Benchmark on the determination of the cleavage triggering sites in a RPV steel in the DBT range

Charlotte Bouchet, Félix Arnoldi, Jacques Besson, Suzanne Degallaix, Valérie Denner, Yannick Desplanques, Olivier Diard, Gilles Espinasse, Pierre Forget, Petr Hausild, et al.

BENCHMARK ON THE DETERMINATION OF THE CLEAVAGE TRIGGERING SITES IN A RPV STEEL IN THE DBT RANGE

C. BOUCHET[1], F. ARNOLDI[2], J. BESSON[1], S. DEGALLAIX[3], V. DENNER[4], Y. DESPLANQUES[3], O. DIARD[2], G. ESPINASSE[2], P. FORGET[5], P. HAUSILD[7], E. MAIRE[6], I. NEDBAL[7], V. RABEAU[5], B. TANGUY[1], C. VERDU[6],

[1] École des Mines de Paris, Centre des Matériaux, UMR CNRS 7633, BP 87, 91003 Evry Cedex, France.

[2] EDF R&D Dpt MMC, Avenue des Renardières, 77 818 Moret sur Loing Cedex, France.

[3] École Centrale Lille, BP 48, 59651 Villeneuve d'Ascq Cedex, France.

[4] Fraunhofer Institut für Werkstoffmechanik, Wöhlerstrasse 11, D–79108 Freiburg, Germany.

[5] CEA Saclay, DMN/SRMA, 91191 Gif sur Yvette Cedex, France.

[6] GEMPPM, INSA, 20 avenue Albert Einstein, 69 621 Villeurbanne Cedex, France.

[7] Dpt of Materials, Faculty of Nuclear Sciences and Physical Engineering, CTU, Trojanova 13, 120 00 Praha 2, Czech Republic.

ABSTRACT

This paper presents the work undertaken as a benchmark on fracture micromechanisms. A systematic investigation of the nature and position of cleavage initiating sites has been carried out on selected specimens of an ASTM A508 Cl.3 steel by seven laboratories. Observations were carried out on three different kinds of specimens, i.e. Charpy V-Notch, Compact Tension and Notched Tensile specimens, at different testing temperatures in the ductile-to-brittle (DBT) range. Participants were asked to follow a proposed methodology to determine both the main cleavage triggering site and its location with respect to the given reference frame. A correct agreement was found between participants for the determination of the cleavage initiating site as far as CVN and CT specimens were concerned. For NT specimens, the presence of a ductile damage zone with debonded inclusions only enabled participants to agree on the mesoscopic scale.

1 INTRODUCTION

The ductile-to-brittle transition in steels remains one important issue of materials science and has been subjected to extensive research. The integrity of key safety components made of steel such as reactor pressure vessels depends on the material resistance against brittle fracture. Cleavage which is a sequential stochastic process of crack nucleation and propagation, is usually induced by particles such as carbide (TiC, Fe_3C) or inclusions (MnS).

The determination of the triggering site, its type, location and of the controlling mechanism is an important step in the construction of embrittlement models (Wang [1]) (Bowen [2]). It is clear that the site scale is quite small (few μm) while the specimen scale is large, therefore the determination of the triggering site is not obvious. Morever, in the transition range, the increasing portion of ductile damage makes the determination of the cleavage initiation site harder. However the position of triggering sites is an important step to determine the local cleavage stress by Finite Element modeling (see e.g. (Rossoll [3])). This type of approach can be used to determine the possible temperature dependence of the cleavage stress. However, there is still little exchange of the experience with the identification of these cleavage triggering sites. This benchmark intends to be a platform for exchanging experiences, discussing difficulties between different laboratories and to define a reliable procedure for the site identification.

2 MATERIAL AND SPECIMEN DESCRIPTION

2.1 Material

Data from a nuclear pressure vessel steel similar to an ASTM A508 Cl.3 grade was used in this benchmark. The heat treatment of the cylinder involved an austenization at about 880°C and a water quench, a tempering heat treatment for 5.75 h at about 650°C and air cooling. The ambient tensile mechanical properties are: yield stress: 472 MPa, UTS: 600 MPa, elongation: 25%, reduction of area: 73%. This steel is characterized by a tempered bainitic microstucture with a prior austenite grain size of about 23 μm (Tanguy [4]). In such steels, inclusions play an important role as they are potential initiation fracture sites for ductile and brittle fracture. MnS inclusions observed can be divided in two populations: spherical (smaller ones) or slightly elongated ones along the rolling (L) direction.

2.2 Specimen geometries

Four types of specimens were studied: (i) Notched Tensile (NT) specimens with two notch radii (1.2 and 2.4 mm) and a mininum diameter, Φ_0, of 6 mm, (ii) Standard Charpy V–Notch (CVN) specimens, (iii) CT(1.2T) specimens (with a 30 mm thickness). For CT(1.2T) specimens, a nominal crack length to specimen width ratio of ~ 0.6 was used. The Charpy specimens orientation was T-S (crack plane perpendicular to the long transverse orientation and crack growth direction parallel to the short transverse orientation (S))and the CT(1.2T) specimens orientation was T-L (crack plane perpendicular to the long transverse orientation and crack growth direction parallel to the longitudinal orientation (L)). It should be noted that the CT specimens were tested without side–grooves.

2.3 Specimen testing

All tests were performed under displacement control conditions. For all the tests, two specimens were tested for a given test temperature. Except for CT specimens, the test temperatures were chosen in the brittle domain close to the DBT range and within the transition domain. CT specimens were only tested at one temperature. Temperatures, ram displacement rate, orientation are reported in Tab. 1 for each specimen geometry. The specimens were cleaned with alcohol and warmed up immediately after the tests to avoid corrosion of the fracture surfaces. No coating was used to protect the fracture surfaces while they were transferred from one laboratory to another. Silica gel was used to prevent corrosion of fracture surfaces. In the different laboratories, the specimens were carefully kept in alcohol or in a vacuum system. In spite of all, the fracture surfaces contamination was not avoided.

3 RESULTS AND DISCUSSION

3.1 Participants

Seven contributions have been received to this benchmark. Tab. 2 gives the name of the different organisations, country and SEM devices used for the observations. For a more comprehensive report

geo.	T (°C)	specimen label	orientation	ram disp. rate (mm/s)	CVN (J) / fracture toughness (MPa$\sqrt{m}$)
CVN	-40	36	T–S	5230	65
	-40	73	T–S	5230	84
	-100	37	T–S	5230	13
	-100	70	T–S	5230	14.5
$NT_{2.4}$	-100	8; 9	T	0.0024	-
	-150	1; 2	T	0.0024	-
$NT_{1.2}$	-70	7; 9	T	0.0012	-
	-100	1; 2	T	0.0012	-
CT(1.2T)	-30	H3	T–L	0.025	K_{Jc}=290
	-30	305	T–L	0.025	K_{Ju}=456

Table 1: Matrix of test data.

and in order to preserve the contributors, each laboratory was denominated by a letter so that the results can be described anonymously.

Organisation and country	SEM apparatus
Centre des Matériaux, EMP, France	LEO 14500 VP and Zeiss DSM 982 Gemini
Commissariat à l'Energie Atomique, France	LEO S260
École Centrale Lille, ECL, France	Hitachi S2500
Électricité de France, EDF, France	Hitachi S2500 and FEG Supra 35
Fraunhofer Institut für Werkstoffmechanik, IWM, Germany	CAMSCAN 24
GEMPPM INSA, France	JEOL 840
Czech Technical University, CTU, Czech Republic	JEOL JSM 840

Table 2: Benchmark participants and SEM apparatus used.

3.2 Determination of triggering sites

Each laboratory was free to choose the appropriate observation conditions (working distance, voltage…). Most of the micrographs were recorded at normal beam incidence, even if some specimens were slightly tilted to the normal of the fracture surface due to the high relief of their fracture surfaces and in order to obtain an ensured determination of the triggering site. Moreover, the same specimen orientation was used by all the participants in order to obtain easily comparable photographs.

Triggering sites were identified by following a network of radiating major tear and river lines. It should be emphasised that such a fractographic analysis is difficult, because in many cases it is not sufficient to observe the fracture surface of only one half-specimen, and examination of matching surfaces (corresponding mirror pattern on the other fracture surface) is necessary. The procedure is illustrated on Figure 1.

Good agreement between the participants was obtained for both Charpy and CT geometries. At -40°C for CVN, where significant ductile propagation is observed ($\Delta a \sim$ 1 mm), a unique site could undoubtly be identified. At -30°C for CT, where final cleavage fracture was preceded by ductile crack growth, and at -100°C for CVN specimens, multiple initiation sites were found and in some cases differs for each participants. However by carefully following the river lines a unique initiation could be found except for one CVN specimen (#37). An example of micrographs which

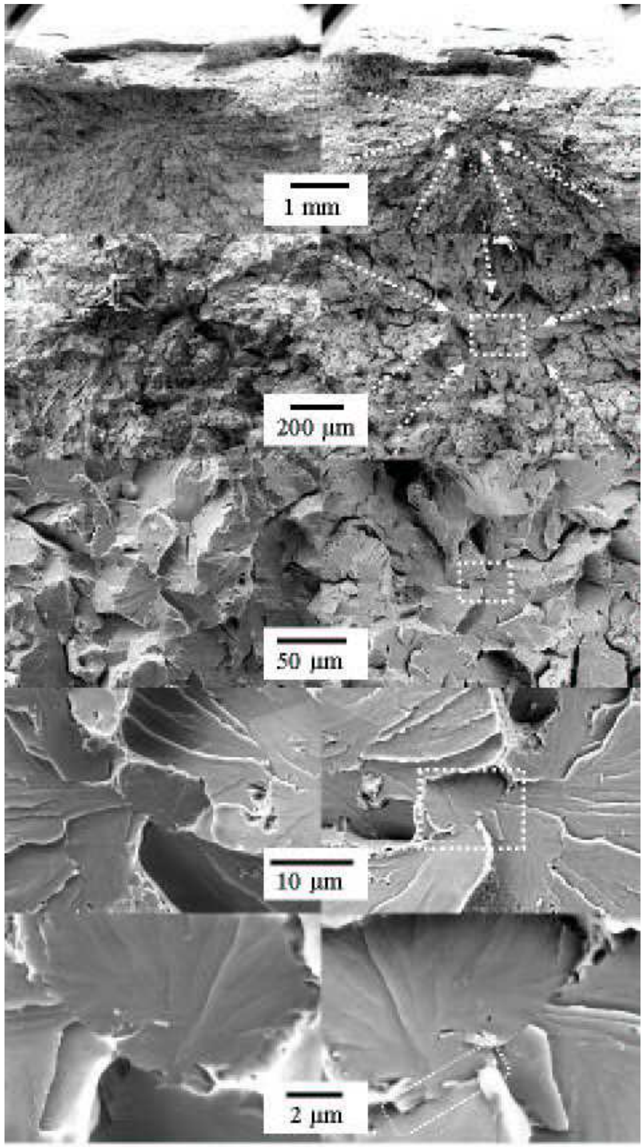

Figure 1: Identification of a triggering site (CVN #73 tested at $-40°C$). Example of the different steps used for the identification. On the matching surface photographs, dotted arrows were drawn to underline the macroscopic river patterns. Afterwards, boxes with dashed line were used to indicate the interest zone. On the last photograph, the triggering site, which is a grain boundary, is indicated by a dashed rectangle.

were supposed to be accurate enough to identify the initiation site for CVN #70 ($-100°C$) is given in Fig. 2. This figure shows that even if the initiation area is located, it may be not sufficient to clearly identify the event at the origin of the cleavage fracture (see Fig. 2, micrograph a). It underlines that a very high magnification, i.e. a SEM device with appropriate resolution, is needed. On this figures,

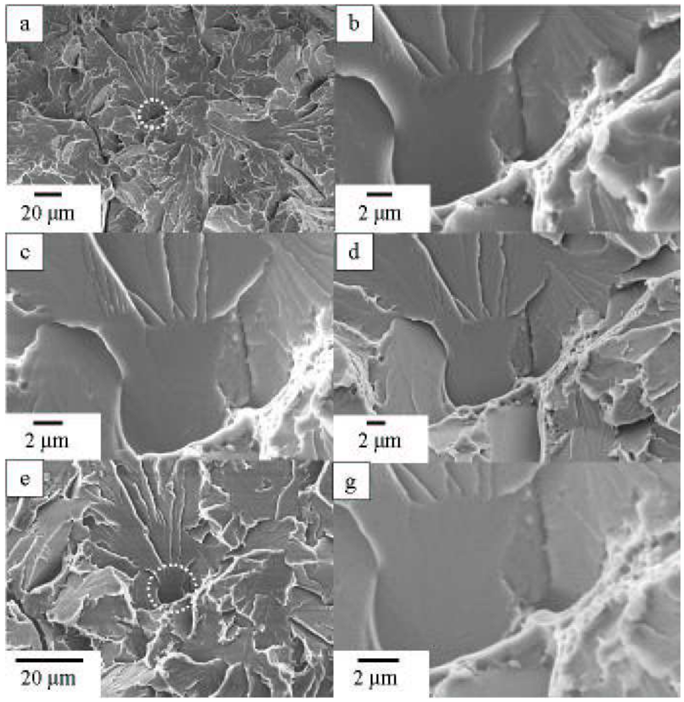

Figure 2: Clivage triggering site for CVN #70 tested at $-100°C$. Micrographs obtained by participants a, b, c, d, e and g.

the similarity between the micrographs obtained by the participants is remarkable.

For NT specimens, the macroscopic river patterns always lead to a zone of ductile damage with debonded inclusions, probably MnS, which makes difficult to follow the microscopic rivers towards the initiation site. For all specimens, it appears that several initiation sites have been identified more or less close around the same cluster of debonded inclusions. However an agreement was not found to identify clearly one identical cleavage triggering site. As shown on Fig. 3, the same ductile damage zone at the origin of cleavage was found but different main triggering sites were determined.

3.3 Position of triggering sites

For CVN and CT specimens, as far as the distance from the left side of the specimen, X, is concerned, a general agreement was obtained when the same matching surface was used. However a difference of 50–60 μm is generally observed. This may be explained by the measurement system precision and also in the way each participant has defined the position of the side of the specimen. Concerning the distance between the initiation site and the ductile crack front (Y), more discrepancy was obtained. Differences as large as 500 μm have been reported. These discrepancies are still under investigation. A possible explanation is the way each partipant has defined the frontier between ductile crack front and cleavage area.

4 CONCLUSIONS

Fractographic examinations were carried out by seven organisations to investigate the damage

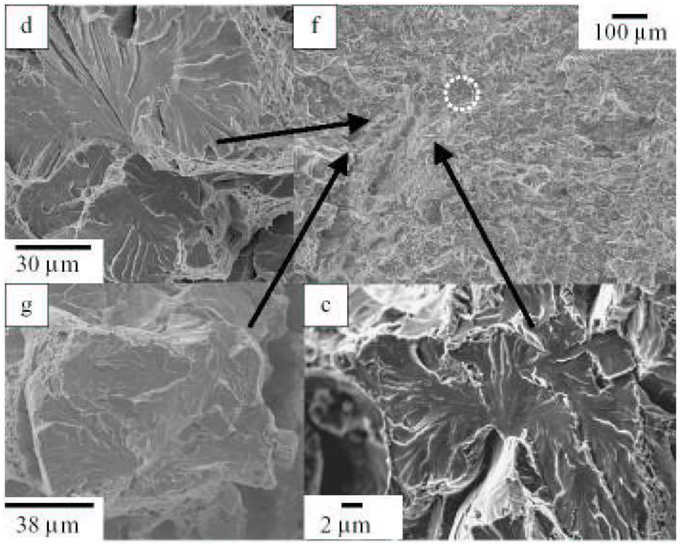

Figure 3: $NT_{1.2}$ #7 tested at $-70°C$. Arrows locate the final micrographs taken by participants around the inclusion stringer. The circled zone drawn shows one potential triggering main site.

processes involved in the DBT range on three different geometry specimens. The nature and location of the cleavage triggering sites were studied for all the specimens tested in this study. Conclusions drawn from this study are given as follows :

(i) A coating is needed in order to protect the surface fracture of the specimens during their transfer between laboratories as a systematic gel pollution of fracture surface was observed;

(ii) The applied benchmark methodology was followed by all participants. Very high magnifications are often needed to determine the cleavage initiation site;

(iii) On the whole study, the determination of the main triggering site position (X and Y), as far as the precise coordinates with respect to a given reference frame are concerned, is rather unsatisfactory.

(iv) For the CVN and the CT specimens, a good agreement was found between participants concerning the determination of the main triggering site. The similarity between some of the micrographs is remarkable especially for CVN specimens;

(v) For the NT specimens, a good mesoscopic agreement was found on the zone where cleavage initiated. The location of the site around the ductile damage zone with debonded inclusions has to be further investigated;

Aknowlegements

Fractographic analysis undertaken in CTU was partly financed by Grant Agency of Czech Republic (project GAČR 106/04/0066).

REFERENCES

1 Wang, G. and Chen, J. H. (2001) *Fatigue Fract Engng Mater Struct*, **24**, 451–459.
2 Bowen, P., Druce, S. and Knott, J. (2001) *Acta metall.*, **35**, 1735–1746.
3 Rossoll, A., Berdin, C. and Prioul, C. (2002) *Int J Frac*, **115**, 205–226.
4 Tanguy, B., Besson, J., Piques, R. and Pineau, A. (2004) *Engng Fract Mech, in press*.