Application of metallography in failure investigations – some case studies

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ABSTRACT

Metallography is a tool to know the internal condition of the material and no investigation can be initiated without this. It can be both nondestructive i.e., in-situ metallography, as well as destructive i.e., cutting the relevant parts from the site to undertake investigation in the laboratory. In both the cases grinding, polishing and etching to reveal the microstructures remain the same. The only difference is the special tools and portable optical microscopes are used in the in-situ techniques to obtain replicas for laboratory use. Two types of etching are used to reveal macro and microstructures. The macro structures are viewed at 5 to 10 x, which reveal flow line, cold rolling fibres, welding zones, pores, dendrites, coarse grains etc. whereas microstructures reveal the micro constituents, pearlite, martensite, inclusions, precipitates, carbides, creep voids etc. at magnification of 50 to 1500 x. Metallography alone gives lot of information regarding the deviations of microstructures which are indication of the causes of failures. Some typical case studies are described in the present paper where metallography plays very important role, like: (i) Improper weld deposits to salvage the undersize rail axle, which led to premature failure, (ii) improper welding joints and lack of post weld heat treatment, which gave stress on the joint of cover dome of pressure vessel tank, which caused failure, (iii) revealing of tool marking and sulphide inclusions in the steel isolation valve of LPG storage tank, which led to premature failure, (iv) sensitization and stress corrosion cracking in S.S. cover dome of centrifuge used in the manufacture of NG explosive, which caused premature failure, (v) Improper heat treatment and forging were indicated by the microstructures which led to the premature failure of mining dragline sleeve. These are only a few investigations carried out in the laboratory in the recent past which justify that without metallography it is not possible to diagnose the causes of premature failures.
INTRODUCTION

Five prematurely failed samples from different areas like Railway, Petroleum Industry, Explosive Industry and Mining fields were received at NML for investigation. Normally the failed samples are visually examined and all the features that are worth recording are noted. The sites showing any abnormality are cut with hacksaw for metallographic study. The sizes are variable. If welding parts are involved, then the size could be large for example in the case of rail axle the sample was 17 cm dia axle plate. This was essential as the whole axle transverse section was helpful in knowing the depth of welding as found to be on etching. Similarly in the case of pressure vessel cover dom, it required bigger joint portion to reveal the weld defects.

However in the case of brittle failed portion of dragline sleeve and a portion of rail axle, the portions were seen in the SEM. By virtue of its depth of focus, the SEM has got unique advantage of over optical microscope to study the fractured surface in the as received condition.

The tool marking on the failed isolation value of the LPG storage tank was yet another example of utilisation of SEM for such investigation. Additionally, the application of EDAX in finding out the nature of inclusions (elemental analysis) gives a better understanding of the causes of failure.

SEM has enhanced the quality of microstructural studies so far done by optical microscopy. Sensitisation and grain boundary studies in S.S. centrifuge was possible only under SEM/EDAX. Chemical analysis of the samples are also carried out to verify whether the materials are conforming to the specification.

Physical characterisations like hardness measurement, tensile test, impact test are also carried out to confirm the desired mechanical properties expected as per specification.

In some cases TEM investigations are also carried out to known the phases of the precipitates/carbides etc. so that a better meaningful reasoning can be arrived at in the end of various studies of the failed samples.

CASE STUDIES

FAILURE OF A LOCOMOTIVE AXLE

The axle failed after having been in use for only three months [1,2,3]. The axle failed near the journal by fracturing transversely. The surface that failed was close to one of the wheels about 45 to 55 mm from the inner wheel seat collar (Fig. 1). The axle had been proof machined and assembled in the carriage after it had been
ultrasonically tested. The chemical composition corresponded to the stipulated ASTM A 21-78 specification as given in Table - 1.

**Table 1 : Chemical composition (Wt. %)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.52</td>
</tr>
<tr>
<td>Si</td>
<td>0.23</td>
</tr>
<tr>
<td>Mn</td>
<td>0.63</td>
</tr>
<tr>
<td>S</td>
<td>0.014</td>
</tr>
<tr>
<td>P</td>
<td>0.02</td>
</tr>
</tbody>
</table>

The failed ends of the axle were examined visually and the longer side was found to be badly mutilated. The other end which was shorted, showed signs of fatigue failure (Fig. 2). The macro investigation revealed that weld metal was deposited all around the circumference of the axle. This was apparently done to build up an undersize area, which is not a recommended practise. Further investigatio
supported that contention and showed distinct wavy patterns of weld pool solidification, fatigue striations and hairline cracks around the circumference of the axle. The depth of the weld was found to vary from 5 to 9 mm. It was concluded that the failure was due to fatigue and cracks were initiated by welding discontinuities.

Microstructural study of the base metal and weld zone revealed (Fig. 3, 4, 5) initiation and propagation of the fine cracks at the edge of the surface. The base metal showed ferrite pearlite structure, whereas weld samples showed paths of fatigue cracking, Widmanstatten ferrite and acicular structures. These are typical cast structures with coarseness of the prior austenite grains. These structures are harmful and can generate stress that could initiate and propagate fatigue cracks. This stress was confirmed by X-ray measurement. Hydrogen content and mechanical properties support the above conclusion, which were evaluated and are shown in Table - 2.

<table>
<thead>
<tr>
<th>Table 2 : Mechanical properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>UTS</td>
</tr>
<tr>
<td>-------</td>
</tr>
<tr>
<td>Base metal</td>
</tr>
<tr>
<td>Weld</td>
</tr>
</tbody>
</table>
This brief metallographic investigation shows that the premature failure of the locomotive axle occurred because of the use of welding as a means of salvaging an undersized axle.

FAILURE OF PRESSURISED STORAGE TANK

Various storage tanks are used by petroleum industries for storing crude and its fractions for final products. They are of different sizes and have dome shaped fixed roofs. These roofs are welded in segments and strengthened to withstand higher than atmospheric pressures in many cases. In case if the welding is not properly done, some voids and fissures along with microstructural anisotropy can persist. Particularly damaging is hydrogen pickup during welding. Since these tanks go through fluctuating pressures during filling in and releasing of the stored product, chances of low cycle fatigue during service are probable. This can result in the coalescence and progress of the voids and fissures When criticality of crack size is reached an unstable and cataclysmic growth takes place and failures of the component takes place.

In the present case a pressurised storage tank handling naptha fraction (60–90°C) as the feed for toluene and benzene separation unit failed. Extensive microstructural and mechanical properties indicated defects in welding, hydrogen pick up and also build up of cyclic stress. The failure had taken place all along the circumferential weld joints. Chemical composition of the material is given in Table 3.

<table>
<thead>
<tr>
<th>Tank No.</th>
<th>C</th>
<th>S</th>
<th>P</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Failed tank</td>
<td>0.18</td>
<td>0.025</td>
<td>0.03</td>
<td>0.0011</td>
</tr>
<tr>
<td>Another tank</td>
<td>0.23</td>
<td>0.023</td>
<td>0.02</td>
<td>0.0076</td>
</tr>
</tbody>
</table>

The composition roughly corresponds to BS–15 and also to ASTM 516.

Microstrucual Study

Macrostructural photographs showed brittle fracture (Fig. 6) which is possible in ductile material only when fluctuating pressure develops. Optical microstructures of the base material indicated ferrite pearlite and banded structure as is normally found in rolled products. However welded zone microstructures showed great variation and defects like fissures Widmanstatten.. structures and voids (Fig. 7). SEM study of weld pool and HAZ showed voids along the grain boundary and transgranular cracks. TEM photographs showed the presence of globular inclusions, which was not desirable.
Hydrogen content

The base material showed the hydrogen content to be about 0.30 ppm, but near the weld joint it was greater than 2 ppm. This too was not desirable.

Mechanical Properties

The hardness was found to be 145 to 150 VPN at 30 kg load whereas it varied from 200 VPN to 220 VPN in welded zone. This naturally generated stress in the welded portion, which was not desirable. Grain size and mechanical properties are given in Table - 4

<table>
<thead>
<tr>
<th>Tank No.</th>
<th>Grain size (ASTM)</th>
<th>Hardness (VPN) Base</th>
<th>Hardness (VPN) Weld</th>
<th>Tensile MPa</th>
<th>RA%</th>
<th>EL%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Failed tank</td>
<td>8</td>
<td>150</td>
<td>220</td>
<td>490</td>
<td>59</td>
<td>27</td>
</tr>
<tr>
<td>Another tank</td>
<td>8</td>
<td>148</td>
<td>220</td>
<td>512</td>
<td>58</td>
<td>35</td>
</tr>
</tbody>
</table>

Remarks

The base material although appeared to be sound, while defects were introduced during welding, either because of shrinkage or because of higher hydrogen pickup [5,6,7]. The flux impurities, fissures, voids in HAZ and weld pool were identified by metallographic technique which justifies its importance in failure investigation.

FAILURE OF ISOLATION VALVE OF LPG STORAGE TANK

Another example of the utility of metallography is shown in the investigation of failure of an isolation valve fitted to a LPG storage tank. The analysis carried out included chemical composition, metallography, fractography and fracture toughness. This case study showed that the normal defects which would pass through
NDT examination, during quality control, could combine to form cracks leading to catastrophic failure, during service.

The chemical composition of the material were comparable to the specified standards as shown in Table 5.

<table>
<thead>
<tr>
<th>Element</th>
<th>Valve</th>
<th>ASTM–A216A(3)</th>
<th>Bolt</th>
<th>AISI4140(3a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.10</td>
<td>0.1–0.25</td>
<td>0.44</td>
<td>0.38–0.43</td>
</tr>
<tr>
<td>Si</td>
<td>0.37</td>
<td>0.6 max</td>
<td>0.19</td>
<td>0.15–0.30</td>
</tr>
<tr>
<td>Mn</td>
<td>0.77</td>
<td>0.7–1.2</td>
<td>0.82</td>
<td>0.75–1.10</td>
</tr>
<tr>
<td>Cr</td>
<td>0.21</td>
<td>0.5 max</td>
<td>0.92</td>
<td>0.8–1.1</td>
</tr>
<tr>
<td>Mo</td>
<td>0.13</td>
<td>0.2</td>
<td>0.18</td>
<td>0.15–0.25</td>
</tr>
<tr>
<td>S</td>
<td>0.02</td>
<td>0.045 max</td>
<td>0.012</td>
<td>0.040 max</td>
</tr>
</tbody>
</table>

Even though the mechanical properties like, hardness, impact, tensile etc. corresponded to the specifications, the material failed because of some other reasons.

The visual examination showed machining defects near the bottom neck of the valve (Fig. 8), where failure took place with brittle fracture. The brittle fracture in a ductile material as concluded from the mechanical properties and chemical analysis indicated some fatigue failure. The ferrite pearlite structure of the valve material and tempered martensite of bolt material were as expected. The D₃ oxide type inclusion was slightly on the higher side for the valve material.

The SEM study of the fractured surface (Fig. 9) showed the presence of high S (0.11%) along with Ca, whereas the S content of the base material away from fracture was 0.02% only. This high S in the fracture was responsible for the brittleness.
of the material [9]. Sulfides are known to inhibit cathodic reaction and thus its presence can cause a build up of absorbed hydrogen in to the metal, which may increase the tendency to cracking. A defect, like the machined notch acts like a crevice, where the volatile S ingress into the material, enhances the local stress intensity and thus initiate cracking. A combination of material defect (inclusion), machining defect (tool mark) and operating environment (volatile sulphur) each of which individually cannot cause a failure of this nature, occurring around the same region has led to the catastrophic failure of the isolation valve [10,11].

Reasons were confirmed by using extensively SEM/EDAX and optical metallography that the isolation valve did not fail due to material used, but because of other factors described above.

FAILURE OF S.S. CENTRIFUGE

Austenitic stainless steels of AISI 300 series are having excellent corrosion resistance. It can handle concentrated oxidising acids without any adverse effect. However, while making containers or equipments cold drawn rolled products are needed. With higher deformation due to cold drawing, a part of the austenite is transformed to α' martensite [12,13]. This may lead to stress corrosion cracking.

Another factor is that if the equipment is heated to ~650°C as heat treatment or some other needs like decontamination, then sensitisation takes place and chromium carbide migrate to grain boundaries. This makes the matrix less corrosion resistance and also the chromium carbide dissolves in acids and grains can disintegrate and S.S. sheet can crumble to pieces. This could happen near the weld zone also. The explosive companies use this material S.S. Centrifuge for the separation of liquid nitroglycerine (NG) from nitrating acid, where H₂SO₄ is a catalyst. Because of some contamination due to HCl acid or chloride ions, the corrosion starts.

![Fig. 10: (a) Corrosion/erosion of cover dome of the centrifuge, (b) corrosion/erosion of top frame x0.50](image)

In the present study [14] one such failure took place, where optical metallography and SEM/EDAX were found to be useful in the conclusion of the above two factors.
Macro photographs are showing the badly corroded surface of the centrifuge (Fig. 10a,b), which indicated the intensity of corrosion.

Chemical analysis confirmed that the material was 304 AISI Stainless steel.

Optical microscopy showed thick grain boundaries and twinning indicating the austenitic structure with sensitisation. Some parts showed delta ferrite islands which cannot exist in 304 S.S. (Fig. 11a,b,c).

SEM Study: Cover dome of the centrifuge indicated intergranular corrosion and porosity in grains. White particles are dye penetrant (Fig. 12a,b).
v) Metallography of Welded part: Weld zone parts showed pores and ferritic structure. This also was not desirable (Fig. 13 a,b).

To conclude

i) improper material was chosen,

ii) welding was not done with care.

To avoid sensitization and α' martensite formation 316L S.S. has been recommended. Also welding has to be done with proper welding rod and post weld treatment was recommended.

In this investigation also, metallography played important role in arriving at meaningful conclusion.

FAILRE OF WALK SHAFT SLEEVE OF DRAGLINE

In mining to remove over burden, a dragline of different bucket capacity (25 cubic meter) are being used. Walk shaft sleeve is a static component made of cast forged steel and is a part of the structural assembly. The sleeve is a heavy steel casting of about seven tons. Proper casting, heat treatment and press forging are
needed to obtain suitable microstructures/grains to provide a fracture resistant material.

The present investigation is a case history of prematurely failed sleeve\textsuperscript{[15]}. The chemical composition and mechanical properties were found to be as per specification. The chemical composition is shown in Table 6.

\textit{Table 6: Chemical composition}

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.20</td>
</tr>
<tr>
<td>Mn</td>
<td>1.80</td>
</tr>
<tr>
<td>Si</td>
<td>0.32</td>
</tr>
<tr>
<td>Ni</td>
<td>0.32</td>
</tr>
<tr>
<td>Cr</td>
<td>0.35</td>
</tr>
<tr>
<td>P</td>
<td>0.006</td>
</tr>
<tr>
<td>S</td>
<td>0.03</td>
</tr>
</tbody>
</table>

This composition is similar to ASTM A487 class 2 steel which is recommended for excavating equipment\textsuperscript{[16,17]}.

**Mechanical Properties**: They were measured in Instron Universal testing machine and Vicker's Hardness measuring machine, and the results are given in Table 7.

\textit{Table 7: Mechanical properties}

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness</td>
<td>200 VPN at 30 kg load</td>
</tr>
<tr>
<td>UTS</td>
<td>57 kg/mm\textsuperscript{2}</td>
</tr>
<tr>
<td>EL%</td>
<td>46%</td>
</tr>
<tr>
<td>02% PS</td>
<td>45 kg/mm\textsuperscript{2}</td>
</tr>
</tbody>
</table>

The macro photo showed typical brittle fracture, which was not expected from such ductile material (Fig. 14).

The microstructure of the failed sample, polished and nital etched shows tempered fine bainitic cast structures indicating that cast structure has not been fully removed and proper heat treatment was still needed to remove the cast structure (Fig. 15a,b).

The ASTM grain size was found to be no 3 indicating rather coarse grain, not desirable for such equipment.
SEM study of fractograph of the sleeve showed that the material was not killed properly (Fig. 16). It shows the presence of coagulated iron oxide in the globular form indicating insufficient deoxidation which may also be responsible for poor resistance to fracture. EDAX was used to confirm that it was only iron oxide and no other inclusion existed.

**CONCLUSION**

In the present paper, five case studies have been presented. In all the cases we observe that optical microscopy is essential for any failure investigation. The inclusion rating, grain size, cast structure, rolled structure, carburisation and its depth welding quality and so many other things can be studied and reasons concluded towards the desirability of the material for various components. The additional use of SEM/EDAX can further add to the authenticity of the conclusions. Particularly fractured surface can be studied by SEM/EDAX alone. Chemical and mechanical properties evaluation are to be done to support the investigation.
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