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Resistance of 10GN2MFA-A Low Alloy Steel to Stress Corrosion Cracking in High Temperature Water

Karel Matocha[1], Petr Čížek[1], Ladislav Kander[1] and Petr Pustějovský[2]
[1]*Material & Metallurgical Research, Ltd., Ostrava,*
[2]*VÍTKOVICE Heavy Machinery, J.S.C., Ostrava,*
Czech Republic

1. Introduction

In the second half of the last century the pressure vessel and the cold and hot collector bodies, ranking among the most stress and corrosion exposed components of the WWER 1000 (Water-Water Energy Reactor) horizontal steam generator (SG), were manufactured of the low alloy steel of type 10GN2MFA, the chemical composition of which is shown in tab. 1 (IAEA-EBP-WWER-07, 1997).

C	Mn	Si	S	P	Cr	Ni	Mo	V	Cu
0,08-0,12	0,80-1,10	0,17- 0,37	≤ 0,020	≤0,020	≤0,030	1,8-2,3	0,40-0,70	0,03-0,07	≤0,30

Table 1. Chemical composition of 10GN2MFA steel in line with standard element mass content, [%]

In the period from 1986 to 1995 cracks due to stress corrosion cracking (SCC) have been revealed after 7000 to 60 000 hours of operation in the ligaments between tube holes on the cold collectors in 25 steam generators at 9 units operated in the former Soviet Union (WWER SC 076,1993; Matocha, Wozniak, 1996, IAEA-EBP-WWER-07, 1997).

The cause analysis of collector cracking, carried out in the former Soviet Union, showed that the steel used, in all cases where cracking was experienced, has been produced by the open hearth furnace process (IAEA-EBP-WWER-07, 1997). Open heart furnace melted collector metal had local impurity concentrations, including manganese sulphide, which have deleterious effect on SCC resistance of low alloy steels in high temperature water environment.

Since the beginning of 1989 a number of modifications have been introduced in the former Soviet Union in the steam generators or in their operational conditions process (IAEA-EBP-WWER-07, 1997). One of them was the modification of the steelmaking process. Only steel produced by electroslag remelting has been used for the manufacturing of collector bodies. The electroslag remelting process (ESR) allows to enhance considerably micro-cleanliness of the steel and consequently the resistance of the 10GN2MFA steel to SCC in high temperature water environment.

In the period from 1991 to 1994, the eight steam generators were manufactured in VÍTKOVICE, J.S.C. for WWER 1000 Temelín NPP. The collector bodies of Temelín NPP steam generators were made of doubly vacuum treated 10GN2MFA steel (first on DH equipment and then by pouring in vacuum) so as to minimize the gas concentration and to secure a homogeneous chemical composition. Steps were taken in all heats to keep down the content of impurity elements. The typical chemical composition of the steel is shown in tab. 2 (WWER SC 076, 1993).

C	Mn	Si	S	P	Cr	Ni	Mo	V	Cu
0,10	0,86	0,27	0,008	0,009	0,19	2,16	0,43	0,05	0,08

Table 2. The typical chemical composition of 10GN2MFA steel of Temelín NPP collector bodies [mass %]

No occurrence of environmentally assisted cracking has been noticed till now after approximately 79 000 hours of operation of the steam generators at Temelín NPP.
According to contemporary Russian technical specifications TU 0893-014-00212179-2004, the WWER 1000 SG collector body must be manufactured of the electroslag remelted 10GN2MFA steel (10GN2MFA-S). Sulphur content must be kept lower than 0,005% and the concentration of phosphorus must be lower than 0,008%. The requirement for the chemical composition and tensile properties of the 10GN2MFA-S steel are summarized in tab.3 and tab.4.

	C	**Mn**	**Si**	**P**	**S**	**Cu**	**Ni**	**Cr**	**Mo**	**V**	**Ti**	**Al**
min.	0,08	0,80	0,17	-	-	-	1,80	-	0,40	0,03	-	0,005
max.	0,12	1,10	0,37	0,008	0,005	0,30	2,30	0,30	0,70	0,07	0,015	0,035

Table 3. The requirements for the composition of the steel used for the manufacture of the WWER 1000 collector body.

Test temperature	Yield point	Yield strength	Tensile strength	Elongation	Reduction of Area
+20°C	345-590 MPa		540-700 MPa	18%	60%
350°C		min. 295 MPa	min. 490 MPa	15%	55%

Table 4. Requirements for the tensile properties of the 10GN2MFA steel.

In the course of the nineties of the last century, the significantly modernised technology of the 10GN2MFA steel production (electric arc furnace, refining in ladle furnace, vacuum degassing, bottom pouring under argon protection into mould) was established in VÍTKOVICE Heavy Machinery, J.S.C. It enables to guarantee the content of sulphur lower than 0,005% and the content of phosphorus lower then 0,008%.
However Vítkovice Heavy Machinery J.S.C. does not own the electroslag remelting plant. To be able to manufacture the WWER 1000 collector bodies for AES 2006 reactor plant, it was necessary to prove that there is no difference between electroslag remelted 10GN2MFA steel (10GN2MFA-S) and the steel manufactured by modernised VÍTKOVICE Heavy Machinery technology (10GN2MFA-A) from the point of view of fracture behaviour and resistance to stress corrosion cracking in high temperature water environment. For this purpose three forgings of WWER 1000 SG collector body were manufactured in VÍTKOVICE Heavy Machinery, J.S.C. (see Fig.1).

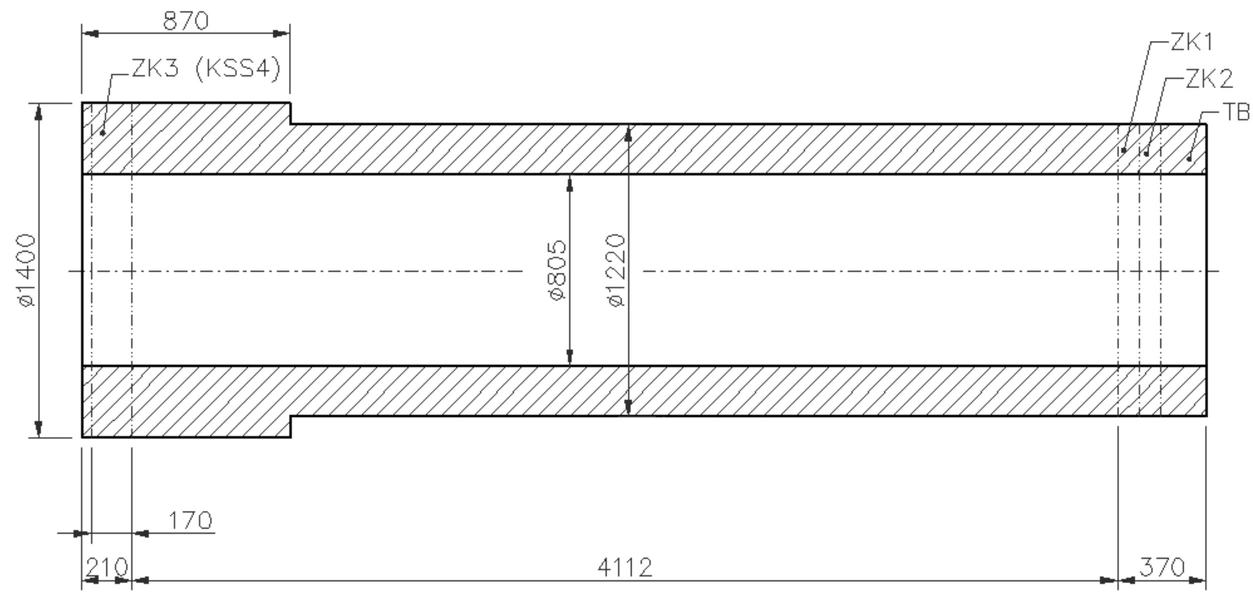

Fig. 1. Dimensions of the collector body after rough machining

This work summarizes the results of the evaluation of the tensile properties, fracture behaviour and resistance to stress corrosion cracking in high temperature water environment of the collector body forging No 1 material. The resistance to SCC in high temperature water environment was evaluated both in laboratories of MATERIAL & METALLURGICAL RESEARCH, Ltd. (M&MR, Ltd.) and also in laboratories of CNIITMASH, a joint-stock company, Moscow (Russia). The testing procedure used was proposed by CNIITMASH. The obtained results were compared with the results published by CNIITMASH for the 10GN2MFA-S steel (Yukhanov,V.A. & co-workers, 2009, Yukhanov,V.A. & co-workers, 2010).

2. Testing material and experimental techniques

The testing ring ZK1 (see Fig.1) after simulated post weld heat treatment was used as a testing material. Its chemical composition is shown in tab.5.

C	Mn	Si	P	S	Cu	Ni	Cr	Mo	V	Ti	Al
0,12	0,90	0,22	0,006	0,002	0,05	1,88	0,048	0,55	0,042	0,004	0,021

Table 5. The chemical composition of the testing material.

Metallographic analysis proved very low content of non-metallic inclusions of globular shape. The fine grained microstructure of the steel is bainitic with the average initial austenitic grain size G= 6-7.
Fig.2 illustrates schematically the location and orientation of the test specimens used for the determination of tensile and fracture characteristics (impact tests, fracture mechanics tests) and the round bar tests used for the evaluation of resistance to SCC by slow strain rate test.

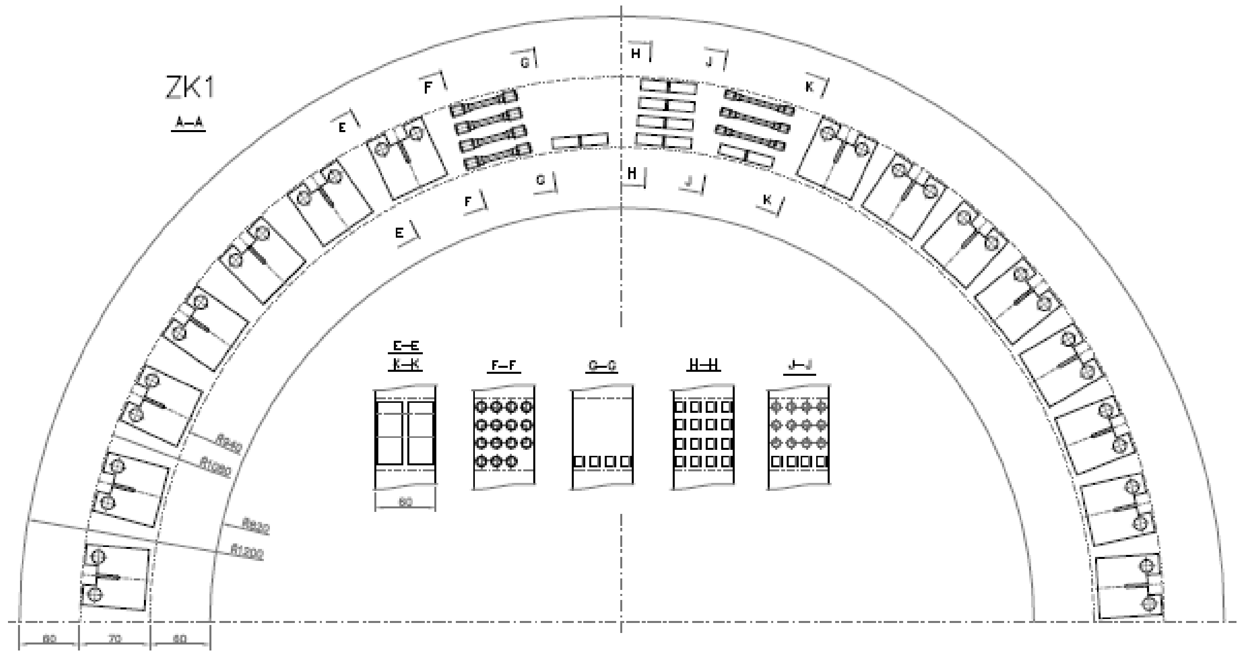

Fig. 2. Orientation of the test specimens in the testing block

As the characteristic feature of the fracture behaviour of 10GN2MFA steel at temperatures from 20°C to 320°C was found to be a ductile stable crack growth (Matocha,K., Wozniak,J., 1995, Matocha,K., Rožnovská,G. & Hanus, V.,2007), the variation in J with crack growth (J-R curve) was investigated at 290°C using multiple specimen method . 1CT (compact tension, 25 mm in thickness) specimens were used for the determination of J_{IC}. Tests were carried out in a stroke control at the speed of 0,5 mm/min. in accordance with GOST 25.506-85 on MTS 500 kN servo-hydraulic testing machine. Loading of the test specimens was realized in the three zone furnace specially developed for the determination of the fracture toughness at elevated temperatures. The design of the furnace enables to measure the crack mouth opening during the test (see Fig.3).

The slow strain rate tests of both round bar test specimens 5 mm in diameter and fatigue pre-cracked 1CT test specimens (ISO 7539-7:2005, ISO 7539-9:2003) were used for the evaluation of the resistance of the 10GN2MFA-A steel to SCC in high temperature water environment (260°C, 290°C). The testing parameters for the evaluation of resistance of the steel against stress corrosion cracking (strain rates, initial dissolved oxygen content) were proposed by CNIITMASH.

SCC tests were performed in static autoclave 11 l in volume fitted with INOVA servo-hydraulic testing machine. Slow strain rate tests of round bar test specimens were carried out under stroke control in demineralised water at temperatures 260°C and 290°C at strain rate $1{,}4.10^{-7}\ s^{-1}$. Initial concentrations of dissolved oxygen 0,5 ppm, 1,5 ppm and 4,5 ppm were obtained by adding 50% H_2O_2 into demineralised water which was got rid of carbon dioxide. The susceptibility to SCC is evaluated on the basis of a comparison of reduction of area determined in air and in water environment at the same testing temperature. Slow strain rate tests of the fatigue pre-cracked 1CT specimens were carried out also under stroke control only at 260°C at initial concentration of dissolved oxygen 4,5 ppm and stroke rate $1{,}8.10^{-6}$ mm/s.

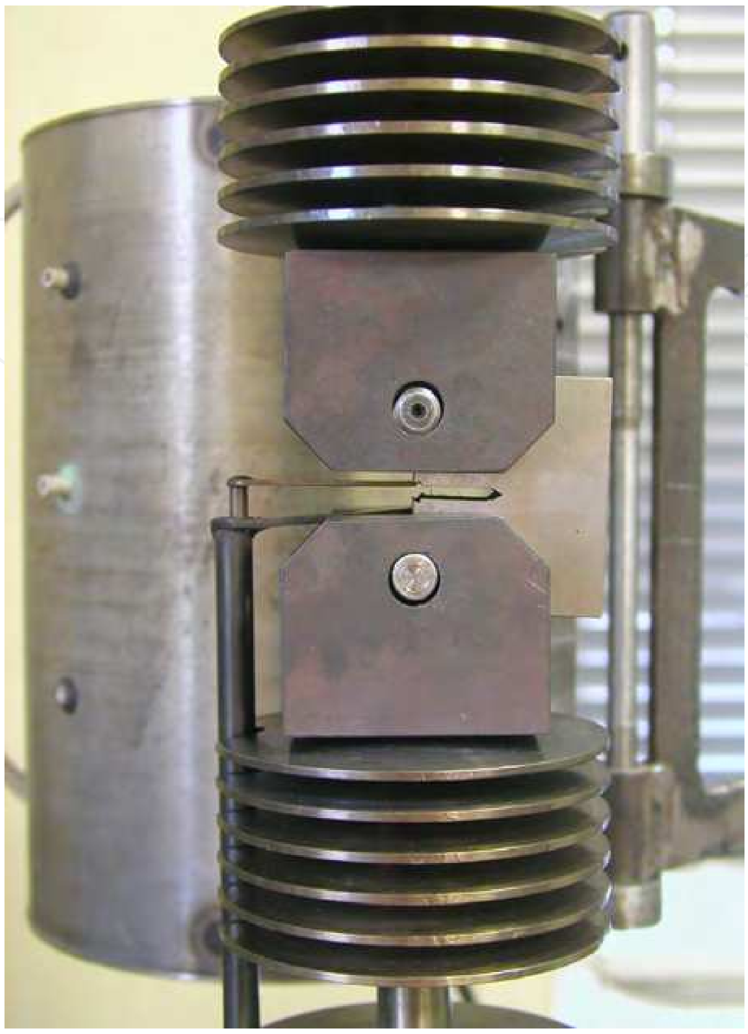

Fig. 3. The testing facility used for the determination of J-R curve in air environment at 290°C

3. Results and discussion

Summary of tensile characteristics is shown in table 6. Tensile tests at +20°C, 260°C, 290°C and 350°C were carried out on MTS 100 kN servo-hydraulic testing machine using round bar test specimens 5 mm in diameter like the test specimens used for the slow strain rate tests in high temperature water environment.

Test temperature [°C]	Yield point [MPa]	Yield strength [MPa]	Tensile strength [MPa]	Elongation [%]	R.A. [%]
+20	512		615	25,9	78
260		458	609	20,5	74
290		460	606	24,0	75
350		442	567	23,4	76

Table 6. Results of tensile tests of the studied steel at ambient and elevated temperatures.

Fig.4 and Fig.5 shows the temperature dependences of impact fracture energy and shear fracture area of the steel investigated the steel investigated in temperature range from -80°C to 320°C.

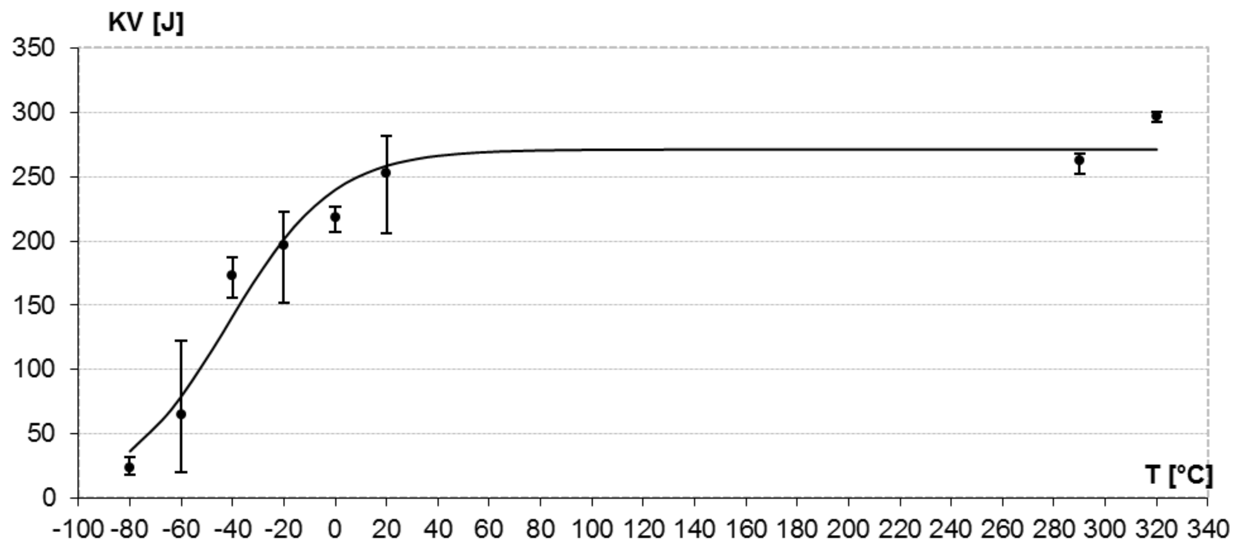

Fig. 4. Temperature dependence of impact fracture energy in the temperature range from -80°C to 320°

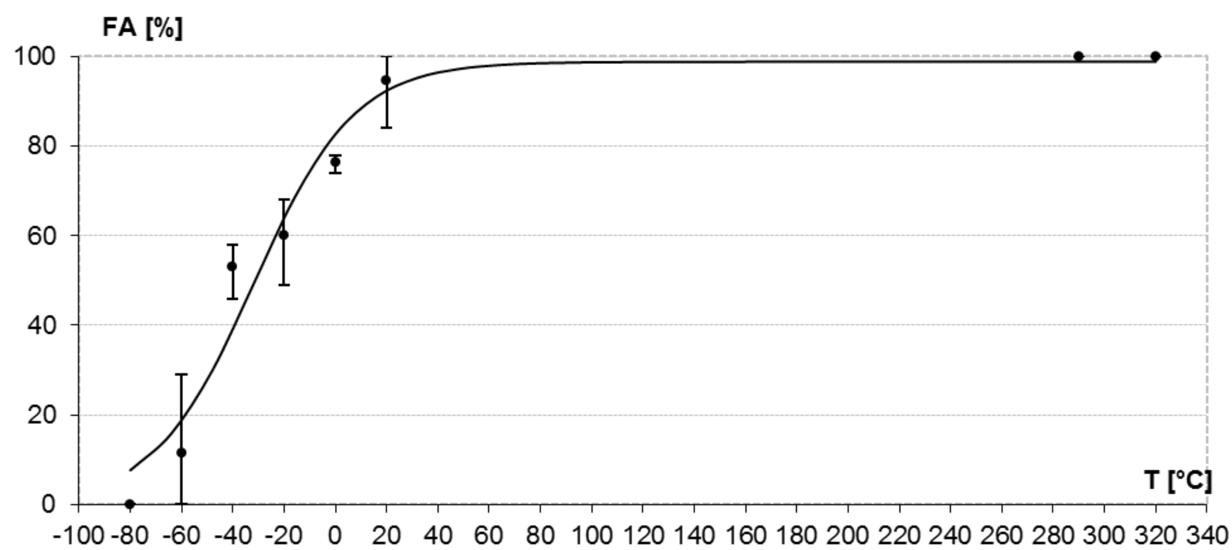

Fig. 5. Temperature dependence of shear fracture area in the temperature range from -80°C to 320°

Critical temperature of brittleness and FATT determined from the results of impact tests were found to be T_{k0} = -50°C and FATT = -31°C. Results of tensile and impact tests satisfy the requirements of the technical specifications TU 0893-014-00212179-2004.
Table 7 summarizes the results of fracture mechanics tests of the studied steel in air at 290°C performed in accordance with GOST 25.506-85. Fig.6 shows the J-R curve obtained. Fracture toughness J_{IC} determined from the J-R curve equals to J_{IC} = 237 N/mm.

No of test specimen	a_o [mm]	P_i [N]	A_{pi} [N.mm]	Δa [mm]	J_i [N/mm]
ZK1-40	26,0	74 671	211 395,1	1,68	629,8
ZK1-41	25,7	73 103	102 362,1	0,53	336,4
ZK1-42	25,6	75 662	144 246,0	0,78	447,2
ZK1-43	25,8	71 759	78 031,2	0,39	273,2
ZK1-44	25,8	72 069	172 442,1	1,28	517,2
ZK1-45	25,7	69 036	52 448,2	0,19	200,6
ZK1-47	25,9	71 008	242 460,2	1,80	700,7
ZK1-48	25,6	75 152	312 651,9	2,09	874,3

Table 7. Results of fracture mechanics tests in air at 290°C

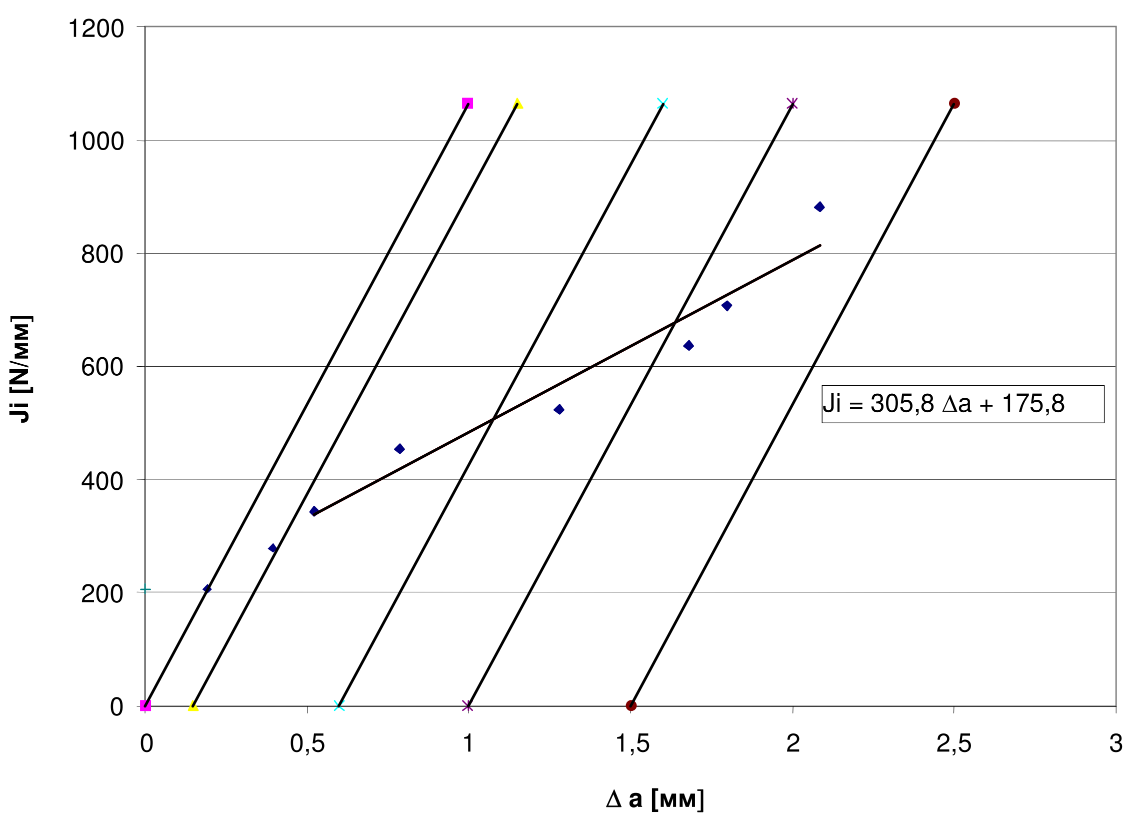

Fig. 6. J-R curve of the studied steel at 290°C determined by multiple test specimen method

This value is in a very good agreement with J_{IC} determined in air at 290°C for 10GN2MFA steel used for the manufacture of the collector bodies of Temelín NPP steam generators (Matocha,K., Wozniak,J., 1995).

Results of slow strain rate tests of round bar test specimens, made of 10GN2MFA-A steel, in high temperature water environment performed in both laboratories are summarized in table 8.

Test temperature	Initial concentration of dissolved oxygen	CNIITMASH	M&MR
		Reduction of area [%]	Reduction of area [%]
260°C	4,5 ppm	9,3	8,6
		9,6	12,3
		8,7	12,4
	1,5 ppm	69,9	> 54,1
		67,5	68,9
		66,5	
	0,5 ppm	76,3	73,2
		77,1	71,1
290°C (M&MR) 300°C (CNIITMASH)	1,5 ppm		75,2
	4,5 ppm	74,8	77,0

Table 8. Results of slow strain rate tests of 10GN2MFA-A steel carried out in CNIITMAS and in M&MR, Ltd.

Table 8 shows a very good agreement between the results obtained in both laboratories for all initial concentrations of dissolved oxygen investigated. Fracture surfaces of the failured test specimens were examined by scanning electron microscope JEOL JM-5510. Fracture surfaces of the test specimens having low level of reduction of area showed the same fractographic features formerly observed on fracture surfaces of 1CT tests specimens tested in aerated distilled water (see Fig.7), (Matocha,K., Wozniak,J., 1995).

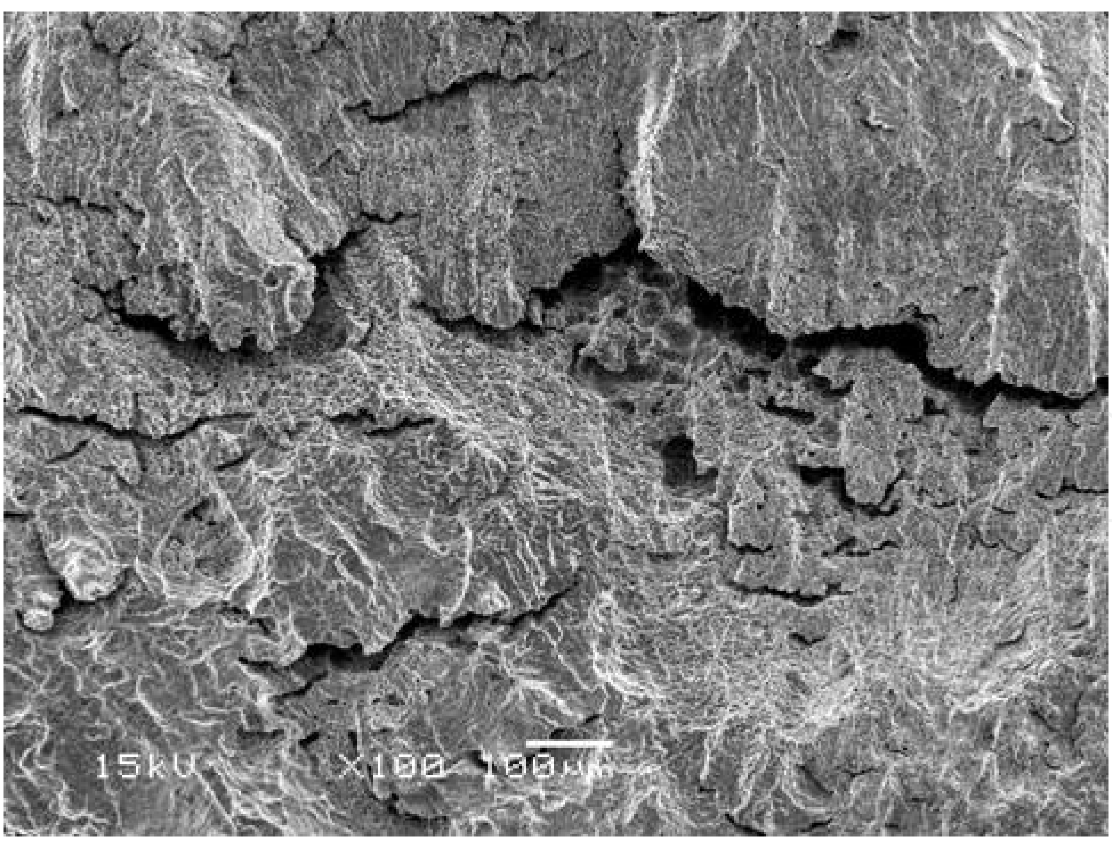

Fig. 7. Morphology of the fracture surface of the test specimen tested in water environment at 260°C (initial O_2 = 4, 5 ppm, R.A. = 12, 4%)

Metallographic evaluation of test specimen cross sections showed that the transverse microcracks observed on the fracture surface (see Fig.7) were initiated in the process zone ahead of the growing crack and probably contributed to the increase of crack growth rate (see Fig. 8), (Matocha,K., Rožnovská,G. & Hanus,V, 2007). In all cases the stress corrosion cracks were initiated from the pits (see Fig.9).

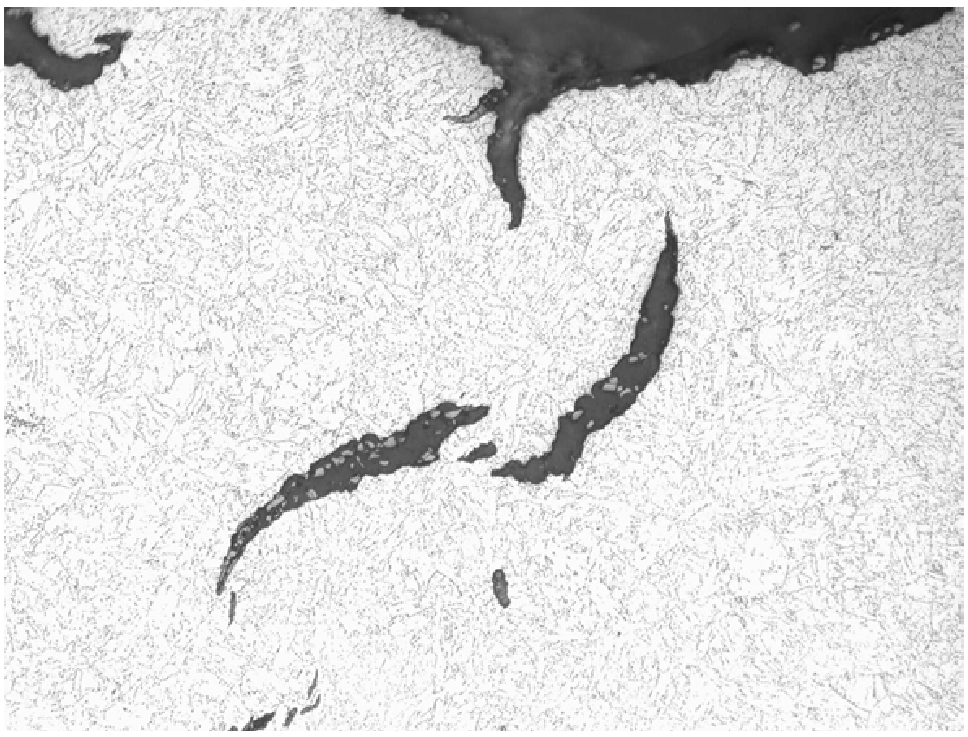

Fig. 8. The occurrence of transverse microcracks on fracture surface and in process zone of the growing crack (Matocha,K., Rožnovská,G. & Hanus,V, 2007).

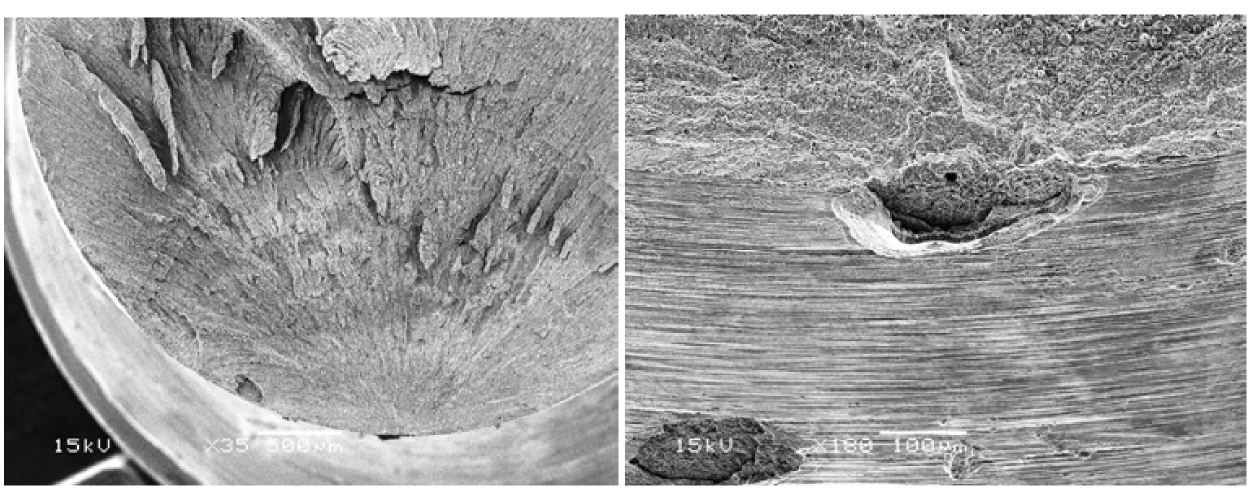

Fig. 9. Fracture surface of the test specimen tested in water environment at 260°C
(initial O_2 = 4,5 ppm, R.A. = 12,4%)

Table 9 shows the comparison of the slow strain rate tests results obtained in CNIITMASH using round bar specimens manufactured of 10GN2MFA-A and 10GN2MFA-S steels at a strain rate $1{,}4.10^{-7}$ s^{-1}.

Test temperature	Initial concentration of dissolved oxygen	10GN2MFA-A	10GN2MFA-S.
		Reduction of area [%]	Reduction of area [%]
260°C	4,5 ppm	9,3	7,8
		9,6	8,7
		8,7	
	1,5 ppm	69,9	54,9
		67,5	
		66,5	
	0,5 ppm	76,3	72,3
		77,1	
300°C	4,5 ppm	74,8	75,4

Table 9. Results of slow strain rate tests of the 1OGN2MFA-A and 10GN2MFA-S performed in CNIITMASH.

The results obtained proved that the resistance of both steels against the stress corrosion cracking in high temperature water environment, evaluated by slow strain rate tests of round bars test specimens, is almost identical.
Results of slow strain rate tests of fatigue precracked 1C(T) specimens carried out in water environment at 260°C, initial dissolved oxygen content 4,5 ppm ppm and stroke rate $1{,}8.10^{-6}$ mm/s are summarized in table 10. The variation in δ (crack tip opening displacement) with crack advance Δa was investigated using a multiple specimen method. Fracture surfaces created by stable crack growth during autoclave tests were examined by scanning electron microscope JEOL JM-5510.

Load line displ. [mm]	v_{pl} [mm]	P_{max} [N]	δ [mm]	K_δ [MPa.m$^{1/2}$]	Δa [mm]
1,66	0,1	36595	0,053	100,7	3,05
1,16	0	28970	0,019	60,3	0
1,13	0	30160	0,019	59,7	0,51
1,09	0,06	27213	0,031	79,6	0
1,18	0,04	27810	0,027	74,5	1,74
1,43	0,09	31783	0,048	98,7	2,94
1,05	0,04	25428	0,024	69,6	0,35
1,86	0,33	23865	0,134	165,4	8,66
0,90	0,02	21462	0,015	54,7	0,68
1,19	0,05	26857	0,029	77,3	1,87
1,42	0,05	30003	0,033	82,8	3,27
0,78	0	16370	0,005	32,7	0
1,02	0,02	23913	0,017	59,0	0,93

Table 10. Results of slow strain rate tests of 1CT specimen in demineralised water at 260°C and initial dissolved oxygen content 4,5 ppm.

Fig. 10 shows the variation of K_δ with crack advance Δa. From the variance analysis of the results obtained follows that for the probability of 95% $K_{\delta in}$ equals $K_{\delta in} = 54 \pm 16$ MPa.m$^{1/2}$. The average calculated environmentally assisted crack growth rate was found to be $v_{cor} = 1{,}8.10^{-5}$ mm/s. Fractographic analysis of the fracture surfaces created by SCC in high temperature water revealed the same fractographic features (see Fig.11) observed on fracture surfaces of round bar test specimens tested at the same initial dissolved oxygen concentration.

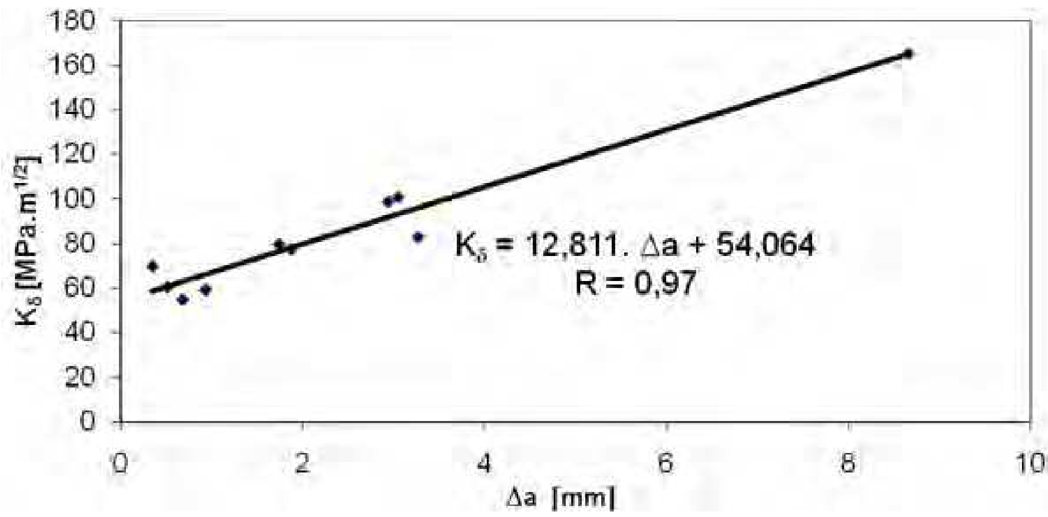

Fig. 10. The variation of K_δ with crack advance Δa (initial O_2 = 4,5 ppm, stroke rate $1{,}8.10^{-6}$ mm/s).

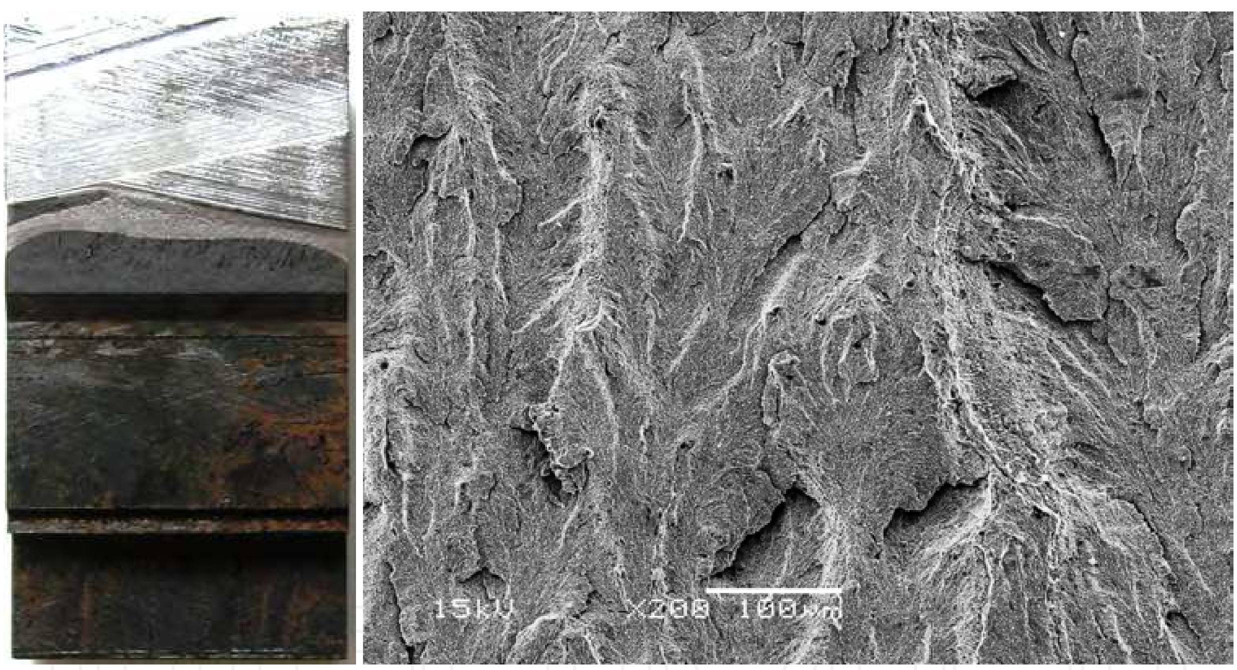

Fig. 11. Morphology of the fracture surface created by SCC, t = 260°C, O_2 = 4,5 ppm, v_{cor} = $3{,}7.10^{-5}$ mm/s

4. Conclusion

From the results obtained in this study it follows that:

1. Results of tensile and impact tests of the 10GN2MFA-A steel manufactured by VÍTKOVICE Heavy Machinery, J. S. C., satisfy the requirements of the Russian technical specifications TU 0893-014-00212179-2004.
2. The resistance of 10GN2MFA-A and 10GN2MFA-S steels against the stress corrosion cracking in high temperature water environment with initial dissolved oxygen contents 0,5 ppm, 1,5 ppm and 4,5 ppm was found to be almost identical.

3. Stress corrosion cracks observed on the fracture surfaces of the round bar test specimens failured due to slow loading in high temperature water with increased initial dissolved oxygen content were in all cases initiated from the corrosion pits.
4. The crack growth in high temperature water with increased initial dissolved oxygen content was accompanied by the significant occurrence of transverse micro-cracks initiated in the process zone ahead of the growing crack.
5. Rates of corrosion crack growth determined by slow strain rate tests are typical for stress corrosion cracking of low alloy bainitic steels in high temperature water environment with increased initial dissolved oxygen content.

5. Acknowledgement

This paper was created in the project No.CZ.1.05/2.100/01.0040 "Regional Materials Science and Technology Centre" within the frame of the operation programme "Research and Development for Innovation" financed by the Structural Funds and from the state budget of the Czech Republic.

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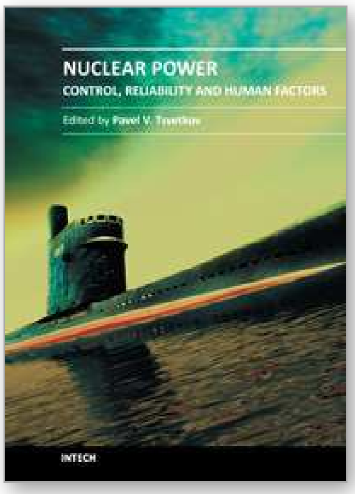

Nuclear Power - Control, Reliability and Human Factors
Edited by Dr. Pavel Tsvetkov

ISBN 978-953-307-599-0
Hard cover, 428 pages
Publisher InTech
Published online 26, September, 2011
Published in print edition September, 2011

Advances in reactor designs, materials and human-machine interfaces guarantee safety and reliability of emerging reactor technologies, eliminating possibilities for high-consequence human errors as those which have occurred in the past. New instrumentation and control technologies based in digital systems, novel sensors and measurement approaches facilitate safety, reliability and economic competitiveness of nuclear power options. Autonomous operation scenarios are becoming increasingly popular to consider for small modular systems. This book belongs to a series of books on nuclear power published by InTech. It consists of four major sections and contains twenty-one chapters on topics from key subject areas pertinent to instrumentation and control, operation reliability, system aging and human-machine interfaces. The book targets a broad potential readership group - students, researchers and specialists in the field - who are interested in learning about nuclear power.

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Karel Matocha, Petr Čížek, Ladislav Kander and Petr Pustějovsky (2011). Resistance of 10GN2MFA-A Low Alloy Steel to Stress Corrosion Cracking in High Temperature Water, Nuclear Power - Control, Reliability and Human Factors, Dr. Pavel Tsvetkov (Ed.), ISBN: 978-953-307-599-0, InTech, Available from: http://www.intechopen.com/books/nuclear-power-control-reliability-and-human-factors/resistance-of-10gn2mfa-a-low-alloy-steel-to-stress-corrosion-cracking-in-high-temperature-water

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University Campus STeP Ri
Slavka Krautzeka 83/A
51000 Rijeka, Croatia
Phone: +385 (51) 770 447
Fax: +385 (51) 686 166
www.intechopen.com

InTech China
Unit 405, Office Block, Hotel Equatorial Shanghai
No.65, Yan An Road (West), Shanghai, 200040, China
中国上海市延安西路65号上海国际贵都大饭店办公楼405单元
Phone: +86-21-62489820
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