

FRACTURE TOUGHNESS AND FATIGUE BEHAVIOUR OF AUSTENITIC STEELS
AT 77 AND 4 K

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Fracture toughness properties of several commercially available austenitic stainless steels were investigated at 77 K and 4 K. The toughness results were determined by J-Integral test using small size specimen. The J_{IC} of the tested materials (German Mat. No. 1.4301, 1.4541, 1.6903, 1.4435, 1.4404, and 1.4429) depend considerably on material fabrication technique. Whereas for the fatigue crack propagation behaviour the material processing plays not an important role. Fractographic, metallographic and SEM-analysis were used to support the above findings.

INTRODUCTION

Austenitic stainless steels are used increasingly for the construction of large superconducting magnets and in structures for cryogenic equipment. According to the engineering design criteria precise knowledge of toughness and fatigue properties of these materials are required to guarantee safe and reliable operation at cryogenic temperatures.

The purpose of the work reported here is to discuss thoroughly the fracture toughness measurements carried out at 77 K and 4 K for several commercially available austenitic chromium nickel steels.

METHOD OF TEST

The determination of the fracture toughness was done by the J-integral method using small size CT-specimens. Considering the standard LEFM method the size requirements for the tested materials give specimen dimensions, which are not practicable for measurements in cryogenic region, because of available test volume in the active zone of the used cryostats and the necessary high losses associated with large forces. The adopted multispecimen J-integral test with the modified Merkle-Corten derivation according to the ASTM E-813 standard procedure (1) has been reported recently by Krauth and Nyilas (2) for cryogenic application. In the present work the CT-specimen thicknesses of the tested materials varied from 10 mm to 40 mm. All other dimensions of the specimen lay within the ASTM dimensional requirements. The measurements were carried out either in liquid nitrogen (77 K) or in liquid helium (4 K) environment.

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For each specimen fatigue crack was initiated at ambient and propagated further at the test temperature before the crack extension.

TABLE 1 - Chemistry of the tested austenitic Cr-Ni steels in wt %.

Heat C	Si	Mn	P	S	Cr	Ni	Mo	Ti	N	Cr _{eq}	Ni _{eq}	
1	0,033	0.66	1.21	0.04	0.010	18.2	9.1	0.21	-	↑	19.4	10.7
2	0,053	0.35	1.47	0.03	0.005	17.9	9.2	0.27	-		18.7	11.5
3	0,063	0.44	1.22	0.03	0.010	17.4	9.8	0.29	0.32	-0.04	19.0	12.3
4	0,064	0.70	1.31	0.03	0.005	17.0	9.7	0.19	0.63		19.5	12.3
5	0,038	0.56	1.40	0.03	0.005	17.2	9.8	0.33	0.35	↓	18.4	11.7
6	0,023	0.35	1.21	0.03	0.010	16.4	12.4	2.7	-		19.6	13.7
7	0,035	0.36	1.24	0.03	0.010	16.7	11.0	2.1	-	0.16	19.3	12.6
8	0,032	0.41	1.26	0.01	0.010	16.7	13.7	2.7	-		20.0	20.0
9	0,021	0.54	0.97	0.02	0.006	17.6	13.9	2.7	-	0.17	21.1	20.1

Material Description

Commercially available materials provided by different vendors have been used for these investigations. Table 1 gives the heat chemistry of the tested materials. The nominal compositions were determined by quantitative spectroscopic analysis. The mean of three readings were taken as the nominal value. Nitrogen contents were taken from the vendor's analysis. In addition, the calculated Ni- and Cr-equivalents (representing the austenitizing power) for these materials are given in Table 1.

Specimens were machined from plate materials, which were in as received condition (1323 K for 1/2 hour and water quenched). Additional tests have been carried out with a microstructural refined and cold worked forging (heat No. 9), where the specimens were machined with different notch orientations.

RESULTS AND DISCUSSION

The evaluated J_I values of each individual J-test were plotted against the measured physical crack length a_p . The tearing line given by the qualified data are intersected with the blunting line to determine the critical J_{IC} of the tested material. Tensile tests of the materials (heat No. 1 - 7) were conducted to receive yield and ultimate tensile strength levels. For the nominally nitrogen free Cr-Ni steels a mean of flow strength σ_{Flow} of 775 MPa at 77 K has been verified. For the nitrogen strengthened and cold worked material (heat No. 9) tensile tests carried out by the vendor revealed 1200 MPa at 77 K and 1600 MPa at 4 K for the σ_{Flow} , respectively.

All J_{IC} values can be converted to fracture toughness (K_{IC}) data with reference to the relation $K_{IC}^2 = J_{IC} \cdot E / (1-\nu^2)$ ($E = 200$ GPa and $\nu \approx 0.3$).

Material 1.4301 (AISI 304 equivalent). Two heats (heat No. 1 and No. 2) were investigated in rolling T-L direction. The machined CT-specimen of the heat No. 1 had a width W of 40 mm and thickness B of 10 mm. Specimen of No. 2 had 30 mm and 23 mm, respectively. The results of J_I vs. a_p given in Fig. 1 reveal no significant difference for the both test series, although the chemical

compositions of these two heats differ considerably. The minor alloying element carbon, which should effect the fracture toughness shows a difference of nearly factor two. Therefore it is assumed that some other mechanism than the alloy composition affects the toughness level. The fracturing at 77 K gave indication of different mechanical working of the two heats. The observed fracture surface and the micrographs of the two heats were quite different in their fracture appearance and the inclusion sizes, respectively. In addition, one specimen of the heat No. 2 was tested at 4 K. A drastic decrease of the J_I value together with a nearly linear elastic load vs. displacement record was observed. With the assumption of a horizontal slope of the tearing line the measured J_I value should not differ much from the critical J_{IC} of this material at 4 K.

Material 1.4541 (AISI 321 equivalent). Heat No. 3 and 4 were investigated in rolling T-L direction. A significant difference of the J_{IC} values at 77 K resulted for these heats as shown in Fig. 2. The chemical composition given in Table 1 show no large variation of the alloying elements for both heats. An influence of mechanical processing was evident after analyzing the fracture appearance of the two heats. The fractured surfaces at 77 K given in Fig. 3 reveal different mechanical fibering degrees for these two series, which is assumed to affect the toughness. Similar observations have been made by English(3).

In addition, the material 1.6903 a version of 1.4541, which is used widely for low temperature applications, has been examined in T-L and L-T directions with 23 mm thick CT-specimen. A high J_{IC} difference between these two plane orientations was observed and the results are given in Fig. 2. One specimen from the L-T batch has been tested at 4 K and a factor of 10 decrease in J_{IC} has been found. The 4 K J_I value is in the vicinity of the blunting line and it is expected J_I corresponds here to J_{IC} .

Material 1.4435 (AISI 316 L equivalent). Figure 4 represents the J_I -test results of this material in T-L and L-T plane orientation. Tests of CT-specimen of this material in L-T orientation were not successful. A crack extension exceeding the blunting line could not be carried out, because of the high anisotropy of this plate material. The measured J_I values are all in the left hand portion of the blunting line. A further crack extension changed the crack plane orientation to the T-L direction, which is the principal direction of the mechanical working. Figure 5 gives the strong influence of the anisotropy on fracture. The Figures 6 and 7 represent the SEM-analysis of the fracture surfaces in L-T and T-L orientation. The clear difference between the two planes explains the toughness variation. It is expected that the real J_{IC} value would be above 1000 N/mm for the L-T orientation. In addition, tests with the material 1.4404 (similar to 1.4435 with a slightly lower nickel content) resulted a higher J_{IC} value in the T-L orientation as given in Fig. 4. Comparing the nickel content and the other alloying elements material 1.4404 should give a lower toughness than the 1.4435 (Table 1). However, the kind of mechanical working, also the anisotropy can overrule small variations of alloying elements and their influence on toughness.

Material 1.4429 (AISI 316 LN equivalent). The toughness behaviour of the material 1.4429 was already investigated by Nyilas and Krauth (4) for the utilization of this material in high strength, high toughness and high stiffness structures as required for the construction of large superconducting magnets e.g.. The measured J_{IC} exhibited a value of ~ 200 N/mm in case of the commercially available plate material (as received condition). However, an enhancement of the toughness was expected for the same material in the microstructural refined version. Therefore tests have been carried out with an Electro Slag Remelted (ESR) and forged material to cover the general characteristics and the metallurgical aspects of this material in a "clean" condition. The materials for these tests were provided by IRD, Newcastle/UK and the specimen were machined from a forged ring with the following dimensions: 913 mm outer, 832 mm inner diameter, length 400 mm. Different plane orientations (CA, AC, and CR) and two degrees of cold work (CW) (22 % and 28 %) were investigated. Although the strength was increased by the cold work a marked enhancement of the toughness could be observed (Fig. 8). 4 K yield and tensile strength of the ESR-material were 1435 to 1515 MPa and 1745 to 1810 MPa, respectively (heat 9). Whereas the plate material exhibits values of ~ 1200 MPa for the yield and ~ 1400 MPa for the tensile strength at 4 K (heat 8). The 4 K J_{IC} has been approximately doubled in the CR orientation compared to the plate material. Between 22 % and 28 % no difference in toughness could be detected. Strong anisotropy behaviour of the forging is also evident as shown in Fig. 8. The high toughness of the ESR-material is related to its microstructure. The micrographs (Figures 9 and 10) of the two materials indicate larger grain size and the high content of the inclusions in the plate material compared to the ESR-material. As seen in the micrograph (Fig. 9) the location of the inclusion are preferentially at the grain boundaries. It is assumed that this weakens the macroscopic feature as e.g. the fracture behaviour.

Fatigue crack growth measurements. Prior to the J_I -measurements the CT-specimen were used for fatigue crack propagation (FCP). The FCP tests were carried out at ΔK levels between 40 to 100 MPa \sqrt{m} . The external cyclic load was kept constant during the measurements. Evaluation was performed by measuring the fatigue crack extension after fracturing the specimen at 77 K. Assuming the validity of Paris Law all 4 K measurements of the material 1.4429 are within a band of $C_0 = 4$ to 12×10^{-12} and $n = 2.60$. The 77 K Paris Law constants of the material 1.4404 has been evaluated to be $C_0 = 14 \times 10^{-12}$ and $n = 4.20$ (da/dN in m/cycle, ΔK in MPa \sqrt{m}), which are not different from ambient FCP. The fabrication process and the anisotropy of the materials have little influence on the FCP-properties. No significant difference could be observed for the materials 1.4429 in different plane orientation.

SYMBOLS USED

- J_I = J-integral of mode I opening
 J_{IC} = critical J-integral value
SEM = scanning electron microscopy
 σ_{Flow} = mean of yield and tensile strength

- CT = compact tension specimen
 T-L = Transverse-longitudinal direction
 L-T = Longitudinal-Transverse direction
 LEFM = Linear Elastic Fracture Mechanics
 DCP = Direction of the crack propagation
 CA = circular axial direction
 AC = axial-circular direction
 CR = circular radial direction
 C_0 = Paris Law constant
 n = exponent of stress intensity range in Paris Law

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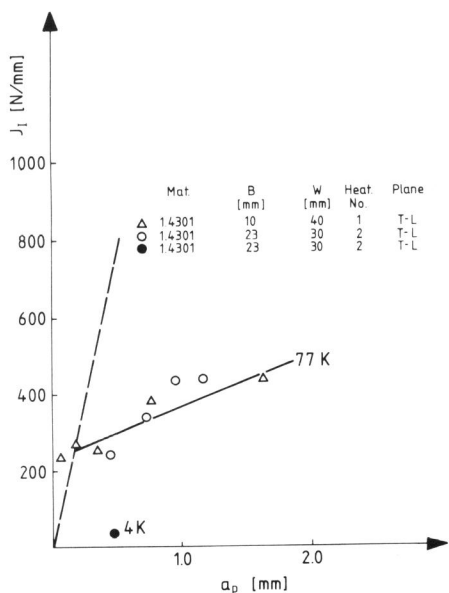


Figure 1 J_I vs. a_p of the material 1.4301

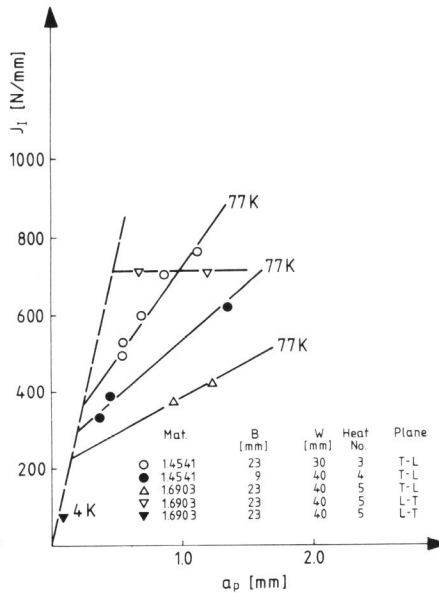


Figure 2 J_I vs. a_p of the materials 1.4541 and 1.6903

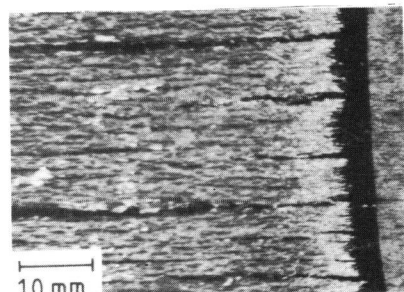
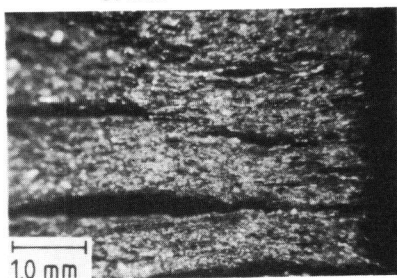


Figure 3 Fractured surface of the material 1.4541 at 77 K a) Heat 3 b) Heat 4 (DCP:←)

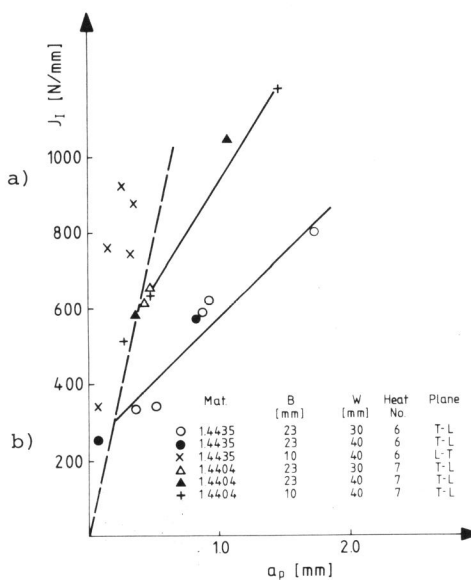


Figure 4 J_I vs. a_p of the materials 1.4435 and 1.4404

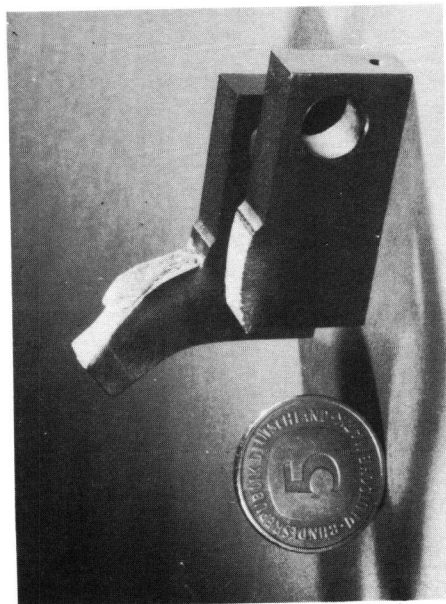


Figure 5 Anisotropic behaviour during fracture of the material 1.4435 at 77 K.

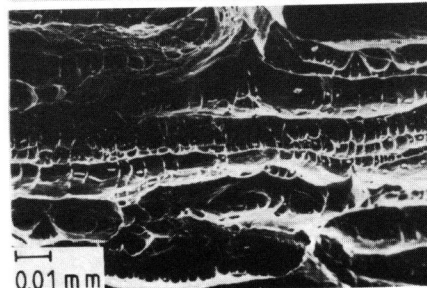
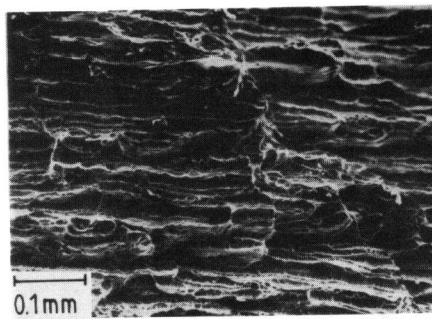


Figure 6 SEM analysis of the fracture surface in T-L direction of the material 1.4435 (DCP:+)

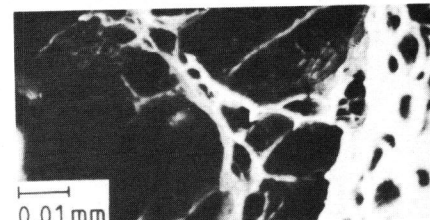
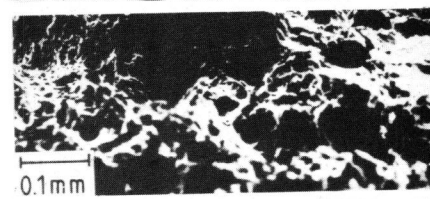
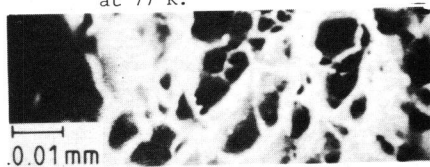
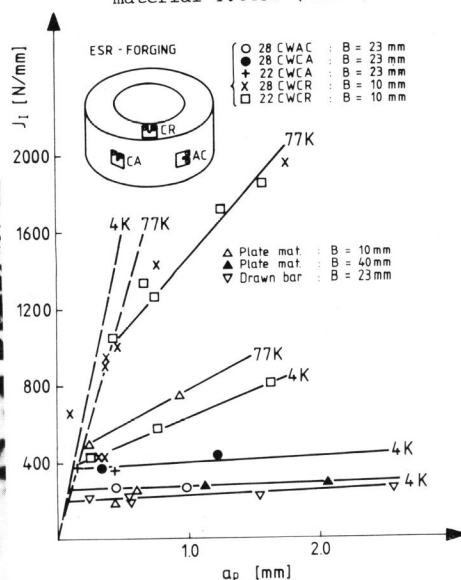


Figure 7 SEM analysis of the fracture surface in L-T direction of the material 1.4435 (DCP:+)



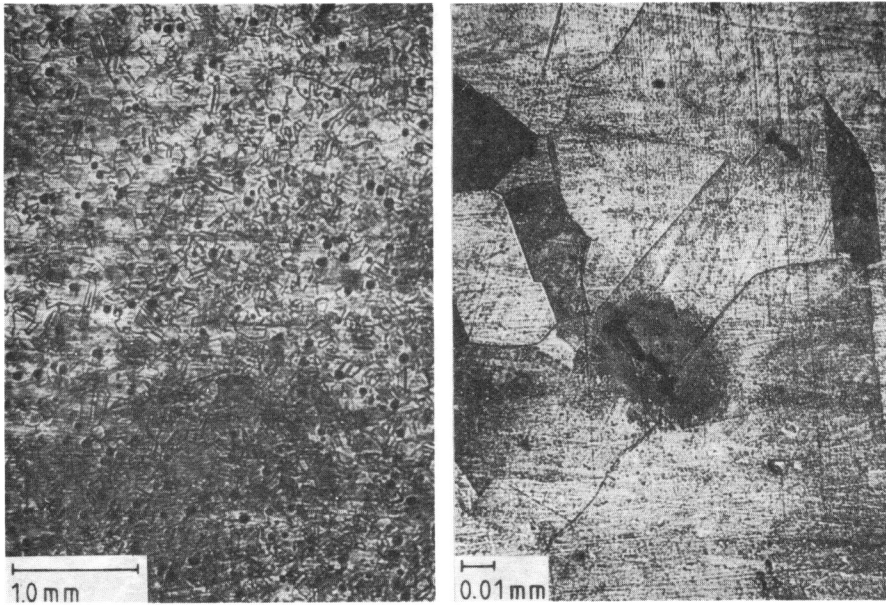


Figure 9 Micrographs of the plate material (1.4429) showing the microstructure
Left: obvious high content of inclusion acting as defects. Right: Site of the
inclusions

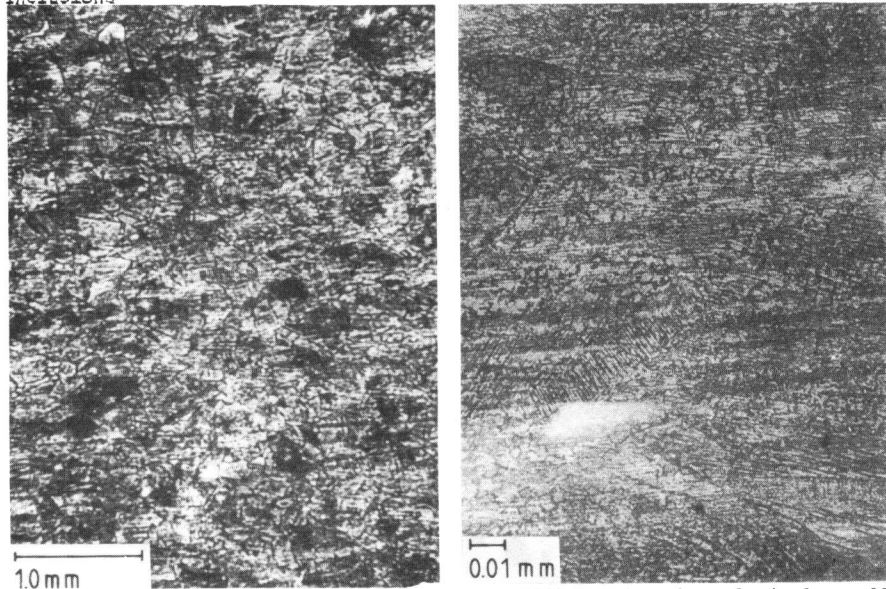


Figure 10 Micrographs of the ESR material (1.4429) showing the relatively small
grain sizes and very low inclusion content.