FRACTURE TOUGHNESS OF NITROGEN STRENGTHENED AUSTENITIC STEELS AT 4 K

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ABSTRACT

In this report the fracture toughness investigations of nitrogen strengthened austenitic steels used as structural material for the casing of large superconducting coils are represented. The safe operation of such large magnetic systems in a liquid helium environment necessitates the determination of fracture toughness data as an important material property for the purpose of preventing brittle fracture. The J-contour integral tests with small scale specimens were used for the measurements of the resistance curves at 4 K for AISI 316 LN type stainless steels. Beside the bulk material fracture properties, the fracture toughness of manual SMA-weldments and EB-weldments were also investigated. The establishment of the K_{IC}-critical data due to the J-integral measurements are demonstrated.

KEYWORDS

J-Integral test; austenitic steels; fusion technology materials; fracture toughness determination; cryogenics; superconducting magnets.

INTRODUCTION

In view of the long term increasing energy demand of the world, fusion technology represents an important milestone and a big challenge. Among the different approaches, magnetic confinement fusion represents a promising option on the way to the use of fusion energy. In this concept the high energetic plasma (ionic temperature ~10^8K) is magnetically confined in a vacuum vessel by high magnetic fields (8-12 Tesla). Such high magnetic fields can be produced only by superconducting coil systems. The presently favored approach is the toroidal configuration of the Tokamak. The international Large Coil Task (LCT) has the objective of demonstrating the feasibility of superconducting magnets of a size scalable to a Tokamak reactor (see Fig. 1).

One coil out of six test coils will be contributed by EURATOM (Komarek, 1978). The responsibility for this coil is with Kernfor-

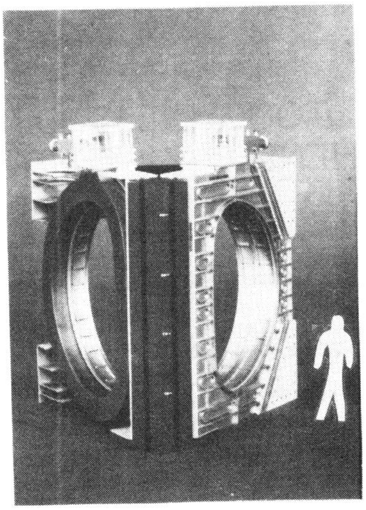

Fig. 1. Model of LCT magnet system

schungszentrum, Karlsruhe (KfK). The superconducting winding using a NbTi superconductor is enclosed in a casing made of structural material to withstand the high mechanical forces during service. The casing consists of two half sections with welded parts and is jointed by bolts in the midplane. The selected structural material for the casing is a fully austenitic stainless steel of about 45 mm thickness with high microstructural stability at 4 K. Because of the operation of this test facility at liquid helium temperatur the structural material must have the following inherent 4 K mechanical properties. A yield strength of >1000 MPa, tensile strength >1500 MPa, and a plane strain fracture toughness of >100 MPa$\sqrt{m}$. The objectives of this work is to represent the fracture toughness investigations of the selected structural material in a liquid helium environment. In addition to the bulk material properties, the determination of the fracture toughness values from different weldments at 4 K were of principle interest. From an engineering standpoint the safe design of the casing necessitates the K_{IC}-determination as an available method to prevent brittle fracture in such a large complex structure operating at extremely low temperature.

MATERIALS

The selected structural material for the casing is a nitrogen strengthened fully austenitic stainless steel with the German Specification No. 1.4429 (~AISI 316 LN) and has superior cryogenic mechanical properties. Material chemistry and tensile data are presented in TABLE 1. The alloy was supplied by the manufacturer in a commercially fully softened condition (heat treated at 1050°C for 1/2 hour and water quenched).

The materials used for the weld tests differed slightly from the bulk material designation. TABLE 2 represents the Electron Beam welded material (EB) with its main EB-Welding parameters and TABLE 3 shows the data for the weldment obtained by the manual Shielded Metal Arc process (SMA).

TABLE 1 Bulk Material Properties (Material 1.4429)

Chemistry, wt %

C	Si	Mn	P	S	Cr	Mo	Ni	N
0.032	0.41	1.26	0.016	0.016	16.7	2.68	13.7	0.16

Mechanical properties as determined by Albrecht (1980)

Temperature	Yield strength	Tensile strength	Elongation	Reduction of area
K	MPa	MPa	%	%
295	431	709	39	70
4	1160	1580	38	44

TABLE 2 EB-weld Material Properties (Material 1.4311)

Chemistry, wt %

C	Si	Mn	P	S	Cr	Mo	Ni	N
0.043	0.51	1.70	0.027	0.011	18.3	0.44	9.6	0.17

Mechanical properties for AISI 304N (Mann, 1977)

Temperature	Yield strength	Tensile strength	Elongation	Reduction of area
K	MPa	MPa	%	%
295	250	620	60	76
77	650	1520	48	59
4	770	1650	34	41

EB-welding parameter

Welding speed	5 mm/s
Weld penetration	~35 mm
Beam current	40 mA
Accelerating voltage	150 kV
Horizontal welding	

TABLE 3 SMA-Weld Material Properties (Material 1.4455)

Chemistry, wt %

C	Si	Mn	P	S	Cr	Mo	Ni	N
0.057	0.33	6.7	0.012	0.022	18.0	2.93	17.0	0.19

Mechanical properties (data from vendor)

Temperature K	Yield strength MPa	Tensile strength MPa	Elongation %
295	476	716	33

SMA-welding parameters

Electrode with commercial tradename Novonit 4455B
Filler material with 4 mm diameter and basic covering
Horizontal multipass welding
Electrode current d.c. 90/115 A

TECHNIQUES

Fracture toughness data K_{IC} at 4 K of similar austenitic materials are according to Mann (1977) in the range of 150-300 MPa$\sqrt{m}$. Therefore the determination of a valid K_Q at 4 K by Standard ASTM E-399 (1976) procedure using the above values result in compact tension (CT) speciment thicknesses between 43-172 mm. The necessary loads for these tests are between 200-3000 kN, respectively and are beyond the maximum load capacity of the present tensile testing apparatus used. Reducing the CT-specimen thicknesses shifts the linear elastic properties to elastic plastic fracture mechanics. The application of the path independent contour integral J, proposed by Rice (1968) and modified by Landes (1977), is assumed in this case as a suitable method for performing K_{IC}-measurements. The J-integral specimen configurations used were all CT-type with a nominal thickness of 2 3 mm as shown in Fig.2.

All dimensions were taken according to the standard ASTM E-399 (1976) and the specimens for the bulk material investigations were machined from a drawn bar of 200 mm diameter. The machined notches of the CT-specimens in this case were all in C-R-direction and have a 60° machined angle. The EB-welded CT-specimens were machined after EB-welding of two plates with an effective 30 mm thickness and the detection of the weldment by etching. The machined notches of these CT-specimens were supposed to propagate the cracks into Heat Affected Zone (HAZ) material. The SMA-welded test specimens were prepared for the evaluation of the weld centerline properties and were machined from a 45 mm thick plate after welding with consumable electrodes. The crack tip radii were measured to be in the range of 0.1-0.15 mm.

Six CT-specimens from bulk material, three from EB-welded, and two SMA-welded materials were precracked at room temperature. The precracking occurred with a pulsator fatigue machine at 30 Hz and at a

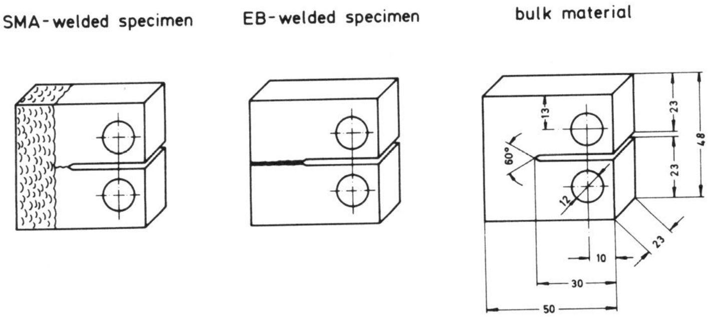

Fig. 2. Tested CT-specimens; all dimenions in mm

load of 8-15 kN. The fatigue crack propagation required 300 000 - 360 000 cycles and was controlled with a travelling microscope. A minimum fatigue crack length of 1.3 mm was achieved and the measured ratios (a/W) after fracture for the tested bulk material specimens were in order of 0.55-0.61. Here a is the original crack size including fatigue precrack and W the specimen width. Each specimen was immersed in liquid helium during the testing period in a cryostat especially built for this purpose to cause a crack extension of between 0 and 2.5 mm, and was single cycle pin loaded with a 200 kN Zwick type tensile testing machine. The cryostat is a closed system with a liquid nitrogen jacketed dewar. Typical liquid helium consumption for the system was approximately 250 litres for each test. The crosshead motion during the testing was kept at 0.1 mm/min. Load deflection was measured with a micrometer calibrated clip gauge (error from different readings <1.0 %, resolution at 4 K ~ 0.01 mm) by attached knife edges at the specimen front side. The displacement of the loading point was calculated by correcting the knife edge displacement with the rotational factor concept. The loading point displacement was in the order of 66 - 69% of the knife edge displacement depending on fatigue precrack length. The load and the deflection were electronically recorded on an X-Y plotter up to the point of the crack extension. After unloading with the same crosshead speed, the specimen was oxidized in air (heat tint) at 400°C for 25 minutes to mark the crack extension. The specimen was fractured then again at 4 K. The fatigue precrack length a_F and the physical crack length a_P due to crack extension were determined as the average of 24 readings along the thickness of each specimen. The J-contour integral J_I for each test was calculated using the formula derived by Merkle (1974)

$$J_I = \eta_r \frac{A}{B \cdot b} + \eta_c \frac{P_M \cdot \Delta_M}{B \cdot b} \qquad (1)$$

which assumes elastic plastic fracture mechanics with correction for the effects of axial forces acting in combination with the bending moment, where:

A = Area under load vs. load point displacement curve

b = initial uncracked ligament

B = specimen net thickness

P_M = load maximum at crack extension
Δ_M = load line displacement at crack extension
η_r = energy coefficient (Merkle, 1974)
η_c = complementary energy coefficient (Merkle, 1974)

Based on the total displacement of the applied load due to the crack for $a/W > 0.5$, the J_I-Integral was calculated for each specimen. The critical value of J_I was determined by the intersection of the assumed linear function $J_I = f(a_p)$ with the blunting line slope $2 \cdot \sigma_{flow}$ (average of yield strength and tensile strength at 4K recommended by Landes (1977)).

RESULTS AND DISCUSSION

The evaluation of test records by equation (1) yield J_I-values for each individual specimen. TABLE 4 represents the test records and the calculated J_I-data of the six CT-specimens for the bulk material.

TABLE 4 Bulk Material Test Results

Specimen No.	a_F mm	a_p mm	b mm	A Nmm	P_M N	Δ_M mm	a/W -	η_r -	η_c -	J_I N/mm
1	4.25	1.55	15.75	29538	56000	0.91	0.61	2.21	0.17	204
2	0.90	0.10	19.10	49380	89000	0.96	0.52	2.25	0.20	291
3	1.28	0.25	18.72	31787	75000	0.77	0.53	2.25	0.19	191
4	1.90	2.56	18.10	38300	74500	0.90	0.55	2.24	0.19	236
6	2.00	0.55	18.00	28375	69500	0.75	0.55	2.24	0.19	177
7	2.69	0.55	17.31	31760	65000	0.85	0.57	2.23	0.19	204

Specimen 2 was eliminated during the course of data qualification, because the fatigue precrack length was less than the recommended value of 1.3 mm. In addition, the fatigue precrack in this case was not thoroughly propagated along the thickness of the specimen, which resulted in the necessary high loads for crack extension. The data for the remaining five specimens have been assumed as qualified J_I-resistance curve data. Figure 3 shows J_I versus a_p of the tested specimens.

The intersection of the linear regression line with the blunting line results in a J_I-value of 181 N/mm. The correlation of the regression line was approximately 87%. The value of 181 N/mm is assumed to be the critical J_{IC} point. Conversion of J_{IC} to K_{IC} by the following equation

$$K_{IC}^2 = J_{IC} \cdot E/(1 - \nu^2) \qquad (2)$$

with the data of elastic moduli E = 220,5 GPa and the Poisson's contraction coefficient $\nu = 0.281$ taken from Mann (1977) for the stainless steel AISI 316 yields a plane strain fracture toughness K_{IC} at 4 K for the tested bulk material of 208 MPa$\sqrt{m}$.

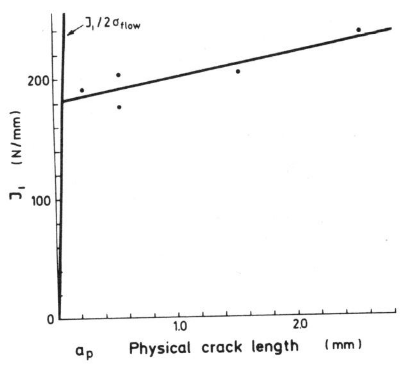

Fig. 3. J_I compared with crack extension for the bulk material

In the course of the data evaluation, certain errors will influence this determined value. For example the measurement of the deflection with the prepared clip gauge has an error of ± 1% due to the calibration uncertainty. The fatigue precrack length measurement is only accurate to about 0.1 mm due to the appearance of the stretch zone during loading, which results again in an error for J_I of about ± 1%. The rotational factor can be influenced by the blunting of the fatigue precrack and this was also estimated to be approximately ± 2%. Further, the accuracy of the J_I values has been estimated to be in the range of ± 10%. Therefore a total of approximately 15% variation of J_{IC} can be expected which, in turn, results in an overall error for K_{IC} of about ± 7%.

The determination of qualified J_I-values at 4 K for the EB-welded CT-specimens presented difficulties due to the high porosity of the welded section. The EB-welding condition of high vacuum decreases the solubility of nitrogen in the weld pool, which results in gas bubbles during the solidification of the weldment as reported by Brooks (1975) and Casey (1976). The fatigue precrack initiation at room temperature succeeded therefore only in one of the three EB-welded specimens. Figure 4 represents the fractured EB-welded specimen at the onset of the crack extension.

The metallographical examination indicated that the crack extension occurred in the vicinity of the HAZ. The determined J_I-values according to the equation (1) was 87 N/mm, considering a nominal specimen thickness of 23 mm. Subtracting the section affected by weld porosity from the nominal specimen thickness results in a net thickness of 12 mm. The J_I-value in this case amounts to 166 N/mm under consideration of the weld pool porosity. K_I values for both cases are 144 and 199 MPa$\sqrt{m}$ respectively.

The J_I-results for the two CT-specimens investigated with a precrack in the SMA-weldment were 112 N/mm and 97 N/mm with a crack extension at 4 K of 0.35 and 1.05 mm, respectively. Figure 5 represents the micrograph of the CT-specimen with the precrack in the SMA-weldment.

The ratio a/W for both SMA-welded specimen had to be selected as 0.69 in order to cause a deep precrack into the weldment. The fractured specimen with a crack extension of 0.35 mm showed, during the fractographic investigation, that approximately 10% of its precrack length was still situated in the bulk material. This fact results in a slightly higher value of J_I as compared with the CT-specimen having

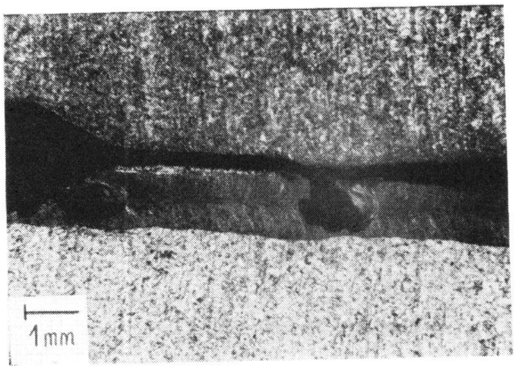

Fig. 4. Fracture of the EB-welded specimen at 4 K

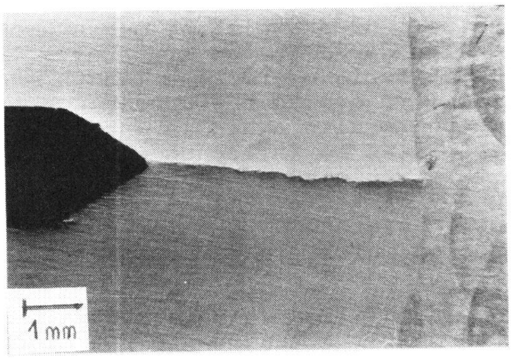

Fig. 5. CT-specimen with SMA-weldment and the precrack

1.05 mm crack extension and a detected total precrack in the weldment. Therefore it was assumed that the value of 97 N/mm with a_p = 1.05 mm is a qualified J_I-data for the SMA-weldment. Further, assumption of a similar slope of the function J_I, versus a_p for the weldment, results in a J_{IC} value of 87 N/mm and a K_{IC} of 144 MPa$\sqrt{m}$. These first results for the weldments must be confirmed by further investigations, which will be carried out in the next future.

The investigations of the fractured surfaces formed at 4 K showed microstructural instabilities for the bulk material and the EB-welded material. Figure 6 represents the fractured CT-specimen out of bulk material and its decoration with ferrous powder after magnetization. (Upper part of the Fig. 6 shows the undecorated specimen).

The flat portion is the precracked section at room temperature demonstrating the absence of ferromagnetism. The blunting of the precrack and the following crack extension at 4 K is ferromagnetic to a depth of approximately 5 - 10 grains along the fractured surface. The plastic deformation at 4 K results in a stress induced martensitic transformation. The SMA-welded fractured surfaces at 4 K showed no phase transformation, which indicates the strong austenitizing power of the elements N, Ni, and Mn in the heat chemistry as given in the TABLE 3.

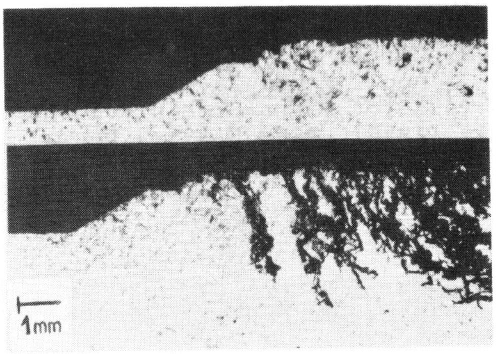

Fig. 6. The phase transformation of the crack extended surface (visualized by decoration)

CONCLUSIONS

1. The plane strain fracture toughness of the material 1.4429 (German specification Number) was measured at 4 K by the J-integral test. The obtained value for K_{IC} at 4 K is 208 MPa$\sqrt{m}$ with an estimated error of ± 7%.
2. At the HAZ of an EB-welded CT specimen (material AISI 304 N type stainless steel) the J-Integral test results in a K_I value of 144 MPa$\sqrt{m}$ at 4 K when not considering the weld defects by nitrogen release during the EB-welding under high vacuum.
3. Investigations of manual SMA-welded specimens by consumable electrodes results in a K_{IC} value for the weldment of 144 MPa$\sqrt{m}$ at 4 K
4. Microstructural stress induced martensitic transformation at 4 K was detected for the bulk material (1.4429) and the EB-welded material. The fractured SMA-weldment at 4 K showed no transformation.

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