

POLISH ACADEMY OF SCIENCES - COMMITTEE OF MATERIALS SCIENCE SILESIAN UNIVERSITY OF TECHNOLOGY OF GLIWICE INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS ASSOCIATION OF ALUMNI OF SILESIAN UNIVERSITY OF TECHNOLOGY

Conference Proceedings

ACHIEVEMENTS IN MECHANICAL & MATERIALS ENGINEERING

Role of non-metallic inclusion shape in hydrogen embrittlement tested using slow strain rate test\*

J. Sojka<sup>a</sup>, P. Beťáková<sup>a</sup>, L. Hyspecká<sup>a</sup>, L. Čížek<sup>a</sup>, M. Sozańska<sup>b</sup>, A. Hernas<sup>b</sup>

<sup>a</sup> VŠB – Technical University of Ostrava, tř. 17. listopadu, Ostrava-Poruba, Czech Republic;

<sup>b</sup> Politechnika Śląska, ul. Krasińskiego 8, Katowice, Poland

# 1. INTRODUCTION

Hydrogen embrittlement of steels tested using slow strain rate test manifests itself especially by a drop of elongation and reduction in area. On fracture surfaces, so called "fish eyes" can be found, oriented perpendicularly to the applied tensile stress. Fish eyes initiate nearly always on non-metallic inclusions and propagate by cleavage or quasicleavage fracture. Shape and orientation of inclusions can influence initiation and shape of fish eyes. An inclusion can act as a fish eye initiator only if the plasticity of the matrix, i. e. its capacity to support three axial stresses, is decreased. Qualitative and quantitative analysis of non-metallic inclusion geometric parameters was performed on metallographic sections and fracture surfaces for three orientations (longitudinal, transversal, through-thickness) of tensile specimens taken from ASTM A516 steel plate. Results showed that in longitudinal and transversal direction globular oxisulphides acted as fish eye initiators while in through-thickness direction fish eyes initiated only on ellipsoid manganese sulphides. Role of tensile stress (specimen) orientation is discussed in relation to the orientation and shape of inclusions.

## 2. EXPERIMENTAL PROCEDURES

Hydrogen embrittlement was tested on ASTM A516 steel plate with thickness of 80 mm using slow strain rate tensile tests. Tensile specimens with 4 mm in diameter were taken in three orientations – longitudinal(L), transversal(T) and through-thickness(TT) - with regard to the rolling direction. Steel was tested after normalisation (890°C/1 hour/air). Its microstructure consisted of banded ferrite and pearlite. Chemical composition is given in table.

<sup>\*</sup> The work was sponsored by Project KONTAKT and CEEPUS CZ-13 and PL-13.

С	Mn	Si	Р	S	Cu	Ni	Cr	Мо
0.24	1.07	0.26	0.010	0.003	0.12	0.42	0.29	0.08

Table 1. Chemical composition of A516 steel (% by mass)

A part of tensile specimens was electrolytically charged in 2.8% water solution of HCl with addition of KSCN (1g/1l) during 24 hours at a current density of 1 mA.cm<sup>-2</sup>. After this charging specimens contained about 1.5 ppm of hydrogen while hydrogen content in initial state was about 0.5 ppm [1]. Charged as well as non-charged tensile specimens were tested at room temperature with strain rate less than  $10^{-4}$  s<sup>-1</sup>. Mechanical properties of both charged and non-charged specimens are summarised in table 2. A hydrogen embrittlement index F is also shown calculated as a relative drop of reduction in area after hydrogen charging [2]. A higher F corresponds to a higher degree of hydrogen embrittlement [3].

Table 2. Mechanical properties of non-charged and charged specimens from A516 steel

Direction	i (mA·cm <sup>-2</sup> )	R <sub>e</sub> (MPa)	R <sub>m</sub> (MPa)	A (%)	Z (%)	F (%)
L – longitudinal		330	543	30.0*	71.3	-
T – transversal	-	315	521	22.7*	73.2	-
TT – through-thickness		328	533	30.9**	66.9	-
L – longitudinal		352	475	21.8*	39.9	44.0
T – transversal	1.0	342	533	16.6*	44.7	38.9
TT – through-thickness	1.0	350	546	14.1**	22.1	67.0

\*  $L_0 = 26 \text{ mm}; ** L_0 = 18 \text{ mm}.$ 

A qualitative analysis of non-metallic inclusions acting as fish eye initiators was performed on Philips XL 30 scanning electron microscope coupled with EDAX micro analyser. Microanalysis revealed that in longitudinal and transversal directions fish eyes initiated on globular oxides and oxisulphides containing to a various extent especially Al, Ca, Mn and Mg (Fig. 1) or on non-deformable globular (Ca,Mn) sulphides. In through-thickness direction fish eyes initiated exclusively on ellipsoid manganese sulphides (Fig. 2).



Fig. 1. Fish eye, oxide inclusion. Transversal direction



Fig. 2. Fish eye, MnS inclusion. Through-thickness direction

#### 3. DISCUSSION

The distinctive fish eye initiators for different specimen orientations can be explained using 2D model based on the stress localisation in the vicinity of inclusions during a tensile test. Stress concentration can be expressed using the following coefficient  $\beta$  [4]:

$$\boldsymbol{\beta} = \frac{\sigma_{max}}{\sigma_{nom}} = 1 + 2\sqrt{\frac{e}{r}}$$
(1)

where

 $\sigma_{max}$  – maximal stress provoked by the stress concentration (MPa)  $\sigma_{nom}$  – nominal stress (MPa) e – half size of a defect (µm) r – sharpness radius (µm).

It is obvious that stress concentration will depend on the specimen orientation and on the inclusion shape. To be able to determine the stress concentration coefficient  $\beta$  a quantitative analysis of non-metallic inclusion geometric parameters was performed on 3 metallographic sections. This analysis was performed separately for globular oxides and ellipsoid manganese sulphides. A schema is depicted in Fig. 3 for a manganese sulphide and corresponding results are given in table 3 for both oxides and sulphides.

Table 3 Average sizes of non-metallic inclusions determined on metallographic sections a, b, c represent half-axes of three axial ellipsoid

Inclusion	2a	<i>2b</i>	2c	R	
Inclusion	(µm)	(µm)	(µm)	(µm)	
oxide	-	-	-	$1.92 \pm 0.07$	
MnS	$9.8 \pm 1.7$	$6.9 \pm 0.7$	$2.7 \pm 0.2$	-	



Fig. 3. Orientation and shape of manganese sulphide on 3 metallographic sections

Knowing the geometric parameters for two most important types of non-metallic inclusions stress concentration coefficient  $\beta$  could be calculated. Results are given in table 4 for manganese sulphides. It is evident that for globular oxides  $\beta$  is equal to 3 regardless the orientation (see eq. 1, e = r = R).

Orientation of	e	r	β
X(L)	b = 3.45 μm	$a^{2}/b = 6.96 \mu m$	2.4
Y(T)	$a = 4.90 \ \mu m$	$b^2/a = 2.43 \ \mu m$	3.8
Z (TC)	$a = 4.90 \ \mu m$	$c^2/a = 0.37 \mu m$	8.3

Table 4. Stress concentration coefficient  $\beta$  for manganese sulphides and different specimen ( $\sigma_{nom}$ ) orientations

The comparison of stress concentration coefficient  $\beta$  for globular oxides and manganese sulphides can be used in the following way: In the longitudinal orientation of tensile specimens manganese sulphides are less dangerous ( $\beta = 2.4$ ) and that is why fish eyes initiate on large globular oxides. In the transversal orientation one can expect the predominant fish eye initiation on manganese sulphides ( $\beta = 3.8$ ). Nevertheless, also in this case fish eyes initiated predominantly on globular oxides. Nevertheless, there is one more parameter that has to be taken into account and it is a maximal inclusion size. An analysis performed on fracture surfaces revealed that globular oxides represented the largest inclusions in transversal orientation. Manganese sulphides become extremely noxious in through-thickness orientation due to a very high stress concentration ( $\beta = 8.3$ ) [5]. That is why in this orientation fish eyes initiate exclusively on them.

#### 4. CONCLUSION

2D model based on the local stress concentration around non-metallic inclusions was used to explain fish eye initiation during tensile tests on hydrogen charged specimens from A516 steel. It was found out that different initiation sites – globular oxides for longitudinal and transversal orientation and ellipsoid manganese sulphides for through-thickness orientation – reflect different stress concentration as a function of specimen orientation and inclusion shape.

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