive plastic deformation of the test pieces,[6,7] and because of this, HIP and Forged variants of stainless steel exhibited comparable Charpy toughness when tested at 300 °C. We have shown here, however, that under high-constraint testing conditions, where the plastic zone is localized to a relatively small region ahead of the crack tip, that a comparable level of reduction in toughness is observed at elevated temperature testing as it is at ambient and cryogenic temperature testing. These data, together with our previous work,[6,7] therefore also highlight the risks in employing the use of Charpy testing to determine mechanistic failure information of high-strength ductile steel, since Charpy impact testing is only able to record the energy absorbed by the test specimen, which is not only absorbed in localized void growth and coalescence, but also in global plasticity.

Although the data herein indicate a clear and significant reduction in fracture toughness between equivalently graded HIP and forged stainless steel, it is important to note that these HIP materials remain to be very tough materials, exhibiting J initiation toughness values between 500 and 1000 kJ m^{-2}. This is further highlighted by the calculated theoretical J_{max} values, which represent the theoretical maximum valid J that can be measured based on the material yield strength and the test specimen thickness. Figures 2, 3, and 4 show that as a result of the large plastic zone ahead of the crack tip with respect to the specimen size, all of the measured J values are technically invalid for the ambient and elevated tests, even when using 25-mm-thick C(T) test specimens.

Because of the nature of the testing, whereby specimens are loaded to incremental displacements on a judgemental basis in order to yield varying levels of crack growth, several of the J-R data points are outside of the exclusion limits as defined by ASTM E1820, and should therefore be treated as invalid. This is most noticeable for the ambient temperature and cryogenic testing of F304L, which gleaned very little ductile crack growth up to the maximum allowable displacement of the clip gauge employed, and J-R data points were located in close proximity to the theoretical blunting line. This highlights the challenges associated with acquiring valid J-R data for extremely tough steels at cryogenic temperatures, whereby data points must fall within an area confined by exclusion lines and a theoretical J_{max}.

Figure 5 shows fracture surfaces for representative test specimens at each test condition. At all testing temperatures, it is clear that the material fails in a purely ductile manner, indicating no signs of brittle cleavage failure. The ductile dimples in the forged specimens can be seen to exhibit larger diameters than the equivalent HIP specimens, and this was an observation made on Charpy fracture surfaces in the previous studies.[6] This difference in ductile dimple diameters was previously attributed[7,8] to the distance over which voids are required to grow before they coalesce with neighboring voids.

The data show that HIP stainless steel can exhibit clear and significant reductions in J-R fracture toughness, when compared to forged stainless steel of equivalent grade. This reduction in fracture toughness is believed to be governed by the same mechanisms that affect Charpy impact toughness,[6] since the fracture surface characteristics are consistent with previous studies in that the Forged 304L exhibits larger diameter ductile dimpling in comparison to the HIP materials. No evidence of brittle failure is observed, with pure ductile fracture being the mode of failure at all temperatures. The larger diameter ductile dimpling observed in the Forged specimens in comparison to the HIP specimens is thought to be the result of fewer initiation and sites for void growth,[6–8] resulting in smaller distances over which voids are required to grow before they coalesce with neighboring voids. This is believed to result in an 'unzipping'-like effect during the ductile fracture of the HIP specimens, since initiated voids are significantly closer together, resulting in crack growth where coalescence can occur at lower levels of plastic strain.

These data are in agreement with the previous Charpy studies, highlighted in Table II, which represents the reduction in toughness as a ratio of the HIP toughness
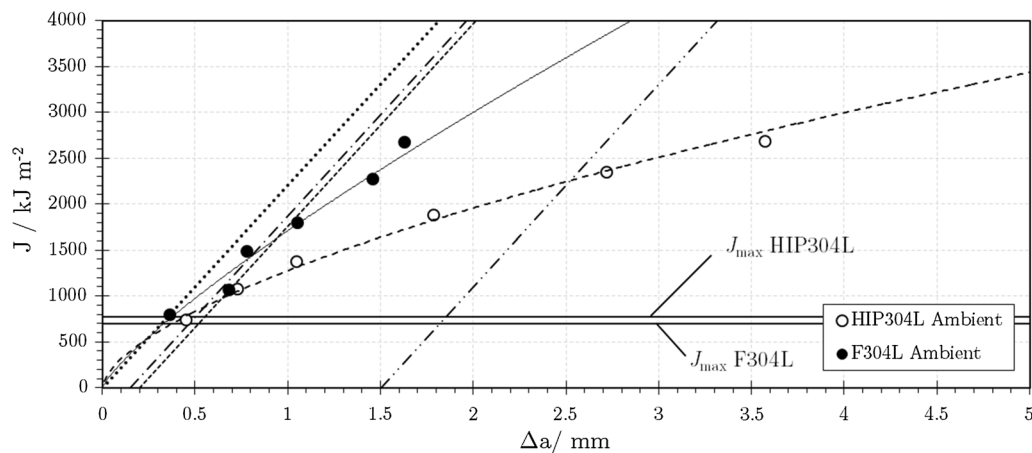
Fig. 2—J-Resistance curves for HIP and Forged 304L at ambient temperature—this figure is a reprint from Ref. [8] for completeness.
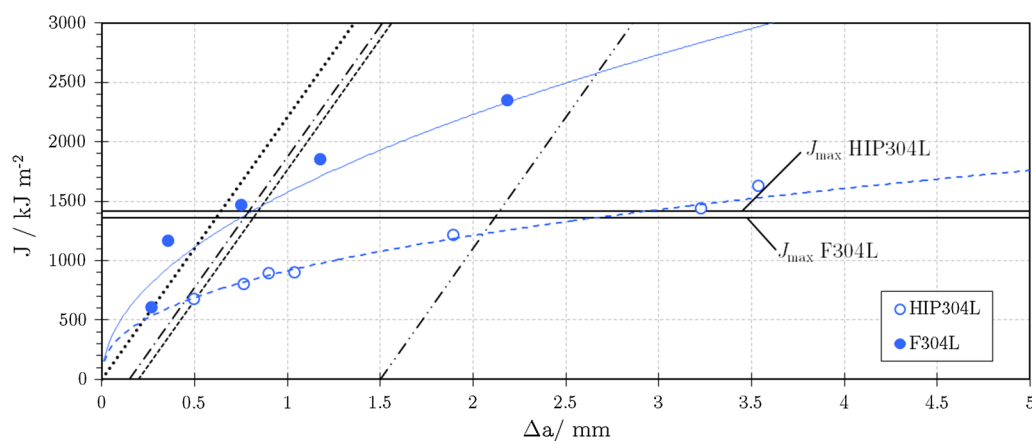

Fig. 3—J-Resistance curves for HIP and Forged 304L at − 140 °C.

Table II. Measured $J_{0.2BL}$ Values

	Test Temperature/°C		
	− 140	20	300
HIP304L $J_{0.2\ BL}$/kJ m^{-2}	700	950	525
F304L $J_{0.2\ BL}$	1450	1550	800
HIP $J_{0.2\ BL}$: Forged $J_{0.2\ BL}$	0.48	0.61	0.66
HIP C_v: Forged C_v/ pct[5]	0.60	0.70	1.03

to the Forged toughness for both the previously reported Charpy tests[5] and the present fracture toughness tests at each respective temperature. The reduction in toughness at ambient temperature was measured to be approximately 39 pct for the fracture toughness test, compared to 30 pct for the Charpy tests. The reduction in toughness at cryogenic temperature was measured to be approximately 52 pct for the fracture toughness test, compared to 40 pct for the Charpy tests. In both cases, the reduction in toughness was greater in the fracture toughness tests than the Charpy tests, by approximately 10 pct, and this is thought to be related to the level of constraint ahead of the crack in the test specimens. In

the fracture toughness tests, the plastic zone size is much more confined to a localized region ahead of the crack tip, and as a result the energy required for fracture is localized in the ductile tearing mechanism. In contrast, excessive plastic deformation was found to occur in the ligaments of the Charpy test pieces,[5] and this form of energy absorption is thought to detract from the localized fracture process. This observation is more apparent in the 300 °C tests; whereas the reduction in fracture toughness of HIP 304L compared with Forged 304L was observed to be in line with the fracture toughness tests performed at ambient and cryogenic temperature, exhibiting a reduction in toughness of
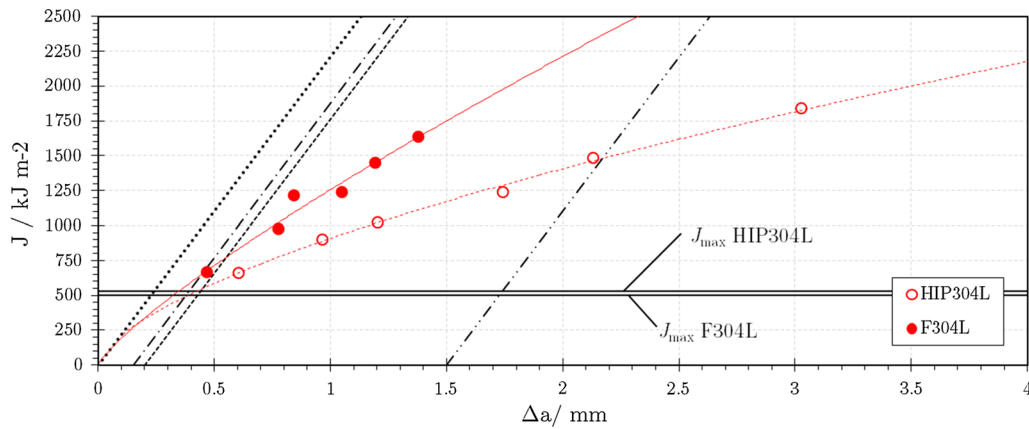
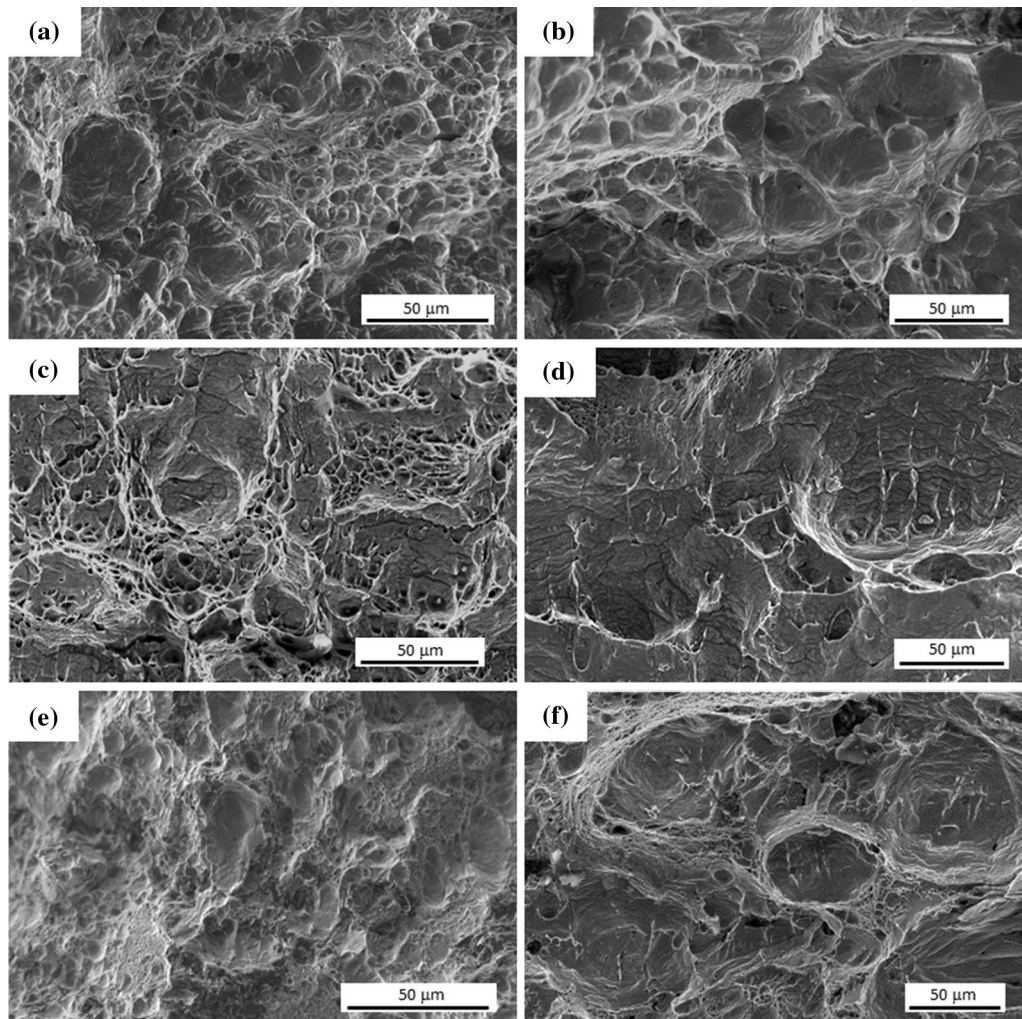Fig. 4—J-Resistance curves for HIP and Forged 304L at 300 °C.

Fig. 5—Scanning electron micrographs of failed compact tension test specimen fracture surfaces for HIP 304L (*a*, *c*, *e*) and forged 304L (*b*, *d*, *f*), tested at 300 °C (a, b), 20 °C (c, d), and − 140 °C (e, f).

approximately 34 pct, no reduction in toughness was observed in the Charpy test results at 300 °C. Incidentally, Charpy test specimens often did not fracture completely when tested at elevated temperature, and exhibited significant plastic deformation of the ligaments. This phenomenon could also help to explain why the reduction in fracture toughness of HIP304L appears to increase at lower test temperatures: yield strength increases as temperature decreases, and thus the size of the plastic zone becomes more confined to the

crack tip, resulting in a more localized energy absorption, which could be considered to be a more accurate description of the localized fracture behavior.

Significantly, the results reveal that a comparable reduction in toughness is present at elevated temperature as that of ambient and cryogenic temperature testing; a statement which could not be made when considering Charpy data alone, due to high levels of ligament plastic deformation associated with the Charpy test.

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