Fatigue and fracture of three austenitic stainless steels at cryogenic temperatures

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Abstract. For the past couple decades, 316LN stainless steel has remained the “go-to” alloy for structural components intended for cryogenic temperature service, partially because of its favorable mechanical properties, but also because of the data available in the literature for T = 4 K. In recent years, some interest has arisen to investigate and develop stronger and tougher alloys for cryogenic structural components, particularly for magnet systems like ITER. This study presents new 4 K fatigue crack growth rate (FCGR) and fracture toughness data for Nitronic® 50 and JK2LB stainless steels, compiles existing data for these alloys, and compares them with 316LN data found in literature. This study intends to further expand the existing cryogenic data set for these alloys, clarify key differences between them to better facilitate mechanical design, and potentially bolster further alloy development.

1. Introduction

316LN stainless steel is currently one of the most commonly selected structural materials used in superconducting magnet systems, thanks in part to its high strength, good fracture toughness and low magnetic permeability after fracture at cryogenic temperatures – qualities which are not present in many steels. Additionally, its excellent 4 K database helps make it a comfortable choice during the design process, as there exists a substantial amount of tensile, fracture toughness, and S-n fatigue data in the literature for the alloy in various conditions at cryogenic temperatures. However, some systems could benefit from different alloys with slightly better, or more specifically matched properties for a given design. ITER is one such project, the scale of which has also facilitated R&D of new alloys for its purposes. The tie plates that maintain pre-compression on the Central Solenoid (CS) modules require higher room temperature yield strength than 316LN can offer, so Nitronic 50 (Fe-XM-19, Fe-21Cr-12Ni-5Mn) was investigated and accepted for the task [1]. Additionally, a brand new alloy, JK2LB, was developed specifically for use as CS conduit material, primarily to minimize the thermal contraction mismatch between the conduit and Nb₃Sn superconducting cables it encases [2]. These alloys have been tested extensively and reported on by several labs in recent years, but are rarely compared with one another in a common study. This investigation aims to report typical ranges of fatigue and fracture behavior of each alloy in various conditions to highlight key differences between them, and potentially aid with material down-selection for future projects at the very least. It is a compilation of some previously-published data as well as brand new data.

2. Materials and methods

Three alloys are investigated in a wide array of conditions: 316LN, Nitronic 50 (N50), and JK2LB. Since 316LN has been studied and improved upon for several decades, relevant data must be selected
for a fruitful comparison with other modern alloys. Arguably, one of the last substantial developments on 316LN specifically for conduit alloys was in Reed’s study on grain boundary sensitization after Nb-Sn heat treatment due to Carbon content [3]. It suggested that Carbon content be kept at or below 0.01% for a 700 °C/100 hr heat treatment to retain adequate toughness. Though the decrease in toughness was not as drastic in specimens whose heat treatment final step was 625 °C/75 hr, which is closer to most Nb-Sn heat treatments today, only 316LN with Carbon content near 0.02% or less was chosen for the present study. Table I tabulates chemical composition of most materials reported here. An ideal academic comparison would normalize as many variables as possible (e.g. all specimens annealed, same orientation, deliberate grain size selection, etc.), but the data generated recently has been application-driven, in which clients need to confirm each materials’ properties in the exact condition they are to be employed in the magnet structure. Thus, several conditions are reported here.

### Table 1 - Chemical composition of available materials.

| Mat’l Comp # | Material Description | Composition (wt%), Fe balance | Pub Ref |
|--------------|----------------------|-------------------------------|---------|
| 1            | JK2LB Conduit (Hamada 2008) | 0.013 21.80 0.002 9.25 12.80 0.98 0.12 0.0036 | 12      |
| 2            | JK2LB Conduit (Walsh 2009) | 0.025 21.42 0.002 12.72 0.08 0.12 0.0013 | 13      |
| 3            | JK2LB Conduit (Walsh 2015) | 0.012 21.55 0.002 0.15 0.015 0.015 0.020 | 14      |
| 4            | N50 F50 (Read 1978) | 0.041 4.96 0.020 0.015 12.83 21.15 0.17 0.15 0.31 | 15      |
| 5            | N50 Forgings, Slab & Head, Vendor A (2013) | 0.035 5.18 0.024 0.004 12.67 21.16 0.12 0.14 0.28 | 16      |
| 6            | N50 Forgings, Slab & Head, Vendor B (2014) | 0.026 5.29 0.022 0.002 0.33 0.13 0.04 0.34 | 17      |
| 7            | N50 Plate (2017) | 0.040 4.92 0.021 <0.001 0.034 0.11 0.20 0.07 0.18 0.32 | new     |
| 8            | N50 Sheet (2017) | 0.017 4.47 0.018 0.001 0.004 1.29 0.20 0.27 0.19 0.34 | new     |
| 9            | N50 Forging Vendor C, Batch 1 (2017) | 0.020 5.40 0.016 0.001 0.23 0.12 0.20 0.21 0.10 0.30 | new     |
| 10           | N50 Forging Vendor C, Batch 2 (2017) | 0.016 4.81 0.020 0.001 0.045 0.03 0.12 0.20 0.26 0.18 | 0.0014 0.004 new |
| 11           | N50 Forging Vendor D (2017) | 0.024 4.80 0.020 0.001 0.34 0.12 0.20 0.21 0.15 0.28 | 0.0016 0.004 new |
| 12           | 316LN, Alloy B (Simon 1988) | 0.006-0.010 1.40 0.002 0.00002-0.0001 0.40 0.11 0.20 0.25 0.16 | 19      |
| 13           | 316LN, Alloy E (Simon 1988) | 0.016 1.58 0.021 0.021 0.48 0.13 0.20 0.25 0.17 | 19      |
| 14           | 316LN, Alloy H (Simon 1988) | 0.016 1.10 0.020 0.015 0.54 0.10 0.20 0.25 0.10 0.14-0.17 | 19      |
| 15           | 316LN Mod (Walsh 1996) | 0.010 1.53 0.011 0.005 0.15 0.13 0.25 0.23 0.10 | 21      |
| 16           | 316LN 80mm plate (Nyilas 2002) | 0.020 1.58 0.027 0.0008 0.49 0.11 0.08 0.10 0.16 | 20      |
| 17           | 316LN Valinix jacket (Nyilas 2002) | 0.012 1.64 0.025 0.05 0.12 0.17 0.20 0.24 0.18 | 20      |
| 18           | 316LN Plate (Ja 2006) | 0.022 1.14 0.020 0.001 0.69 0.10 0.10 0.17 0.21 | 21      |
| 19           | 316LN Conduit (Walsh 2006) | 0.012 1.58 0.020 0.001 0.36 0.13 0.27 0.13 0.15 | 21      |

All of the 4 K tensile and fracture toughness tests performed at NHMFL are conducted on servo-hydraulic test machines that feature a load reaction frame and cryostat that facilitate testing in liquid helium. Typically, a 100 kN capacity load cell is used for standard tensile specimens as well as all sizes of load-line compact tension (CT) specimens for FCGR and fracture toughness. Shepik-style clip-on extensometers are used to measure strain in tensile tests, and a clip-on crack opening displacement (COD) gage is used to monitor load-line displacement of CTs. Tensile tests are performed according to ASTM E1450, and FCGR and fracture toughness tests are performed according to procedures outlined in E647 and E1820 respectively. Fracture toughness analysis is performed with commercial software via J-integral method to obtain $K_I$ per E1820.

### 3. Test data and discussion

#### 3.1. Fracture toughness and yield strength

Table II shows all materials compared here, with their tested condition, grain size, and selected mechanical properties. The composition number column corresponds to the one shown in Table I for these materials, where available. The new data presented here is denoted with “new” in the Reference column. All mechanical property values shown are averages of several individual test specimens.
Table 2 – 4 K Mechanical properties of all materials included in this study.

| Mat'l Comp # | Material Description | Material Condition | Grain Size | Yield Strength | Tensile Strength | Elongation | Area Reduction | Kc(J) | FCGB Porta Parameters
|--------------|---------------------|-------------------|------------|---------------|-----------------|------------|---------------|-------|----------------------|
| 1            | JK2LB Conduit TL (2000) | AA*†              |            | 787           | 1322            | 58          | 55            | 254   | 12                   |
| 2            | JK2LB Conduit TL (2000) | AC/1 + A06/J     |            | 1006          | 1370            | 47          | 52            | 224   | 12                   |
| 3            | JK2LB Conduit TL (2000) | AC                |            | 1015          | 1395            | 40          | 45            | 154   | 13                   |
| 4            | JK2LB Conduit TL (2000) | AC/1 + A06/J     |            | 1063          | 1397            | 38          | 38            | 167   | 13                   |
| 5            | JK2LB Conduit TL (2000) | AC                |            | 1035          | 1458            | 41          | 32            | 357   | 14                   |
| 6            | JK2LB Conduit TL (2000) | AC + CW + AG     |            | 1029          | 1424            | 42          | 30            | 407   | 15                   |
| 7            | JK2LB Manifold Forging TL (2017) | AA*†              |            | 995           | 1358            | 44          | 28            | 276   | 15                   |

A common graphical presentation for 4 K mechanical performance of these alloys is a $K_c(J)$ vs yield strength plot. For comparison of materials on the plot it is good practice that the applied load direction be the same for both the yield strength and toughness values plotted. Test standards ASTM E399 and E8 provide the standard nomenclature for referencing the test specimen orientation. Figure 1 portrays all data chosen here, with appropriate respective loading directions. Several data now appear outside the NIST trend line for 300 series austenitic stainless steels [4] on the plot, but one should approach this comparison with caution. The data used to establish the trend lines are for annealed alloys that are J-tested in TL orientation to generate conservative design data. We suspect that specimens are likely to deviate from this trend when tested in either LT or LS directions. However, in the case of ITER’s mechanical qualification testing, these values are needed in LT and LS orientation, as it is a direct measurement of the real-life loading direction, using the exact manufacturing processes that will be employed on the full scale magnet system. This makes it useful data primarily for ITER and any other entity interested in properties in the same orientation, but trickier to compare with existing 316LN values. Additionally, several fracture toughness values on the plot are obtained from thin CT specimens that violate ASTM thickness requirements. But, since a significant amount of characterization has been done for thin-wall conduit [5-8], they are data points that must be carefully integrated with the rest. Previous studies have been done to quantify the effect of CT thickness on measured $K_c(J)$ at 4 K [8-10]. The largest variance observed was a 10% increase on a 12.7 mm thick specimen [9], but both other studies concluded the difference to be inappreciable within the error of

* AA: As-Received
† AC: As-Compacted; ITER CS conduit undergoes transverse compaction before winding
‡ AG: Aged; subjected to simulated Nb3Sn injection heat treatment
§ C/3h, WQ: Cold-worked; welded using weld on applied
** Specimens failed on gage marks
the measurement [8, 10]. However, when testing the N50 plate for this report, we machined standard-thickness (12.7 mm) 0.5-CTs as well as 1.6 mm thick 0.5-CTs to assess the effect of specimen thickness study on this alloy; the average results were 147 MPa$\sqrt{m}$ for the thin specimens, and 109 MPa$\sqrt{m}$ for full thickness - a 35% increase. It is unclear whether the degree of this thickness dependence is different for each alloy, but it highlights the care that must be taken when comparing non-standard thin-specimen results with those from full-thickness specimens.

![Fracture Toughness vs Yield Strength, T = 4 K](image)

*Figure 1 - Fracture toughness vs yield strength, T = 4 K.*

The relationship between $K_{IC}$ and yield strength in a given material is known to be inverse [11]. When comparing similar alloys on the plot, there are no singular factors present that stand out to explain the mechanisms that differentiate their properties relative to one another. Rather, they are the result of a combination of several variables - grain size, orientation, composition, manufacturing process, etc. Unfortunately, investigation into the effects of composition, microstructure, and other metallurgical variabilities was not included in the contracts that funded this work, so speculation on alloy optimization or improvement is outside the direct scope of this paper. However, reporting many of these variables in one common comparison alongside mechanical properties – as is the present intent – could make that investigation easier for the near future.

The three alloys do seem to populate broad common areas of this plot with one another. Most 316LN portrayed here does nearly follow the NIST trend, albeit with slightly higher strength and toughness than the upper band. JK2LB has consistently exhibited remarkably high toughness, in some cases exceeding maximum valid J-capacity of 0.5-CT specimens, thus requiring larger specimens. However, its strength has not yet achieved nearly that of some of the N50 investigated to-date. N50
generally has exceptionally high strength, often with lower toughness than that of JK2LB. Overall, these alloys combined appear to create a trend that surpasses the existing NIST trend, but additional testing could lend more confidence to this potential claim; for example, testing 2013-2015 N50 forgings in TL direction would help answer the question of orientation dependence, as would testing JK2LB from the same conduit and/or forging in two different directions.

3.2. Fatigue crack growth rate
Figure 2 shows FCGR curve fit data for all available materials here, separated by material – JK2LB, Nitronic 50, and 316LN at top left, top right, and bottom left, respectively. The graph at bottom right compares the range of crack growth data experienced in all three alloys. N50 in particular contains some dramatic outliers, though the bulk of data gathered here is grouped together and not dissimilar to 316LN and JK2LB. Those extreme values are from custom-specified thick forgings, received from different suppliers. However, other specimens from the same forgings behave similar to commercially-available material. There does not appear to be a characteristic crack growth rate dependence on crack plane orientation in most of these materials; the difference in da/dN curves between crack plane directions has often been observed to be on the same scale as variability between individual specimens of the same orientation. Thus, the high and low N50 curves are either a function of an exceptionally high degree of anisotropy or inhomogeneity within only those special forgings. None of these three alloys appears to have inherently higher or lower crack growth rate than the others.

3.3 Error Propagation Assessment
4 K mechanical properties testing is inherently challenging, and the test results exhibit data scatter due to both experimental procedure as well as material property variability. An experimental procedure error estimate is performed by evaluating the resolution and accuracy of the physical measurands that are used in calculating tensile and fracture toughness results. Using methods outlined in [22] and [23], which are based on standard error propagation calculation techniques, experimental procedure error is estimated to produce roughly 1-2% error in tensile test results, and can approach up to 4-6% for fracture toughness values presented here. These vary primarily with specimen size and equipment used to measure load, strain, load-line displacement, and specimen geometry. The typical data scatter observed in 4 K tensile and fracture toughness tests for a given material are quantified [13] with the standard deviations in a range closer to 10-20%, thus the additional spread is presumed to be attributed to variability within the material itself.
4. Conclusions

4 K mechanical test data for three modern austenitic stainless steels has been compiled into one study for comparison. On the surface, they seem to represent an evolution of materials available for cryogenic structures, but more work is required for some concrete conclusions to be made. This report can serve as a launch pad for further alloy development - by organizing a considerable amount of research data into one source, potentially highlighting the next logical steps to take - but more immediately, it may serve as a useful tool during initial mechanical design decision-making.

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