PAPER • **OPEN ACCESS**

The low temperature mechanical properties of a Nitronic 40 forging

To cite this article: R P Walsh *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **756** 012001

IOP Conf. Series: Materials Science and Engineering **756** (2020) 012001 doi:10.1088/1757-899X/756/1/012001

The low temperature mechanical properties of a Nitronic 40 forging

R P Walsh, K J Radcliff, J Lu and K Han
National High Magnetic Field Laboratory, Florida State University
800 E. Paul Dirac Dr.

Abstract. Nitronic 40 forged shells are typically used for structural reinforcement in high field pulse magnet design and applications. To better understand the mechanical performance of this versatile high strength austenitic steel a series of mechanical tests were conducted. Tensile were performed at 295 K, 77 K and 4 K, and cryogenic fracture mechanics tests were performed at 77 K and 4 K. The effect of temperature on strength, ductility, toughness and fatigue crack growth rate are evaluated. Microstructure and composition effects are also presented and discussed.

1. Introduction

Pulse magnet reinforcement shells are required to have good mechanical properties over the temperature range from 77 K to 350 K. Pulse magnets operate at temperatures ranging from 77 K to 350K with peak stress at the low end of the temperature range. The austenitic steel alloy Nitronic 40 is a material of choice for pulse magnet reinforcement shells and experience has shown that process and quality control of this complex alloy is necessary [1]. The mechanical properties of a Nitronic 40 forged reinforcement shell have been investigated at three test temperatures (295, 77, and 4 K) with tensile, fracture toughness, and fatigue crack growth rate (FCGR) tests. The fracture toughness and FCGR tests are focused on the 77 K test temperature while the 4 K tensile and fracture toughness tests were conducted to obtain additional low temperature data on this versatile alloy.

The test material was obtained from a LANL pulse magnet shell that was available after retirement from some years of pulse magnet service. We tested the forging in two different conditions, the as-received (AR) condition and a heat-treated condition (HT). The HT condition was achieved with a relatively short-time, low-temperature heat treatment (400C for 20 min) that simulates the proposed insulation curing process to be used on future pulse magnets. Fracture toughness measurements were made at 77 K to obtain design data to make sure the heat treatment did not compromise the materials properties. The magnetic permeability, before and after low temperature deformation, were measured and are reported here too.

2. Material Information

The chemistry of the forging was determined commercially at two locations within the forging and is given in Table 1. Nitronic 40 is a commercially available high-manganese nitrogen-strengthened austenitic steel that is often referred to as 21-6-9, due to the nominal percentages of Cr/Ni/Mn. It has extremely low magnetic permeability and good low temperature properties with higher yield and tensile strength at 295 K than typical of the 300 series austenitic steels.

Table 1: The Nitronic 40 forging chemistry at 2 locations within the forging is shown below.

Element (wt %)		C	Mn	P	S	Si	Cr	Ni	Mo	Cu	N
Required Specifications	Min	---	8.00	---	---	---	19.0	5.50	---	---	0.15
	Max	0.04	10.00	0.060	0.030	1.00	21.5	7.50	0.75	---	0.40
110 mm from end		0.05	9.39	0.024	0.00	0.47	19.67	7.28	0.25	0.20	0.34
560 mm from end		0.05	9.44	0.023	0.003	0.49	19.67	7.29	0.25	0.20	0.34

The heat treated (HT) material condition was achieved by heat treating (4) tensile and (3) CTs. The specimens were placed in the preheated furnace retort, with an Ar atmosphere and subjected to the thermal profile in Figure 1.

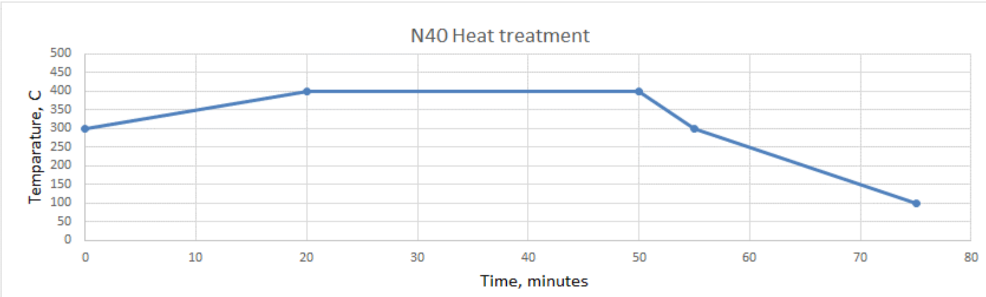

Figure 1: The nominal heat treatment temperature profile for HT condition specimens.

3. Procedures

The schematic in Figure 2 shows the ASTM compliant tensile and fracture toughness test specimen location and orientation as they were removed from the forged tube. All the tests were conducted on either a 100 kN or 250 kN capacity MTS machines that are equipped with a cryostat to enable testing at 77 K and 4 K with the test specimen and fixture immersed in liquid nitrogen or helium respectively.

Tensile tests are conducted, according to procedures in ASTM E8 and ASTM E1450, in displacement control at a rate = 0.5 mm/min (initial elastic strain rate = 2.2e-4 s^{-1}). The cylindrical tensile specimens (6.35 mm dia. X 38 mm long gage section) were oriented with their axes in the circumferential direction (C). Strain was measured using a set of (3)-25 mm gage length clip-on extensometers. Elongation is determined from scribe marks made 25 mm apart on the specimen gage

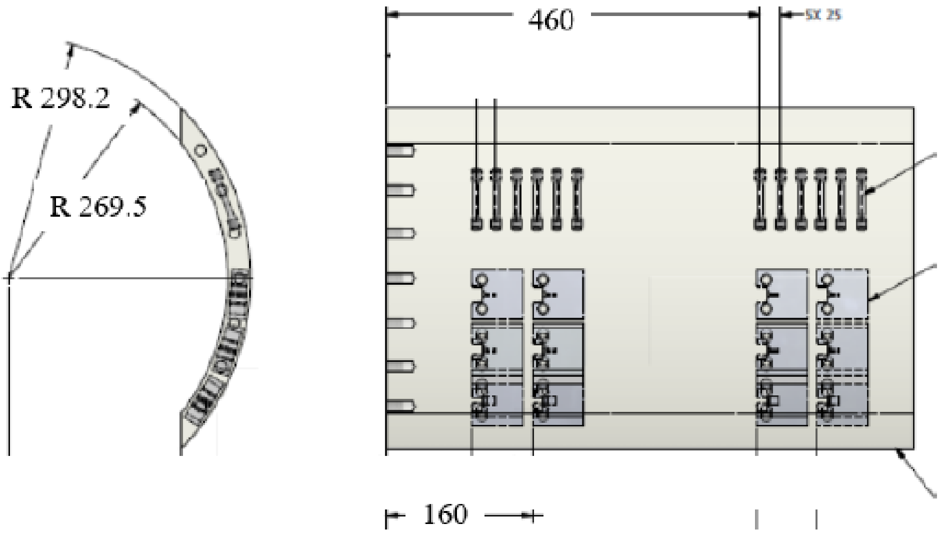

Figure 2: Nitronic 40 Forged tube specimen layout drawing.

IOP Conf. Series: Materials Science and Engineering **756** (2020) 012001 doi:10.1088/1757-899X/756/1/012001

section prior to testing. The 77 K FCGR and the 77 K and 4 K fracture toughness were performed according to the guidelines in ASTM E647 and ASTM E1820 respectively. The 1.0 CT specimens are oriented CL (circumferential load direction, longitudinal crack direction). The CT specimens were Electro-Discharge-Machined (EDM) with a short notch (15.5 mm) so they can be used for both the FCGR tests and the J_{IC} tests.

The specimens were initially pre-cracked ~2 mm at 77 K before FCGR testing at 77 K or 4 K. The FCGR test is conducted from the crack length (a) = 17.5 mm until (a) reaches ~ 30 mm for an (a/W) ratio of 0.6 that is desired for the J_{IC} test. The FCGR test was performed in constant load control (an increasing Δ K mode) and during the test the Δ K ranges from ~ 30 MPa√m to 70 MPa√m until the 30 mm crack length is reached. The fracture toughness test is performed next.

For ductile materials, the elastic-plastic fracture mechanics test method ASTM E1820 allows for the determination of $K_{IC}(J)$ in a J-integral (J_{IC}) test where K_{IC} is converted from J_{IC} using the equation:

$$K_{IC}(J) = (J_{IC} * E^1)^{1/2}$$

Where; $E^1 = E/(1-\nu^2)$
E = Young's Modulus
ν = Poisson's Ratio

The values determined in 77 K and 4 K fracture toughness tests typically do not satisfy all the ASTM E1820 requirements and are considered estimates of the 4 K toughness.

Magnetic permeability measurements were made on test specimens using a Low-Mu Permeability Indicator according to ASTM A342 procedures. The indicator can sense the relative permeability (μ) of low μ materials (μ of 6.0 or less).

4. Results and Discussion

The tensile test results are shown in Table 2 and Figure 4 plot of strength vs test temperature. The 295 K tensile properties of both the AR and the HT material are similar, suggesting no effect from the HT process. The forging has a high yield strength and tensile strength (432 MPa and 791 MPa) at 295 K that increases almost linearly with decreasing temperature to approximately 1400 MPa and 1845 MPa respectively at 4 K. The tensile results for the forging compare favourably to previously published low

Table 2: Nitronic 40 tensile test results for circumferential direction.

Temp	Condition	Spec. No	Modulus of Elasticity GPa	Yield Strength MPa	Tensile Strength MPa	Elong. in 25 mm %	Reduction of Area %	Relative Permeability μ
295	AR	T1	199	430	784	63.0	76.2	<1.01
		T2	205	434	794	60.5	76.1	<1.01
		T3	208	433	795	64.1	75.0	<1.01
		AVG	204	432	791	63	76	
	HT	T3	204	440	790	69.0	79.2	<1.01
		T4	202	441	787	60.5	76.3	<1.01
		AVG	203	441	789	65	78	
77	AR	T4	NA	1125	1577	35.1	29.5	3.0
		T5	210	1133	1593	45.9	39.0	5.0
		T6	205	1129	1585	35.6	29.4	2
		AVG	208	1129	1585	39	33	
	HT	T5	204	1107	1490	35.9	28.9	2.0
		T6	204	1118	1434	42.5	33.9	3.0
		AVG	204	1113	1462	39	31	
4	AR	T1	209	1414	1855	19.1	24.3	2.0
		T2	205	1393	1836	15.9	17.1	1.2
		AVG	207	1404	1845.5	18	21	

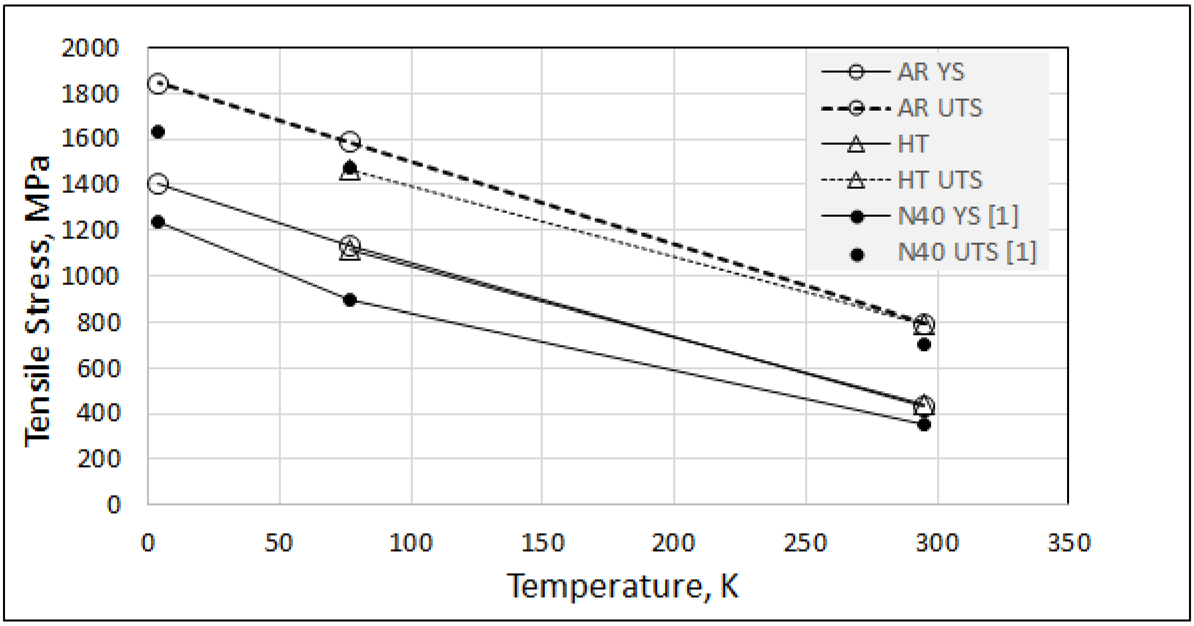

Figure 3: Plot shows an almost linear relationship between strength and temperature.

temperature data [2-4] for Nitronic 40 which is included on the graph in Figure 3 and this forging is somewhat stronger at all three temperatures probably since the reference data is for annealed plate. Both the AR and HT material exhibit good ductility at 77 K with almost 40% elongation and about 30 % reduction in area.

The fracture toughness and FCGR results are shown in Table 3. The 77 K fracture toughness of the forging is excellent with a slight difference in the average toughness (3 tests each) of the HT and AR

Table 3: Nitronic 40 fracture toughness and FCGR test results.

Temperature		Specimen	K_{IC}(J)	Paris Law Constants (see note)		Delta K Range
K		Number	MPa√m	C	m	MPa√m
77	AR	110-CT-1	357	1.16E-08	2.63	32 to 66
		110-CT-2	353	5.85E-09	2.82	33 to 66
		110-CT-3	n/a	7.39E-09	2.78	34 to 67
		460-CT-1	348	n/a	n/a	n/a
		Average	352	7.49E-09	2.76	34 to 67
	HT	110-CT-4	318	7.49E-09	2.76	35 to 70
		110-CT-5	344	1.12E-09	3.27	28 to 55
		110-CT-6	366	2.07E-09	3.16	28 to 55
		Average	343	9.42E-10	3.36	28 to 70
4	AR	460-CT-4	144	3.53E-10	3.88	28 to 55
		460-CT-5	n/a	1.19E-08	3.00	27 to 40
		460-CT-6	164	7.99E-10	3.74	31 to 68
		460-CT-2	118	5.33E-09	3.23	27 to 57
		Average	142	1.54E-09	3.54	27 to 68

1. K_{IC}(J) values are estimates since validity requirements of ASTM E813 are not met
2. Relevant units for Paris Law constants: ΔK = MPa√m, da/dn = mm/cycle
3. Specimen Orientation is (CL)

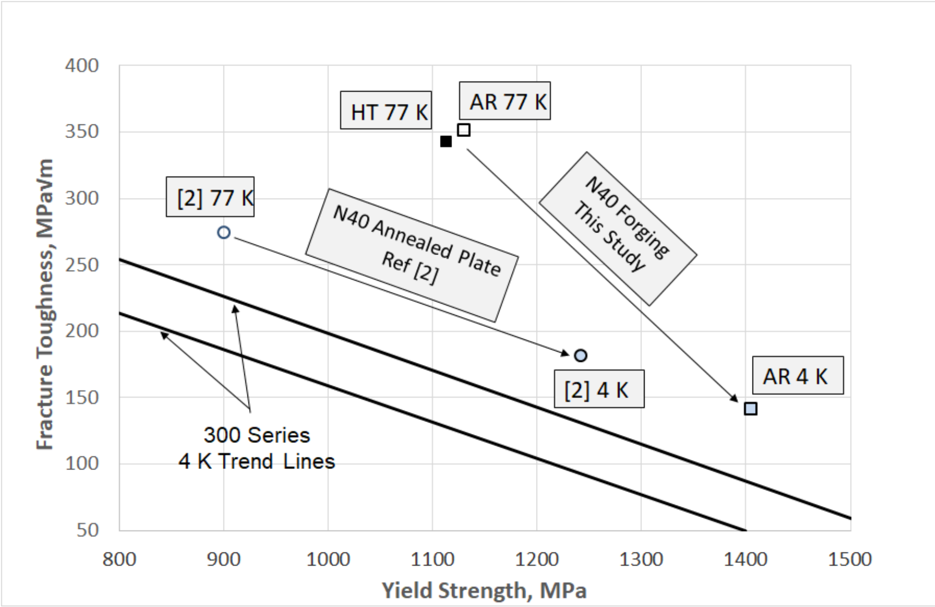

Figure 4: Fracture toughness vs yield strength comparison with the published data [3]. The NIST 4 K trend lines for 300 series stainless steel are also included for reference.

conditions, the $K_{IC}(J)$ values are 355 MPa√m and 343 MPa√m respectively. About a 60% decrease in toughness was observed for the AR material from 77 K to 4 K.

The 4 K toughness of the AR is still good with an average value = 142 MPa√m for 3 tests. Low temperature fracture toughness in austenitic steels is usually inversely dependent to the tensile yield strength. The Figure 4 graph of Yield Strength vs Fracture Toughness shows the 77 K and 4 K results for the Nitronic forging and compares them with published data [3]. The NIST trend lines show the scatter band typical for austenitic steels at 4 K. This Nitronic 40 forging has better performance than the annealed plate [3] at 77 K while the 4 K strength toughness relationship is similar.

FCGR tests have been conducted to determine the Stage II (Paris Regime) crack growth behaviour. In the Paris regime (FCGR from about 10^{-5} mm/ cycle to 10^{-3} mm/cycle) the crack growth rate obeys the simple power law;

$$da/dn = C^*\Delta K^m$$

where;
da/dn = crack growth increment/cycle
ΔK = stress intensity factor range (Kmax-Kmin)
C, m = constants related to material, test temperature, etc.

The plot of da/dn vs ΔK in Figure 5a is typical FCGR data from the test of one specimen (Specimen AR-CT2). A power law curve fit is applied to the data and the Paris law parameters (C and m) for this test are shown on the graph and in Table 3. The average Paris law parameters shown on Table 3 are obtained by combining the FCGR data specific to one material condition (AR or HT) and one test temperature (77 K or 4 K) from all the specimens tested for that condition and curve fitting the entire data set. The combined data set for the (3) 77 K tests of AR material are plotted in Figure 5b providing a visual representation of the FCGR data scatter.

The Paris Law parameters are used to present the average FCGR for the test and material conditions in Figure 6 and 7. The 77 K FCGR results for the AR and HT are very similar, and for example for a

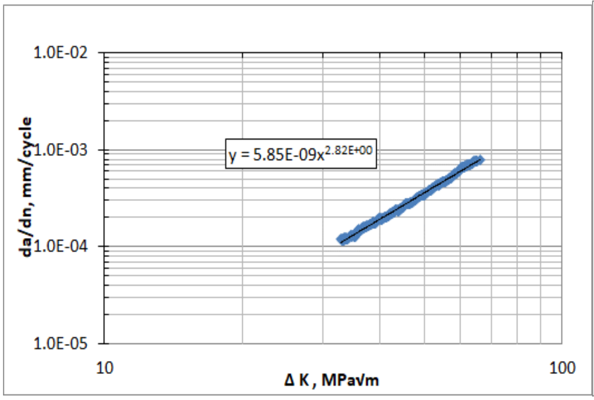

Figure 5a: Power law curve fit of FCGR test data for one specimen.

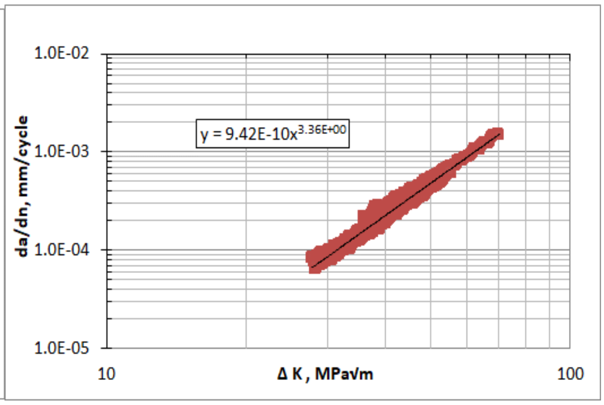

Figure 5b: Power law curve fit FCGR test data for three tests to get the average parameters.

ΔK of 30 MPa√m the crack growth rates are 8.84x10^{-5} and 8.65x10^{-5} mm/cycle respectively. This rate is comparable to the previously published 102 K data that is about 8.76 x10^{-5} mm/cycle at 30 MPa√m. Figure 6 shows the 4 K FCGR is quite a bit higher than the 77 K rate, approximately 2 times higher at ΔK of 30 MPa√m and about 4 times higher at ΔK of 70 MPa√m. Although we could not find any published FCGR data for Nitronic 40 at 4 K, we compare the 4 K FCGR of Nitronic 40 to Nitronic 50 in Figure 7. This Nitronic 40 forging has considerably higher crack growth rates than published values for Nitronic 50.

More research is necessary to understand why Nitronic 40 FCGR is higher at 4 K than 77 K and why the FCGR is higher than Nitronic 50 at 4 K. Nitronic 50 is susceptible to low temperature strain induced martensitic transformation so we made magnetic measurements on the fractured Nitronic 40 tensile specimens.

The Nitronic 40 forging is non-magnetic due to its composition and metallurgical condition (austenitic phase with FCC crystal structure). Magnetic permeability (μ) measurements are made on the fractured tensile specimens as a measure of austenitic stability after low temperature deformation. The measurements are localized around the fracture region and the results are shown in the last column in

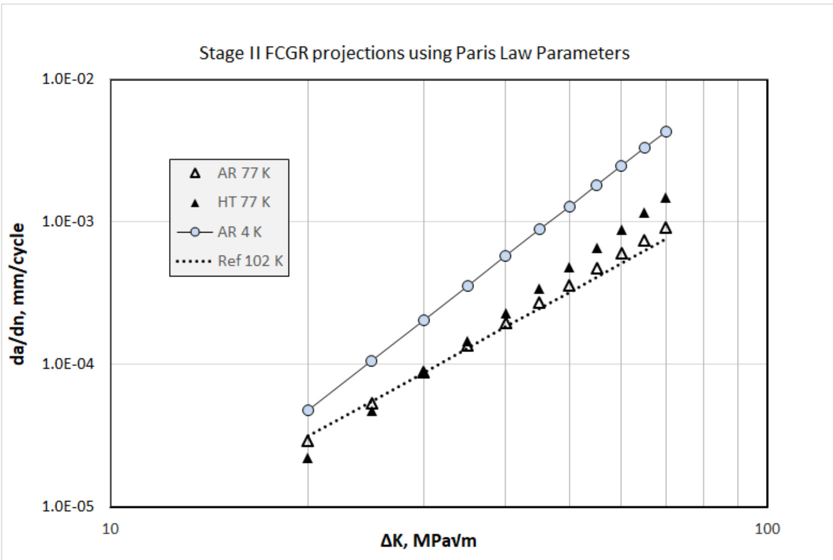

Figure 6: The 77 K projections compare well with the 102 K reference [2] data. The 4 K FCGR trendline shows a much higher crack growth rate than at 77 K.

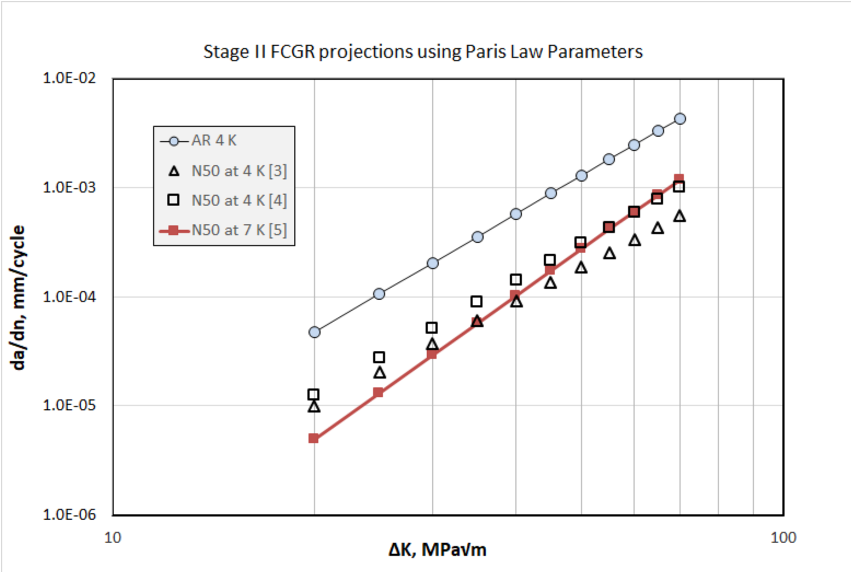

Figure 7: The 4 K Nitronic 40 data show a considerably higher crack growth rate than Nitronic 50 at 4 K [4-6].

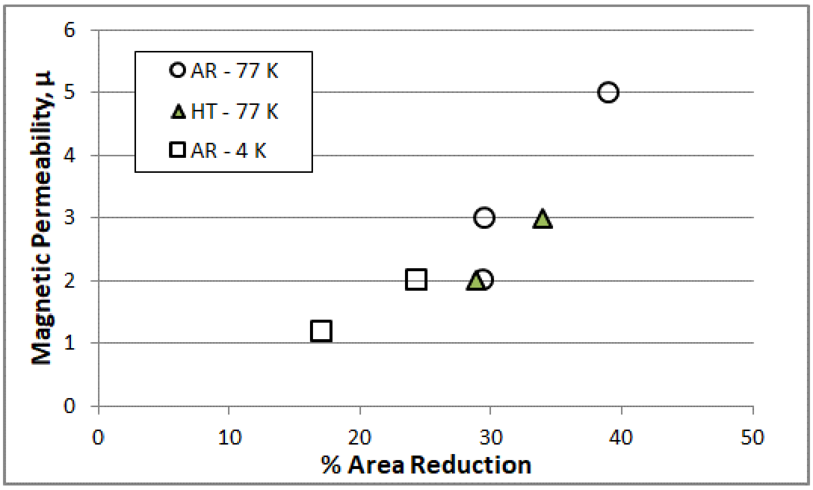

Figure 8: Relative permeability measurement taken at fracture locations on tensile specimens.

Table 2 and on the graph in Figure 8. While there was no indication of change in magnetic permeability after 295 K deformation, there was a noticeable increase in permeability after 77 K and 4 K deformation as was also observed in [3]. The increase in permeability is attributed to martensitic transformation where a percent of the FCC austenite crystal structure is converted to BCC martensite which may contribute to a reduction in toughness at cryogenic temperatures. Figure 8 also shows the magnetic transformation appears to be insensitive to the deformation temperature (77 K or 4 K) which is different than the findings in [3] where the amount of transformation at 4 K was supressed compared to 77 K.

5. Conclusions

The Nitronic 40 forged material researched here has excellent mechanical properties at both room and cryogenic temperatures that agree well with expectations and reference data in the literature. The mechanical properties are not degraded after exposure to a low-temperature short-time heat treatment that the material must undergo in future magnet construction procedures. Although the fracture toughness and FCGR are significantly reduced at 4 K compared to 77 K the retained values are considered excellent for cryogenic applications.

6. References

[1] JR Sims, J B Schillig, G S Boebinger, H Coe, A W Paris, M J Gordon, M D Pacheco, T G Abeln, R G Hoagland, M C Mataya, k Han, and A Ishmaku, The U. S. NHMFL 60 T Long Pulse Magnet Failure, IEEE Transactions on Applied Superconductivity, Vol. 12, No. 1, March 2002.
[2] Domack, M A, Fracture toughness and flaw growth in Nitronic 40 at cryogenic temperatures, Langley Research Center, Hampton, Va, NASA Technical Paper 2312, 1984.
[3] Tobler, R L and Reed, R P, Tensile and fracture behavior of a nitrogen-strengthened, chromium-nickel-manganese stainless steel at cryogenic temperatures, Elastic-Plastic Fracture, ASTM STP668,1979, pp. 537-552.
[4] D M McRae, R P Walsh, E N C Dalder, S Litherland, M Trosen and D J Kuhlman, Fatigue and fracture properties of a super-austenitic stainless steel at 295 K and 4 K, Advances in Cryogenic Engineering- Materials Vol. 60 (2013), pp. 59-66.
[5] R P Walsh and D M McRae, Report on CS tie plate base metal and weld characterization program, FSU/NHMFL Internal Engineering – Memo # MD&C-EM11-009E, 2013.
[6] A. Nyilas, Fatigue crack growth rate and fracture toughness properties at 7 K of forged Nitronic 50 material and its welding, Cryogenic Engineering & Materials Expertise (CEME) Inc., Germany, Document: 060741-14, CEME 2013.

Acknowledgments
This work was performed at the National High magnetic Field Laboratory which is supported by the National Science Foundation Cooperative Agreement DMR-1644779 and the State of Florida.