3 CR 12 TUBE FAILURES IN KESTNERS – KOMATI MILL

DC OLWAGE

Transvaal Sugar Limited, Komati Mill

Abstract

During the project phase of the Komati Mill various alternative technologies, plant and materials were considered. The decision was made to make use of 3CR12 material for the manufacturing of tubes used in the evaporators, pans and scalding juice heaters. After installation leaks were detected in the first two stages of the quadruple evaporation train near the end of the first milling season. Some of the process changes made to manage the contaminated condensate and technical investigations undertaken into the origin of tube failures are described.

Keywords: Factory, tubes, Kestner, failure.

Introduction

The tubes used in the first and second effect evaporators of the Komati Mill are 50,8 mm in diameter, with a wall thickness of 1,2 mm. They are 7,2 m long and are made of 3CR12. The tubes in the third and fourth effects, waste heat evaporators and pans are of the same material but differ in length and diameter. Only the first and second effect evaporator tubes will be discussed in this paper. Close to the end of the first crushing season sugar traces were recorded in the first effect and V1 evaporator condensates. Since this condensate is used for boiler feedwater it caused a great deal of concern and immediate investigations were carried out to determine the source of the contamination.

The higher than normal sugar traces suggested possible tube failure and pressure tests were done on the vessels. These tests revealed that 120 tubes in the first effect and 7 tubes in the second effect evaporators had leaks which ranged from cracked tubes to pin-hole leaks. These failed tubes comprised 1,87% of the total of 6774 tubes in both first and second effect evaporators. Although this was a small percentage of the tubes, the magnitude of the leaks could have brought the whole factory to a stop. These tubes were immediately plugged and only pulled and replaced by spare 3CR12 tubes during the 1994/1995 off-crop. The evaporators were pressure tested before being put into use for the 1995 season. Samples were taken from the failed tubes and sent to independent metallurgical laboratories for analysis. The results will be discussed under the section on Technical investigation.

On 23rd June 1995 high sugar traces in the evaporator condensate alarmed the Komati Mill personnel and on 26th June 1995 both first and second effect evaporators were pressure tested. Eighteen tubes and 28 tubes in evaporators 1 and 2, respectively, leaked. It was decided to plug the leaking tubes and to pay special attention to the quality of the evaporator condensate which would determine the frequency of samples taken, pressure tests, inspections and action necessary. The following steps were taken to manage the situation:

- The frequency of condensate monitoring was increased.
- The demineralising and softener plants were commissioned.
- A contingency plan for spare tubes/alternative tubes was set in place.
- Rubber and steel plugs were made available.
- Material analysis (mechanical and chemical) of tubes was performed.
- The trend of tube failures and positions was studied.
- The condensate pipeline to receive waste heat condensate was redesigned.
- Analyses of products (juice and steam sides) were done.
- Control parameters (conductivity, sugar trace) were evaluated.

All of the above led to a large number of investigations, analyses of material and product, checking of operational and control philosophies, and meetings with consultants, suppliers and contractors. The outcome will not only assist in the determination of the present modus operandi, but also the strategy for selecting tubes for future use at the Komati Mill.

Operation and control

The operation and control of the evaporator condensate pumping system is relatively simple and is as follows: the first effect (exhaust) condensate and, if available, the waste heat condensate is pumped to the boiler de-aerator. V1 condensate is pumped to the boiler feed water tank. The third and fourth effect condensates are pumped to the process water tank as final condensate. The conductivities of the condensates are monitored and if above setpoint, the relevant condensate is dumped to the process water tank. The leaks in the tubes of the first and second effect evaporators caused contamination to such an extent that the level of the boiler feed water dropped to a critical level. The following contingency plans were undertaken:

- The conductivity setpoint was increased from 20 to 40 micro siemens.
- The frequency of taking sugar trace samples was increased.
- When V1 condensate sugar trace exceeded 50 ppm, dosing of caustic into boilers was commenced.
- The demineralising and softener plants were put into service to assist in the supply of boiler feed water.
- Piping modifications were done with the effect of accepting condensate from final condensate. Provision had also to be made to dump V1 condensate to the process water tank. Although far from ideal, it did help to keep the boiler feed water tank to a level where the boilers and factory could operate.

Technical investigation

Various samples of 3CR12 tubes that leaked were given to metallurgical laboratories to analyse and report on the aspects given below. During the 1995/1996 off-crop 10 tubes which did not show any visible leaks were pulled from the evaporators in discussion (5 each from the first and second effect evaporators, selected randomly). They will be reported on under the respective headings.

Visual examination

Some of the tubes exhibited cracking along the length of the tube, close to the weld but not in the weld or heat affected zone (HAZ). The longest crack was approximately 300 mm which was extraordinary and isolated. Most of the cracks were between 3 mm and 15 mm in length and located randomly. Prominent scratches running adjacent to the weld seams were observed and appeared to be in line with the cracking observed and as if the cracking could be related to these scratches.

Severe pitting was observed on the tubes and the extent of pitting corrosion appeared to be higher on the inside of some of the tubes. Other tubes showed limited pitting corrosion on the inside of the tube and marginal pitting on the outside.

Another sample had a large rounded pit close to the seam weld and associated with a distinct blue tint. Similar pits were observed well away from the weld. This and the non-penetration of other elongated pits showed the initiation to have occurred on the outer surface of the tube.

Mechanical damage on the inside of the tubes was observed and may have occurred during abrasive cleaning by mechanical descalers to remove scale deposit on the inside surface of the tube. This operation was only implemented two or three times and the damage is minor and appears inconsequential. The mechanical damage on the outer surface of the tube is of greater significance (see Figure 1).

From Figure 1 it can be seen that the tubes generally conformed to the SX 3CR12 specification. However, the carbon content of the samples submitted to Lab B and Lab C indicated a higher carbon content than that of the 3CR12 specification. The difference is in the analysis technique used and sample preparation for carbon detection which can influence the accuracy of the results. At the time of writing the said variation was still under investigation by Transvaal Suiker Bpk (TSB) as to what relevance it has to the failure of the tubes. From the titanium (Ti) content of the samples it is clear that all the tubes analysed were from the unstabilised 'new' 3CR12. The 'old' titanium stabilised 3CR12 has a much higher titanium content (0.143% versus 0.006%). In the case of tubes manufactured from the 'old' titanium stabilised 3CR12, martensite formation in the weld is limited to the grain boundaries which, combined with the large grain size that results, yields a brittle weld structure. Welding of tubes manufactured from the 'new' unstabilised version of 3CR12, however, yields a much greater volume fraction of martensite in the weld metal and heat affected zone, which refines the grain size and improves weld toughness (van Bennekom et al., 1995).

Mechanical properties

Specimens were cut from the sample tubes and Vickers hardness tests were performed. Table 2 summarises the results obtained.

Residual stresses of the non-cracked and cracked tubes were determined by the 'change in diameter' method. It was possible to determine the following average residual stresses:

- Cracked tube: 370 MPa
- Non-cracked tube: 252 MPa

From the above results it can be seen that the cracked tubes are subjected to both higher residual stresses and hardness than the tubes without cracks. Although the material passed the initial internal SX 3CR12 mechanical property specification, the tubes under test had undergone work hardening through the tube production process. Hardening could also be attributed to occasional over-night interruptions to the production and annealing process. It is also interesting to note that cracking occurred away from the area of higher hardness, i.e. the heat affected zone (HAZ). With the tubes subjected to high enough residual stresses, it only required a stress raiser in the form of pits to initiate a crack.

Metallographic examination

Optical microscopy, Scanning Electron Microscopy (SEM) and Energy Dispersive Spectrographic (EDS) analysis were...
used in the examination of pitted areas. These examinations revealed the following:

- The pitting corrosion was observed on both inside and outside the tube – randomly distributed and appeared to be more prominent on the inside of some of the tubes examined. Other samples showed severe pitting originating on the outside of the tube.
- The pitting corrosion observed could not be related to the weld metal or HAZ but occurred randomly with the worst pitting corrosion occurring in the parent metal adjacent to the weld (in the approximate vicinity of a scratch). Figure 2 shows that the pitting corrosion occurred in the mechanically damaged (scratched) areas.

Sample A: Products found on the outside of the tubes consisted of Fe, Cr, Mn, Ca, P, Si, S, Cl, Al and Mg. Products found on the inside of the tubes consisted of Ca, Si, P, Mg and S.

Sample B: Products found inside a pit of a tube sample consisted of Si, S, Cr, Fe and Ni, with high levels of chromium and nickel, above the specification of 3CR12. Although these elements may have been detected due to the presence of some form of precipitate, it is possible that they were deposited during a weld repair or from an arc strike during welding of another region.

• Isolated clusters of numerous tiny pits were observed on the outer surface of the tubes during SEM examination. Although not of concern as they are very small and shallow, they are an indication of the origin of the pits.
• Although the presence of inter-metallic inclusions could initiate localised corrosion, no evidence of these particles was observed. However, the large amount of corrosion in these regions would have resulted in any foreign particles being removed.
• Mechanical damage in the form of wear grooves and imbedded particles was visible on the inside of the tube. The damage is minor and inconsequential.
• More serious damage, in the form of intergranular attack, was observed on the inner surface of the tube examined. This attack, although still relatively minor, is most probably a result of boiling caustic soda solution as an alternative to mechanical cleaning of the tubes.
• SEM analysis indicated that the mode of failure changed from intergranular to transgranular as the stresses per cross sectional area leading up to failure increased. The failure of the tubes could therefore be attributed to intergranular corrosion (see Figure 3).
• EDS analysis conducted in the pits and surrounding areas revealed the following:

**Figure 2: Mechanical damage (scratch) with pitting corrosion products visible (114 X).**

**Table 4: Water analyses**

<table>
<thead>
<tr>
<th></th>
<th>Raw water</th>
<th>VI condensate</th>
<th>HP steam</th>
<th>HP water</th>
<th>Filter water</th>
<th>Exhaust condensate</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH value</td>
<td>7,55</td>
<td>8,4</td>
<td>7,55</td>
<td>8,90</td>
<td>7,65</td>
<td>9,00</td>
</tr>
<tr>
<td>Conductivity (mS/m @ 25°C)</td>
<td>84.4</td>
<td>45.7</td>
<td>21.8</td>
<td>3.02</td>
<td>96.5</td>
<td>2.04</td>
</tr>
<tr>
<td>Chloride (ppm)</td>
<td>146</td>
<td>8.0</td>
<td>5.0</td>
<td>5.0</td>
<td>118</td>
<td>3.0</td>
</tr>
<tr>
<td>Fluoride (ppm)</td>
<td>0.5</td>
<td>0.2</td>
<td>0.1</td>
<td>0.1</td>
<td>0.3</td>
<td>0.1</td>
</tr>
<tr>
<td>Sulphate (ppm)</td>
<td>12</td>
<td>-</td>
<td>1</td>
<td>&lt;1</td>
<td>18</td>
<td>1</td>
</tr>
<tr>
<td>Calcium (ppm)</td>
<td>47</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Magnesium (ppm)</td>
<td>45</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

**Discussion**

The chemical composition indicated that the material examined conformed to the chemical specification of SX3CR12 material. However, slightly higher carbon percentages were found in the material by independent laboratories and further investigations will reveal its relevancy to the tube failure problem.

It was categorically stated in most of the laboratory reports that no excessive non-metallic inclusions could be found when the material was microscopically examined in the unetched condition. The results of the mechanical examinations performed revealed higher residual stresses and hardness in the cracked tubes than in the tubes that did not crack. The material did pass the internal SX3CR12 mechanical properties specification on release and it can be said that the higher mechanical properties could therefore be attributed to work hardening (cold working) of the material during manufacturing. The problem could be corrected with a suitable...
post weld stress relieve heat treatment which would assist in the prevention of cracking, but the corrosion pitting problem should receive a great deal of attention since it is the originator of further deterioration.

The pitting corrosion appeared both on the inside and outside of the tubes, with the latter more prominent. These pits then act as stress raisers and cause subsequent cracking when the stresses are high enough. The origins of the pits on the outside of the tubes were mostly near or at mechanically damaged areas (scratches and/or indentations) but were also found randomly near and away from the weld and HAZ. The possibility of pitting resulting from corrosion around intermetallic stringers (string out inclusions) formed during the rolling of tube material should not be ignored. The pitting corrosion taking place on the outside of the tubes could be explained by the presence of inorganic ions, e.g. Ca in the cane juice. The S levels detected could be attributed to inorganic ions in the cane juice or the Sulphamic acid rinsing that had been in use but has now been discontinued. The method of removing scale deposit from the inside of the tubes was changed from mechanical cleaning (only done 2-3 times during the season) to boiling a caustic soda solution in the juice space of the evaporator.

The EDS analysis also indicated the presence of Cl on the outside of the sampled tubes. Although the levels detected were low, it could cause localised corrosion, i.e. pitting. The water analysis indicated high chloride levels but it was in the raw water and filter water. The only time this water will be in contact with the outer surface of the tube is during pressure testing of the vessel when raw water is used.

The VI and exhaust condensates have low chloride levels which is an indication that the corrosion would probably be the result of factors other than excessive chloride contents of the condensates. However, more frequent sampling of both VI and exhaust condensates is advised in order to accept the chloride levels confidently.

Conclusions

Experience in the sugar industry has to date not proven 3CR12 as the most cost effective and reliable material to use for evaporator tubes. Kestner tubes are long (7.2 m) and semi-flexible, and will be vibrating under working conditions, introducing additional stresses to the inherent residual stresses. They will crack at any place where the stresses are high enough and initiated by pitting corrosion. The pitting corrosion is a function of the presence of ions such as Cl and areas of mechanical damage. The most important aspect of the corrosion resistance of stainless steel and 3CR12 is the existence of a film of chromic oxide and if this oxide film is removed by mechanical damage, scratching or indentation, corrosion will take place and initiate the process that will lead to tube failure.

The utmost care should therefore be taken in the use of 3CR12 tubes with specific attention being paid to:

- Material specification
- Prevention of metallic/non-metallic inclusions
- Contamination during manufacturing
- Pickling and passivation
- Stress relieving heat treatment
- Packaging and transport
- Installation
- Water treatment/condensate quality
- Method of scale removal inside tubes
- Costing.

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