RELATIONSHIP BETWEEN MICROSTRUCTURE PARAMETERS AND STATIC FATIGUE OF STRUCTURAL STEELS IN SOUR ENVIRONMENT

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ABSTRACT

The paper reports a study of the susceptibility to static fatigue in sour environment of twelve heats of C 105 grade steel used for the production of drill pipes. The heats differed in the sulphur content and in the microalloying with titanium, niobium and vanadium, respectively. The tests were performed in NACE solution (in the presence of H_2S with a pH value of 3,5) at loads of 500, 550 and 600 MPa. The optimum microstructure properties for attainment of the higher SSC resistance of the investigated steel are presented.

KEY WORDS

Static fatigue, sulphide stress corrosion (SSC), hydrogen embrittlement, hydrogen trapping, SSC resistance.

INTRODUCTION

The static fatigue of structural steels in sour environment represents a very important material property, e.g. of the drill pipes, and this defines the level of the safety and reliability in service of those products during their exploitation. This degradation effect, known as the sulphide stress corrosion (SSC) is due to the hydrogen embrittlement of the investigated steel.

The paper presents a contribution to the evaluation of the influence of microstructure parameters (a distribution of sulphides and carbides and/or carbonitrides of microalloying elements) on the attained SSC resistance (Mazancová, 1991; Mazancová and Mazanec, 1992). These particles are acting as the potential traps of hydrogen atoms and contribute to the uniform hydrogen distribution in the matrix.

EXPERIMENTAL MATERIAL AND TECHNIQUE

The investigation was performed on the twelve heats of the C 105 grade steel used for the production of drill pipes applied in the petroleum industry. The heats differed in their sulphur content and in their microalloying with the addition of titanium, niobium and vanadium. The chemical compositions of the investigated heats are listed in Table 1. The tin content varied between 0,008 and 0,012% and the arsenic content was not higher than 0,007%.

Table 1. Chemical composition of investigated heats in wt%

Heat	C	Mn	Si	P	S	V	Nb	Ti	B
A	0,39	1,31	0,43	0,015	0,013	0,11	0,050	0,040	0,0020
B	0,39	1,30	0,43	0,014	0,014	0,13	0,040	0,040	0,0020
C	0,39	1,30	0,43	0,033	0,020	0,10	0,050	0,030	0,0020
D	0,38	1,27	0,42	0,030	0,018	0,11	0,044	0,034	0,0018
E	0,34	1,42	0,35	0,013	0,004	0,07	0,040	0,040	0,0015
F	0,32	1,25	0,23	0,012	0,004	0,13	-	-	-
G	0,34	1,29	0,22	0,013	0,003	0,15	-	-	-
H	0,36	1,49	0,38	0,011	0,006	0,09	0,047	0,041	0,0012
I	0,35	1,39	0,31	0,012	0,008	0,10	0,050	0,036	0,0014
J	0,37	1,28	0,35	0,012	0,007	0,11	0,048	0,042	0,0018
K	0,38	1,38	0,33	0,014	0,011	0,12	0,045	0,040	0,0017
L	0,36	1,30	0,35	0,026	0,021	0,11	0,042	0,038	0,0020

The investigated steel (C 105) has a Re_{min} of 724 MPa. Apart from the specified strength and plastic properties, this steel is also required to withstand 720 hours of exposure in

Table 2. Average values of the Re, Rm, A and KCV

Heat	Re MPa	Rm	A(2") %	KCV/8(+20°C)	KCV/8(-40°C) Jcm^{-2}
A	739	834	23,9	111,1	47,4
B	732	801	24,5	109,8	50,2
C	791	865	23,1	122,2	48,2
D	776	857	22,7	94,6	49,4
E	730	829	23,0	142,2	64,7
F	783	858	23,7	173,0	66,3
G	762	836	23,7	181,3	75,8
H	752	838	23,7	115,6	56,2
I	761	852	24,2	114,2	51,4
J	741	848	23,8	114,5	54,2
K	749	854	23,2	119,5	52,5
L	746	836	23,8	124,8	57,6

the presence of H_2S in NACE solution (TM-01-77 with a pH value of 3,5) under a load equal to 80% of the Re_{min} (in this case a load of 579 MPa) in an SSC susceptibility test.

After water quenching from 840°C and tempering at 615 to 630°C the mean room temperature Re values of the examined heats ranged from 730 to 791 MPa. The test specimens has a microstructure of tempered martensite and/or of mixture of martensite and of low bainite with uniform distribution of carbide particles. The received mechanical properties of the individual heats are summarized in Table 2. The SSC resistance of the examined heats were judged by their endurance in the 720 hour exposure tests in NACE solution at loads of 500, 550 and 600 MPa (as a minimum three specimens at each load).

Figure 1 summarizes the findings of the SSC susceptibility tests characterized by the fraction of minimal Re of the

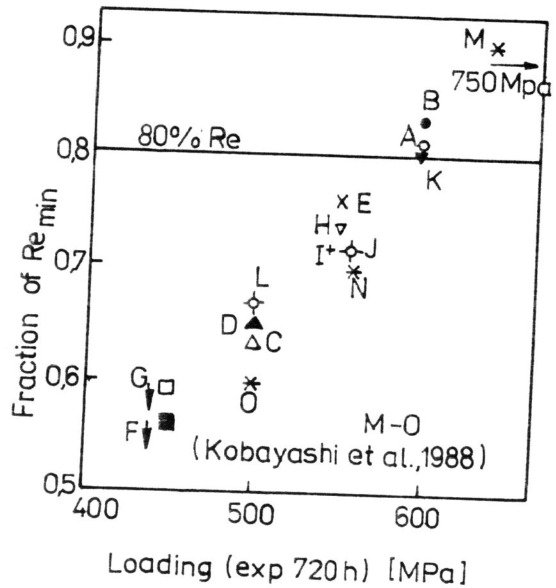

Fig. 1. SSC susceptibility in dependence on exposure of 720 hours

investigated heats (Table 2) in NACE solution in dependence on the loading exposure of 720 hours. The arrows indicate some specimens cracked in shorter exposure than 720 hours. The dependences of the applied loading by the SSC susceptibility test on the sulphur and the phosphorus contents of the examined heats for exposure of the 720 hours are presented in Fig. 2 and 3. A point to note is that the tests revealed no systematic decline in the susceptibility to hydrogen embrittlement in SSC test as the sulphur contents of the examined heats diminished.

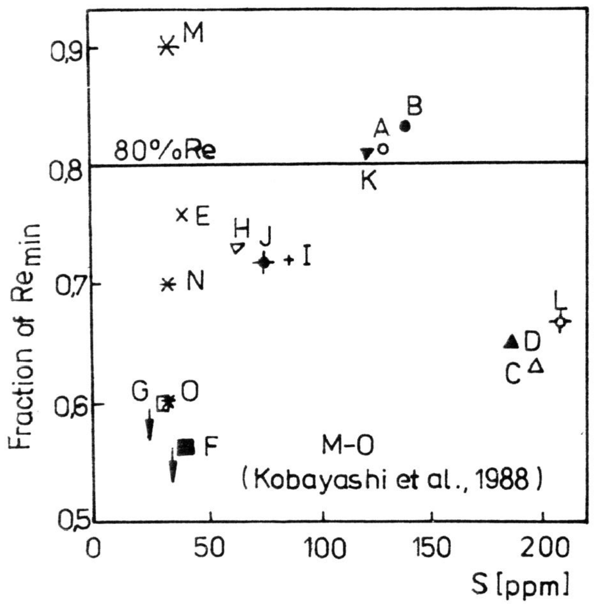

Fig. 2. SSC susceptibility in dependence on the sulphur content

Microfractographic analysis confirmed that the cracks were usually initiated at sulphide type inclusions, where signs of cleavage were detected in their vicinity (Mazancová, 1990). The crack may also be initiated at oxides, e.g. alumina particles in the minority of examples. In compared heats the content of these inclusions, in view of the applied steelmaking technique, was very similar. Figures 1 to 3 prove that the worst results of the SSC resistance were recordered in heats microalloyed with vanadium alone, despite the very low sulphur contents of these heats (Table 1). For this reason, the SSC resistance of these heats was in addition evaluated at lower load than 450 MPa.

DISCUSSION

The relationship between sulphur contents and static fatigue

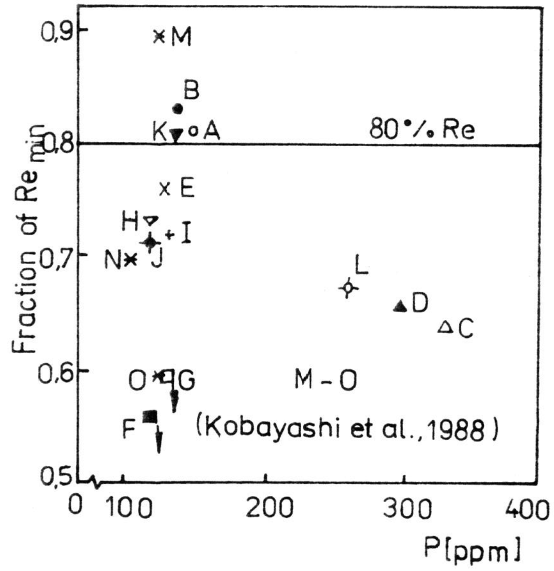

Fig. 3. SSC susceptibility in dependence on the phosphorus content

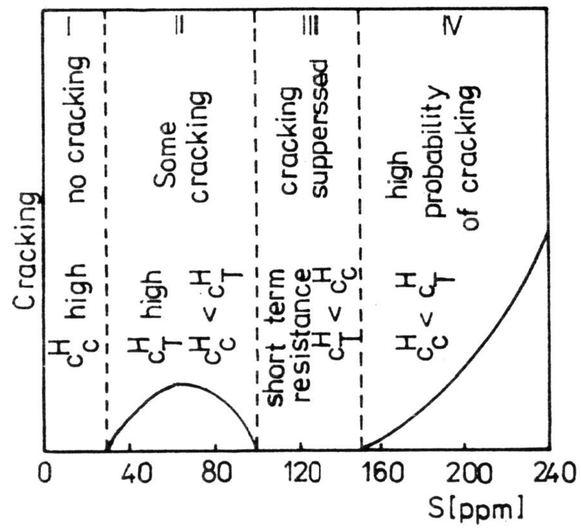

Fig. 4. Frequency of cracking in SSC tests in dependence on the sulphur content

in sour environment (in terms of life in SSC tests) is illustrated in Fig. 4. Under these conditions, the hydrogen embrittlement represents a very important parameter of the degradation processes accompanying the sulphur stress corrosion. The behaviour observed in the examined heats is evidently related to their sulphur or sulphide contents, and to some extend to the presence of other hydrogen traps which contribute to a more uniform distribution of hydrogen atoms. We may view the hydrogen embrittlement as the outcome of two mutually competing factors: the hydrogen concentration c_T^H in the traps and the level of the local critical concentration c_c^H at which the stress induced at the crack tip under the superimposed action of hydrogen σ^H exceeds the cohesive stress σ_c^H. Besides the primary trapping effect of the sulphide inclusions the effectiveness of potential hydrogen traps depends on uniform distribution of carbides or carbonitrides of microalloying elements (Ti, Nb and V). These hydrogen traps contribute to a more uniform distribution of hydrogen atoms, thus in effect preventing any exceeding of the local critical concentration of hydrogen atoms.

The dependence presented in Fig. 4 can be divided into 4 regions with the different SSC susceptibility. The most favourable results were obtained with heats A, B and K, in which 720 hours of SSC testing at 600 MPa or at 0,83 of the Re_{min} value produced no failure. Naturally, we cannot rule out that longer exposure would have resulted in crack initiation, especially in heats with sulphur content that falls into the region marked III in the dependence in Fig. 4. Heat E is worth special mention. In spite of its microalloying (Ti, Nb and V microalloying additions) and the low sulphur content of a mere 40 ppm, its resistance was disappointing. Only under the lesser load of 550 MPa did specimens of this heat withstand 720 hours of testing without failure. This places heat E in the lower boundary of the region denoted II in Fig. 4. The same results were found with heats H, I and J, which with their sulphur contents of 60 - 80 ppm can be placed in the region II.

The results of the SSC resistance received with heats C, D and L conformed to expectations, in view of the higher phosphorus and sulphur contents when, as it is evident in Table 1, all the other examined heats had virtually identical phosphorus contents (0,011 - 0,015%P). The least favourable results were recorded with heats F and G, which survived very short exposure to the NACE solution only. At the loading of 450 MPa (approx. 0,60 Re_{min}) only one (F) and/or two (G) specimens (tested three specimens) recorded the time to failure 720 hours. Their sulphur contents place these heats in region II of the Fig. 4. We must bear in mind that the

absence of carbides or carbonitrides of titanium and niobium in these heats reduced the number of potential hydrogen traps in them as these heats were microalloyed with vanadium alone. Vanadium carbides or carbonitrides form much weaker hydrogen traps than titanium - based or niobium - based precipitates (Pressouyre, 1980). We cannot rule out the possible influence of boron addition. In heats A to E and H to L, boron could to some extent have helped to strengthen the interphase boundary and particularly the grain boundaries (Mazanec, 1988).

The results of this work corroborate findings published in Kobayashi et al. (1988), where satisfactory performance were attained with heats having low sulphur contents of 30 to 50 ppm, and microalloyed with addition of titanium, niobium and vanadium. The phosphorus contents were not higher than 70 ppm, which are about half of the phosphorus content in our examined heats (Table 1). The results of the SSC resistance recorded with these heats are simultaneously presented (heats M, N O) in Figs. 1 - 3.

Furthermore, the results of our investigation apply to strictly limited periods of time only. For instance, longer exposure of heat A, B and K specimens would most probably have raised the local concentrations of hydrogen atoms to exceed the critical threshold value and this causing failure.

CONCLUSIONS

The investigation of the static fatigue resistance conducted under the extreme testing conditions in NACE solution (the SSC resistance) has indicated that failure analysis and service life prediction must always take into account a whole set of microstructural and metallurgical effects.

The best results can be recorded with heats their sulphur content is situated in region I and III (Fig. 4) and microalloyed with titanium, niobium and vanadium.

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