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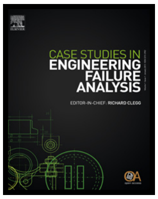

Case study

Internal reversible hydrogen embrittlement leads to engineering failure of cold drawn wire

Arup Mallick *, Souvik Das, Jitendra Mathur, Tanmay Bhattacharyya, Arthita Dey

R&D and Scientific Services, Tata Steel Limited, Jamshedpur 831001, India

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1. Introduction

Hydrogen embrittlement is very common type of phenomenon that causes industrial failure of steel material. It has been observed that embrittlement may also take place due to interfacial segregation of hydrogen in high strength steel, which leads to delamination or decohesion. Cold drawn high carbon steel wires are amenable to being weaker along the longitudinal planes compared to the transverse planes due to fibre-like deformation of pearlitic phases in the longitudinal direction. Hydrogen embrittlement is prone to cause spontaneous splitting in cold drawn wire. Generally, atomic hydrogen is absorbed initially by the metal surface and transforms to molecular hydrogen when the concentration reaches higher value. The molecular hydrogen accumulates in voids, pores and interfaces among many other defect sites. If these defects are not present in the vicinity of the high hydrogen areas, blisters or hair line cracks are formed to release the high pressure. Experimentally it was found that the segregation of molecular hydrogen generates 10^5 times atmospheric pressure at the interstitial sites [4]. During electroplating or pickling, hydrogen can enter the lattice, but it is usually diffused-out by a subsequent patenting [1] operation (heat treatment).

There is considerable literature, which discusses hydrogen embrittlement and proposed different models to describe the phenomenon. One of the models, the planar pressure mechanism, predicts that the high pressure developed due to molecular hydrogen within the gas pores inside the material causes cracking.

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* Corresponding author at: Metallurgical Laboratories and QA Group, R&D and Scientific Services, Tata Steel Ltd., Jamshedpur 831001, India. Tel.: +91 657 66 48906; fax: +91 657 234395.

E-mail addresses: arup.mallick@tatasteel.com, a.arup29@gmail.com (A. Mallick).

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The process of hydrogen cracking is the result of one or more of the micro-mechanisms such as: (a) cleavage, (b) intergranular decohesion and (c) microvoids coalescence. Microstructure of eutectoid patented steel is primarily pearlitic with a very few ferrite grains. Steels are having the same microstructure, when it is tested in hydrogen atmosphere, leads to surface cracking. In parallel, microvoids are generated in the centre of the specimen followed by residual ductile shear fracture of the remaining cross section. Fracture in air is characterized by very fine dimples, whereas, fracture in presence of hydrogen appear much rougher fracture surface [2]. Here we will discuss a particular case of failure of cold drawn wire due to the hydrogen embrittlement.

Internal reversible hydrogen embrittlement [3] can occur after a very small average concentration of hydrogen has been absorbed from the environment. However, the local concentration of hydrogen may be greater than average bulk values, because the absorbed hydrogen diffuses into the interface between grains or preferentially in to the grain boundary. The mechanical effects may leads to loss of ductility to cracking. Internal reversible hydrogen embrittlement has also been termed slow rate embrittlement and delayed failure. Heavy levels of cold working, especially in parts that are not subsequently stress relieved, can create residual stress levels that are very near the yield strength of the material. This is an ideal situation for hydrogen-assisted cracks to propagate.

2. Experimental details

Investigation was carried out on high strength 5 mm diameter cold drawn wire, which was broken during a drawing operation. Material was basically LRPC grade .Low Relaxation PC Stranded (LRPC) wires are used in Pre-stressed concrete girders for road, river and railway bridges and flyovers, pre-stressed atomic reactor domes, slabs, silos, hangars, aqueducts, high rise buildings, viaducts and railway sleepers [6]. Details of the broken wire samples are provided in Table 1. Broken wire samples that underwent breakage at the time of drawing operations had been processed from high carbon (0.82–0.84 wt.% C) wire rod. The samples investigated varied in diameter from 5.01 to 5.07 mm. Details of the wire rod and broken wire samples are provided in Table 1.The 5.00 mm diameter wires were pre-drawn from 10 mm diameter wire rod. Subsequently, the 5 mm wires were pickled and dipped in hot water for quenching and then drawn through a flux coating for better adhesion of coating materials to 2 mm diameter. This was followed by galvanizing.

Metallographic tests are carried out on:

(a) visual observation,
(b) microscopic examination of wire sample,
(c) fractography.

2.1. Sample preparation

Two pieces of wires that had broken during drawing were collected. The samples were cleaned with acetone to remove dirt and other adherent debris for visual examination prior to Fractography and metallographic sample preparation. Transverse and longitudinal specimens were prepared from the fractured end of each failed wire samples for conducting light microscopic examination. These samples were individually mounted in electrically conductive copper-containing resin and polished by conventional metallographic techniques. The failed surfaces were examined under upright metallurgical microscope as un-etched and etched conditions. Etching was done using 3% Nital with care.

2.2. Visual observation

Failed wire samples were observed visually under macro dual zoom microscope (see Fig. 1a). In the case of drawn wire samples, finger nail fractures were observed at the broken ends. The fresh fracture surfaces were very shiny, indicating the possibility of hydrogen embrittlement [1]. Deep surface tearing was also observed from the above figure in all the wire samples. Point 2, 3 and 4 are showing the transverse crack on the drawn wire piece.

Fig. 1b and c reveals the transverse cracks on the surface that may be due to the huge pressure build-up of hydrogen in interstitial sites. The present investigation was carried out to confirm embrittlement either due to metallurgical problems or due to the H concentration in material. Further, metallographic investigation was also done to characterize the failure.

Table 1
Details of wire rod and failed wire samples.

Spec	Sample type	Chemical composition (wt.%)							Section (mm)
		C	Mn	S	P	Si	Cr	N_2 ppm	
1	Wire	0.82	0.71	0.010	0.017	0.22	0.27	59	5.01
2		0.86	0.73	0.011	0.015	0.21	0.25	56	5.07
3		0.84	0.70	0.011	0.018	0.20	0.28	58	5.01

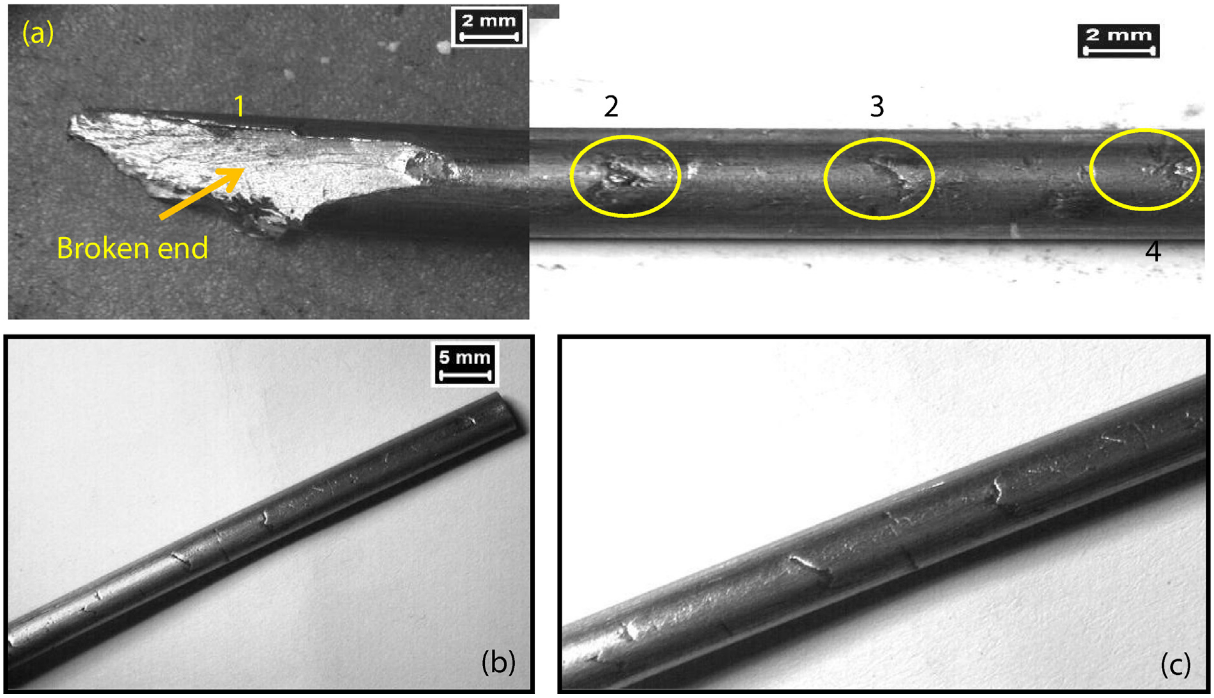

Fig. 1. (a) The broken wire samples; (b) and (c) the transverse crack on the wire samples at different magnifications.

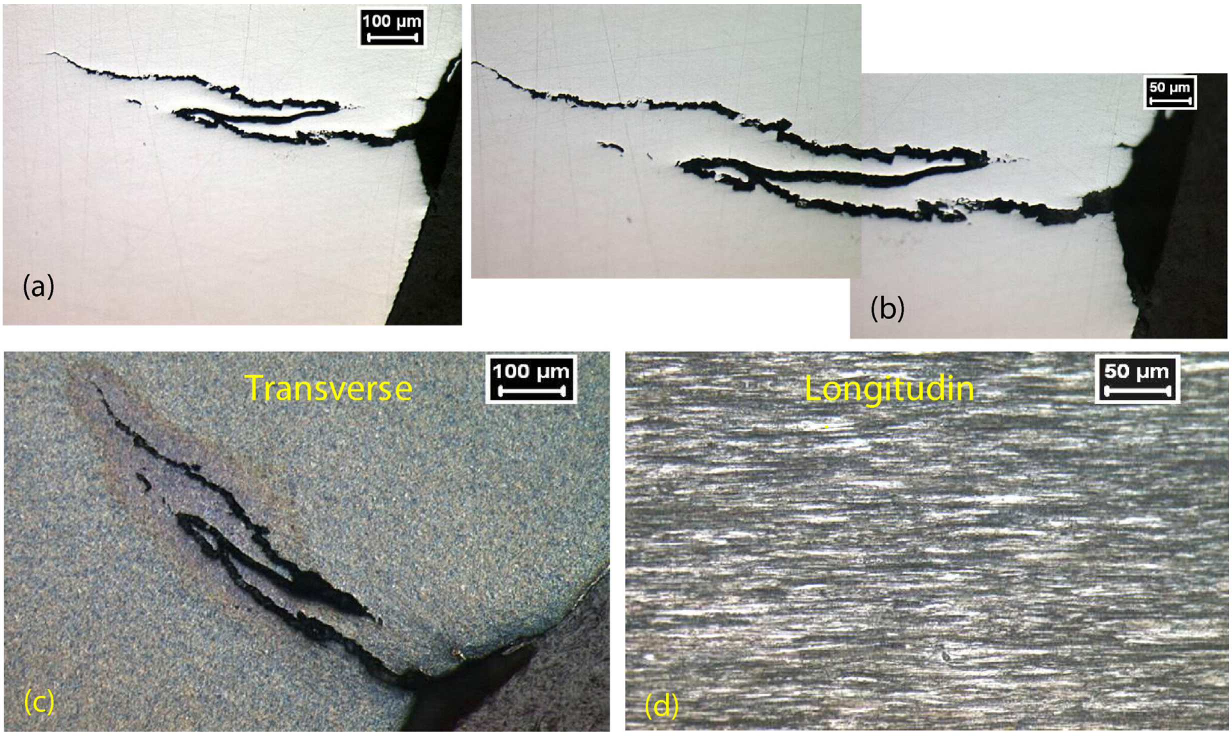

Fig. 2. (a) and (b) Zigzag crack at un-etched conditions in different magnifications, (c) etched structure in transverse section and (d) etched structure in longitudinal section.

3. Metallographic examination

Microscopic examination was conducted to observe microstructural features of defect area. Fig. 2a and b reveals zigzag cracks in transverse microstructure at un-etched condition. Typical image of un-etched microstructure was taken at 100× and 500× magnification. It may be noted that all cracks were almost perpendicular to the axis of the wire. The morphology of the crack propagation may indicate the absorption of hydrogen.

A cold deformed pearlitic microstructure was observed in Fig. 2c and d after etching with 3% Nital in both transverse and longitudinal samples. The microstructure did not indicate the presence of decarburization in the vicinity of cracks. It depicts

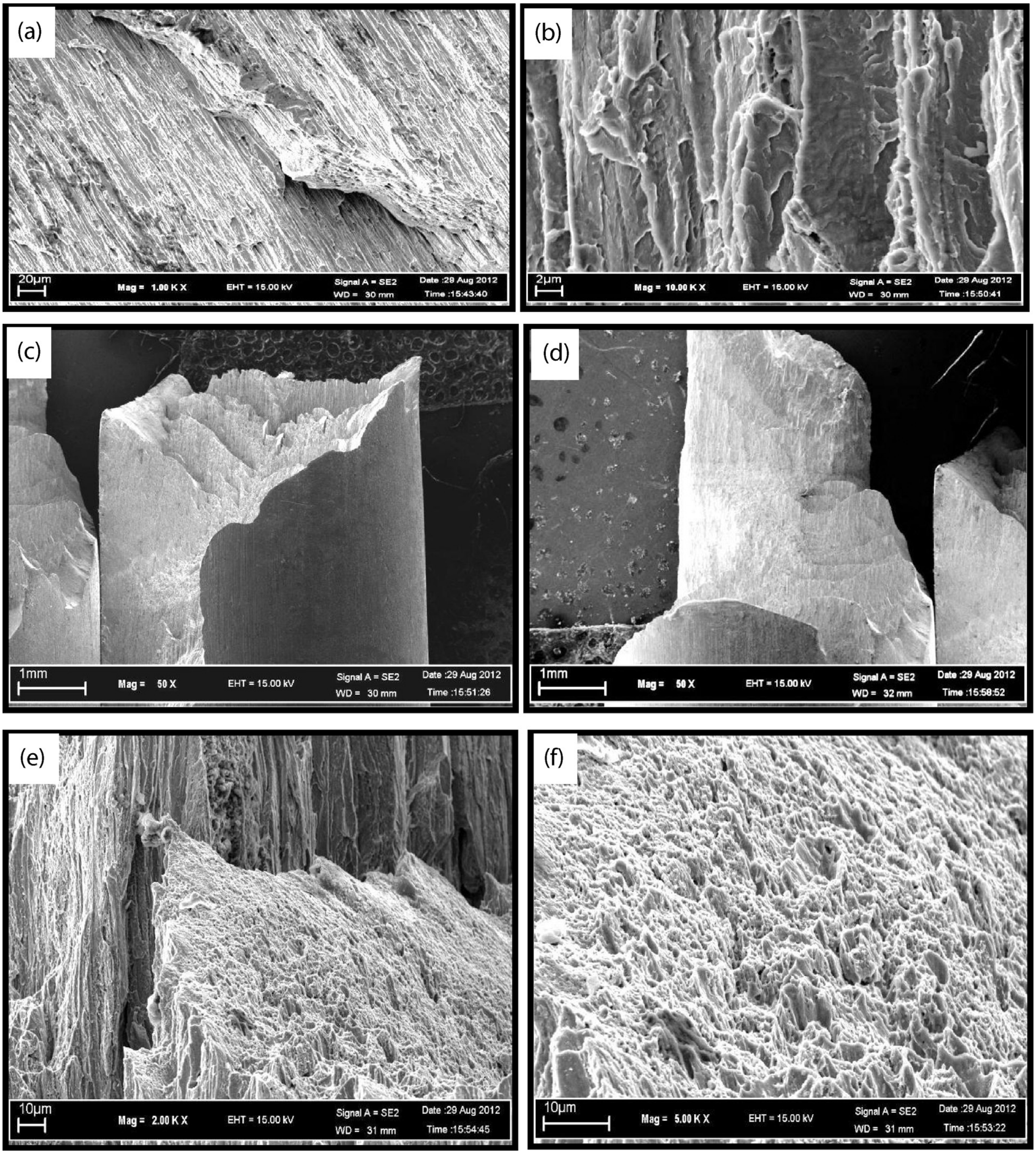

Fig. 3. (a) and (b) The fracture surface of the failed sample (as received conditions) at different magnifications, (c) and (d) the fracture surface of the failed sample (baked) after tensile testing and (e) and (f) the fracture surface of the failed sample (after tensile testing) at different magnifications.

that the crack was not generated at the stage of high temperature operation. Basically it is a phenomenon of intergranular cracking. A fibrous appearance comprising elongated shaped pearlite was formed during cold drawing (Fig. 2d).

4. Fractography

A comparative study was entitled on the fracture surfaces of as-received material with baked material (45 min). About 1.5 cm of longitudinal as-received sample with a fracture surface was taken for fractographic analysis under the SEM. This showed a fibrous appearance with secondary cracks (see Fig. 3a) at a magnification of 20 μm [5]. However, a detailed looked at higher magnification (see Fig. 3b) revealed brittle fracture with some dimple appearance, which clearly conforms the hydrogen attack of some form or other.

To diminish the effect of hydrogen, the sample was then baked at a temperature of 200 °C for 45 min and was then broken by tensile overload. Sample failed in a typical shape as seen in Fig. 3c and d. Both longitudinal and transverse fracture surfaces were visible in the sample.

The fracture surface revealed the fibrous ductile fracture morphology on the longitudinal cracked surface. Dimple appearances on the both transverse and longitudinal fracture surfaces were also seen (see Fig. 3e). A closer view of the transverse fracture surface (Fig. 3f) shows a dimpled appearance.

5. Hydrogen ppm tested by Leco

To confirm the quantity of hydrogen entrapment in interstitial lattice sites, as-received material was tested in a LECO (Model No. DH603). Six samples were drawn from the as-received long sample (25 mm) and the amount of hydrogen (in ppm) was measured and the results were averaged. The furnace temperature of LECO machine was 1000 °C and the liquid nitrogen flow rate was 3 l/min. The average result measured was 1.5 ppm this is considered to be a detrimental level and will leave the steel prone to hydrogen embrittlement.

6. Discussion

The microstructure of the cold drawn wire consisted of elongated pearlite in the longitudinal direction. The material has a tendency to absorb the hydrogen in an atomic form. Generally, hydrogen remains at the interfacial regions. During cold drawing high level of internal stress are generated [1]. Therefore, the evolution of hydrogen in the interfacial regions can lead to brittle hydrogen-induced cracking. In industrial practice hydrogen is absorbed in wire rod at the stage of pickling with of HCl. Acid reacts with iron and nascent hydrogen is released from the solution. This may be absorbed by the parent material. Only small amounts of hydrogen present at interstitial sites is required to cause cracking [3]. And as little as 1 ppm hydrogen results delayed cracking [3].

Fractography analysis is used here to help determine the root cause of this particular failure. As discussed above, that hydrogen was removed from the samples by baking. A comparative study of the fracture surface of as-received and baked sample demonstrates the difference of surface morphology [7]. The as-received fracture surface showed a fibrous structure which means a brittle in appearance. On the other hand, the baked sample revealed a dimples fracture surface. This demonstrated that the removal of hydrogen could reduce the susceptibility of the material to fracture.

LECO hydrogen analyser was used for further confirmation of hydrogen entrapment and a level of approximately 1.5 ppm hydrogen was found in the sample. This was considered sufficient to induce fracture.

7. Conclusion

On the basis of the above experiments, it was determined that the failure had occurred due to the hydrogen embrittlement. It is likely that the hydrogen came from the HCl solution at the stage of pickling operation and the subsequent baking was not carried out properly. That led to the retention of hydrogen atom in the interface and during the drawing operations failure resulted. Therefore, minimizing of the pickling operation time and proper baking should prevent this type of industrial failure.

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