



## **CORROSION FATIGUE AS A DAMAGE MECHANISM AND ITS PREVENTION AT THE VAPOR ZONE INNER SURFACE OF A KRAFT PULPING PROCESS CONTINUOUS DIGESTER: A CASE STUDY**

**Alexandre Nakayama**

Klabin S.A. Fazenda Monte Alegre s/n, Telemaco Borba - Paraná - Brazil  
nakayama@klabin.com.br

***Abstract.** The continuous digester of a Kraft pulping process is a vertical pressure vessel subjected to severe operational conditions due to the alkaline cooking environment along with high temperatures and pressures needed to separate the lignin from the wood chips to form the cellulose pulp. Among the several damage mechanisms that can be active during a continuous digester operation, the corrosion fatigue is one of the most important because it is hard to detect and monitor during inspections besides its propagation rate and initiation are widely variable and dependent on cooking parameters. This paper describes how corrosion fatigue has been detected, the condition it initiates and the mitigation method applied at the upper part (vapor zone) along the life cycle of a continuous digester manufactured in 1975 under AD Merkblatt code with 750 tons per day of current capacity.*

**Keywords:** corrosion fatigue, continuous digester, structural integrity.

### **1. INTRODUCTION**

At the Kraft pulping process, that is used by 97% of cellulose pulp mills in Brazil (Bracelpa, 2012), the continuous digester is a key equipment since it requires large investment in acquisition and maintenance, is responsible for the wood chips cooking that allows the pulp fibers separation and is highly critical in operations terms due to multiple input variables which demands advanced control systems (Correia, 2010)

The continuous digester is basically a vertical pressure vessel where occurs the delignification of the wood chips under high temperature, pressure and alkaline environment. Its downtimes have a huge economical impact over the whole process and it is one of the most serious and potentially severe exposures in a pulp mill (Hayes, 2002). Therefore, the understanding, controlling and minimizing of the hazards and potential losses associated with operation, maintenance and inspections are of primary importance.

An important issue to be addressed in order to keep the lifecycle of a continuous digester economical is to know what potential damage mechanisms can occur and determine which mechanisms are currently present in the equipment. This allows the maintenance and operational staff to improve damage detection in early stage, preview a trend of damage rates and avoid unplanned stoppages.

Among the damage mechanisms that can be active in a continuous digester operation, the corrosion fatigue is one of major importance to understand. The correlations between operational parameters and equipment construction materials make it of poor predictability since it may vary widely in growth rate and nucleation points according to mechanical, metallurgical and chemical variables involved in the cooking process (Gangloff, 2009).

This paper describes how the corrosion fatigue presents in a digester with over thirty-five year of operation time and the measures taken to prevent it at the digester upper sector called vapor zone. The continuous digester of this case study is built entirely in non coated carbon steel, 750 tons of cellulose per day of capacity and built according to AD Merkblatt code.

### **2. CONTINUOUS DIGESTER**

Digesters of the Kraft pulping process can be batch or continuous. At this case study, the digester is a continuous type where the cooking reactions occur continuously, the wood chips along with a chemical aqueous solution composed mainly of sodium hydroxide (NaOH), sodium sulfide (Na<sub>2</sub>S) and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) denominated white liquor are fed continuously at the top of the pressurized vessel through rotating valves and the delignified fibers are discharged through the bottom device. It presents in its interior during cooking process simultaneously liquid, solid and vapor phases and its operational behavior is strongly dependent on the conditions of heat and mass transfer in addition to the evolution of the chemical reactions inside (Correia, 2010).

Continuous digester can also be classified in hydraulics or vapor zone (Ribeiro, 2007). Hydraulic digesters are completely filled with liquor and wood chips and vapor zone digesters have the upper sector filled with steam coexisting with white liquor liquid phase and wood chips solid phase. Since the upper cone of the equipment depicted on this paper coexists three phases, it is considered a vapor zone digester. Three main distinctive regions are formed inside during normal operation: impregnation zone where the wood chips are impregnated with heated white liquor,

Alexandre Nakayama  
Corrosion Fatigue as a Damage Mechanism and its Prevention at the Vapor Zone of a Kraft Pulping Continuous Digester

cooking zone where the delignification reactions occur and the washing zone where fibers washing and residual lignin removal occur (Paoliello, 2010).

The shell is manufactured in welded carbon steel without coating or weld overlay. The bottom cap and the whole cylindrical part are made with ASTM 285 Gr C with 37,5 mm thickness and the upper cone the material used is NTU SAR 55 with 19 mm thickness. “Figure 1” is a schematic drawing of the digester that is 26 m height and 4,6 meters of maximum diameter and “Fig. 2” is a detailed drawing of the upper section denominated vapor zone.

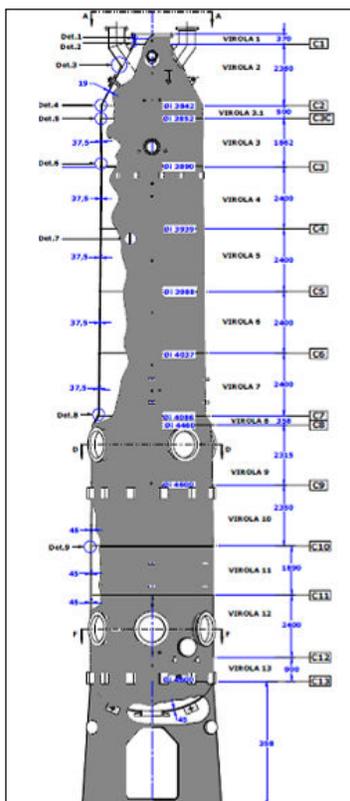


Figure 1. Digester schematic drawing

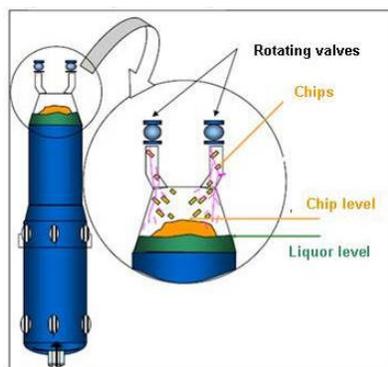


Figure 2. Detail of the upper sector denominated vapor zone

### 3. CORROSION FATIGUE AND OPERATION CONDITIONS

Corrosion fatigue is defined as the sequential stages of metal damage that evolve with accumulated load cycling, in an aggressive environment compared to inert surroundings, and resulting from the interaction of irreversible cyclic plastic deformation with localized chemical or electrochemical reactions (Gangloff, 2009). Or basically, as described in Wensley (1996), corrosion fatigue is the simultaneously action of cyclic stresses and corrosive environment.

The crack resulted of corrosion fatigue damage mechanism generally initiate at the material surface, unless previous defects exist near the surface that actuate as stress concentration point and, in consequence, favoring crack nucleation (Silva, 2006). As for the crack propagation, different variables can influence: mechanical (stress load cycle, maximum load), metallographic (grain size, alloy composition, alloy components and impurities distribution) and environmental (temperature, pH, viscosity).

Specifically in vapor zone continuous digester, the region most susceptible to a severe corrosion is where the inner surface of the shell is “washed” with white liquor (Paoliello, *et al.*, 2011). As seen at “Fig. 2”, the wood chips and the white liquor enter the digester through rotating valves creating a continuous flow that, partially, washes the inner surface of the upper cone. The white liquor, depending on its composition, can be very corrosive to carbon steel. The main components of the white liquor are sodium hydroxide (NaOH) and sodium sulfide (Na<sub>2</sub>S) which are active reagents. As cited by Paoliello (2010), historically the liquor corrosivity has been regarded as function of its inorganic components sodium hydroxide (NaOH), sodium sulfide (Na<sub>2</sub>S) and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) with the concentration of sodium hydroxide (NaOH) playing a major role.

Although the sodium hydroxide is a corrosive agent, some content is beneficial to maintain a passive layer in carbon steel (Wensley, 2003). The content in a narrow concentration range of 10 to 20 grams/liter contributes to the carbon steel passivation while above 25 grams/liter it becomes progressively more corrosive as seen at “Tab. 1”. Continuous digesters have three distinctive regions during normal operation (impregnation, cooking and washing) and each one may present a different sodium hydroxide concentration due to the reactions dynamics. Typically higher concentrations are found at the upper zones decreasing downwards. The white liquor used in this case study digester has an average of 90 gram/liter of sodium hydroxide when fed with wood chips.

Table 1. Concentration of sodium hydroxide NaOH in the white liquor and the corrosion regime in carbon steel.

Concentration of NaOH (grams/liter)	Corrosion regime
<2	High
2 to 12	Moderate
13 to 25	Low
> 25	Progressively higher

According to Paoliello (2010), the corrosion caused by white liquor in digesters may be anodic or cathodic reaction. Carbon steel can have active or passive behavior in white liquor environment depending on its electrochemical potential. The active condition is a high corrosion rate whereas the passive is a low corrosion rate. Polarization curves demonstrate the relation between electrochemical potential and corrosion rate or corrosion density (Wensley, 2003). “Figure 3” is a polarization curve for materials that present a transition passive-active behavior such as carbon steel (Nunes, 2007).

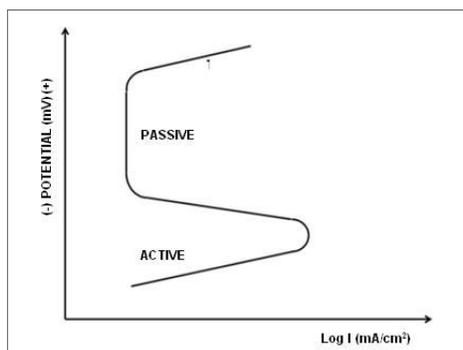


Figure 3. Polarization curve for materials with passive-active behavior.

The continuous digester of this case study processes a category of cellulose pulp denominated K1. K1 has an average 17 Kappa number that represent the delignification degree of the pulp and is measured by the oxidation of potassium permanganate (KMnO<sub>4</sub>) which indicates the amount of oxidizable material (Ribeiro, 2007).

Since the digester is a vapor zone type, the operation pressure is regulated by injection of steam at the upper cone and the pressure fluctuates as seen at “Fig. 4” according to the demand of the production process, wood chips parameters and level inside de digester. Regarding corrosion fatigue damage mechanism, the inner surface of the vapor zone is highly susceptible since it presents a cyclic stress and a corrosive environment with low passivation due to the washing flow of white liquor through the cone walls.

Alexandre Nakayama

Corrosion Fatigue as a Damage Mechanism and its Prevention at the Vapor Zone of a Kraft Pulping Continuous Digester

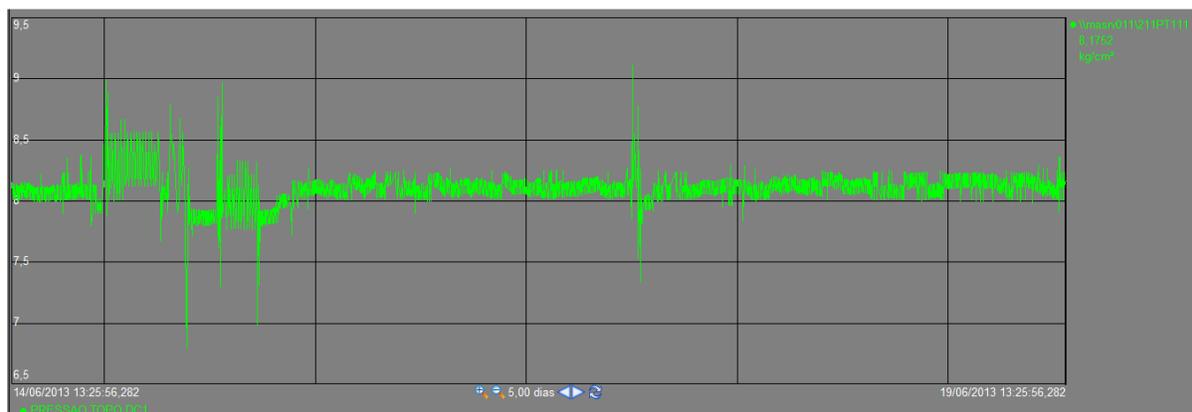


Figure 4. Digester cooking pressure fluctuation.

#### 4. MITIGATION METHOD

Since the internal pressure of the digester is not sufficient to cause considerable stress on the shell, the residual stresses from manufacture or maintenance procedures are the main sources of risks to the corrosion fatigue mechanism. According to Ferreira (1984), besides contractions of weld bead solidification, there are two other main mechanisms that form residual stresses in a given steel structure: faster surface cooling in relation to the center of the piece known as quenching effect and volume expansion of the weld bead and adjacent region due to phase transformation known as transformation stresses. All these three residual stresses generation mechanisms can be active during procedures of maintenance and manufacture of the vessel that alongside a corrosion environment can cause corrosion fatigue damage. “Table 2” (Usiminas, 2013) shows the material composition and mechanical properties of the vapor zone shell, carbon steel NTU SAR 55 with 19 mm thickness.

Table 2. Composition and mechanical properties of NTU SAR 55 carbon steel

C (% max)	Mn (%)	S (% max)	P (% max)	Tensile strenght (MPa)	Yield strenght (MPa)
0,18%	1,2 - 1,5	0,03	0,03	550 - 630	360

According to Cheong *et al.* (2006), shot peening has excellent effects on fatigue life. Shot peening increases hardness and induces a compressive residual stress in the material surface. When repeated load is applied to the material, the compressive layer will compensate partially the tensile strength, increasing the resistance to corrosion fatigue.

The inner surface of the vapor zone was shot peened during an annual outage of the digester. Three main parameters must be controlled during shot peening process: peening media, intensity and coverage (Cheong *et al.*, 2006). The peening media must be uniform in size and hardness. Intensity is the control of energy transmitted to the peening media and has direct influence on velocity which the peening media hits the surface and determine the thickness of the compressive layer. Coverage must be total over the surface in order to avoid residual stresses due to different compressive stresses on different regions.

The peening media used was steel spheres grade S-330 with 0,85 mm diameter. The method was manual, therefore it was established the air pressure at the nozzle at 6,5 kgf/cm<sup>2</sup>, nozzle diameter of 38 mm, nozzle work distance from the surface of 140 mm and incident angle of 80° to 85°. The coverage rate was established by performing a test similar to the Almen Strip Test as show in “Fig. 5”.

Material samples with the same hardness of the NTU SAR 55 and 1,6 mm thickness where placed in holders and shot peened during different time in order to specify the linear velocity of the nozzle and consequently the coverage rate. This test was performed to determine the shot peening intensity through the arc height inflicted by the peening media under determined circumstances of pressure, angle and nozzle distance.

The velocity determined by the test was approximately 1,15 m<sup>2</sup>/h based on the deflection of the samples which determined the compressive layer thickness and visual characteristics of the surface according to Standard Almen Strip Test (2013). The result is designated as B at “Fig. 5” representing a half thickness arc height. The coverage through the whole inner surface of the vapor zone was controlled visually with an overlap of 10 % considering the nozzle diameter.

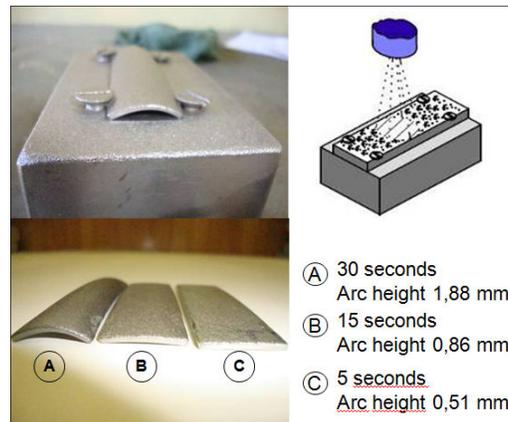


Figure 5. Test performed to determine the coverage rate.

## 5. RESULTS

The shot peening was applied at the inner surface of the upper cone sector during the 2011 annual outage. The inspection plan is based on the guidelines of technical information paper 0402-27 of Technical Association of the Pulp and Paper Industry TAPPI (2010) and section 13.10 of Regulatory Norm 13 (NR-13) of the Brazilian Labor Ministry. Visual and Non Destructive Testing such as Magnetic Particle, Dye Penetrant and Ultra-sonic thickness measurement inspections are performed annually during plant outage.

In 2011 inspection it was detected 12 cracks at the vapor zone inner surface. “Figure 6” shows the morphology of one of the cracks detected with MP inspection. The cracks were grinded off and filled with SMAW weld. The 2011 to 2012 operation regime of the continuous digester did not change in terms of average pressure, production output and Kappa number of the cellulose pulp.

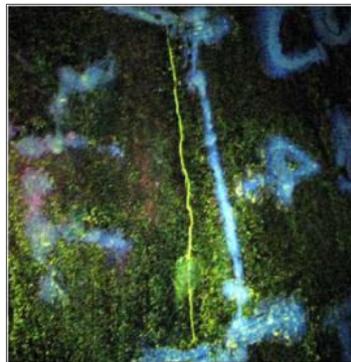


Figure 6: Crack detected in 2011 with Magnetic Particle Inspection

In 2012 inspection performed in June, it was detected 3 cracks, all of them shallow (up to 2 mm depth) and with smaller length than observed during 2011 outage.

## 6. CONCLUSION

Comparing the NDT results performed during 2010 through 2012 annual inspections, the quantity and severity of the cracks found at the inner surface of the digester vapor zone decreased significantly after the application of shot peening. The shot peening procedure induced a plastic deformation at the inner surface of the vapor zone which in turn produced a compressive layer that acted as a pre-load to the internal pressure of the digester. This reduced the cyclic tensile strength at the surface during digester normal operation and consequently reduced the corrosion fatigue damage mechanism.

Since operational regime did not change in terms of pulp output, temperature and chemical composition of the white liquor, the shot peening with main parameters adjusted to the conditions of the shell material NTU SAR 55 showed to be beneficial in corrosion fatigue cracking prevention at the digester studied.

Alexandre Nakayama  
Corrosion Fatigue as a Damage Mechanism and its Prevention at the Vapor Zone of a Kraft Pulping Continuous Digester

## 7. REFERENCES

- BRACELPA, 2012. “Dados do Setor”. Jun. 2013 < <http://www.bracelpa.org.br>>.
- Cheong, C.K., Nam, J.H., Lee, J.H. and Kim, T.H., 2006. “Effects of Shot Peening on the Corrosion Fatigue Life of Al 7505-T6” In *Proceedings of the 2nd Conference on Shot Peening*. Seoul.
- Correia, F.M., 2010. *Análise de distúrbio de compactação de cavaco de eucalipto em digester contínuo fase vapor*. Dissertation, UFV, Viçosa.
- Ferreira, M.L.R., 1984. *Martelamento como método de alívio de tensões em juntas soldadas*. Dissertation, UFRJ, Rio de Janeiro.
- Gangloff, R.P., 2009. “Corrosion Tests and Standards Manual”. Jun. 2013 <<http://www.virginia.edu/ms>>
- Hayes, M.J., 2002. “Extending digesters outage intervals – the insurance company perspective and managing risks”. In *TAPPI Fall Conference & Trade Fair*. San Diego, USA.
- Nunes, L.P., 2007. *Fundamentos de Resistência à Corrosão*. Editora Interciência.
- METSO, 2009. “Report EN-MCA-885: Mechanical project review of the Esco continuous digester – Ad Merkblatt standart ed. 1986”
- Ministério do Trabalho e Emprego, 2008. “Norma regulamentadora 13 – Caldeira e Vasos de Pressão”. Brasília, Brazil.
- MKS, 2011. “Inspection report RV 46512”
- MKS, 2012. “Inspection report RV 50588”
- Paoliello, F.A., 2010. *Corrosão de digestores contínuos: influência dos processos de cozimento e análise das tecnologias protetora*. Dissertation, UFV, Viçosa.
- Paoliello, F.A., Lins, V.F.C. and Cardoso, M., 2011. “Influence of Cooking Conditions on Continuous Digester Corrosion on a Brazilian Pulp Mill” *TAPPI Journal*.
- Ribeiro, R.N., 2007. *Utilização de Redes Neurais Artificiais e Tecnologia FT-NIR para Predição do Número Kappa em um Processo Kraft de Cozimento de Madeira em Indústria de Celulose*. Dissertation, Unileste, Coronel Fabriciano.
- Silva, J.A.S., 2006. *Desenvolvimento e construção de uma bancada de testes para ensaios sob corrosão-fadiga em dutos e estruturas metálicas*. Dissertation, UFCG, Campina Grande.
- Standard Almen Strip Test. Jun 2013 < [www.ewp.rpi.edu/Almen-DesignNote5A2.pdf](http://www.ewp.rpi.edu/Almen-DesignNote5A2.pdf) >
- TAPPI, 2010. “TIP 0402-27 Guidelines For Inspection of Continuous Digester and Impregnation Vessels”. *Technical Information Paper*.
- USIMINAS, “Catálogo chapas grossas” Apr 2013 <[www.usiminas.com/irj/go/km/docs/prtl\\_doc/usiminas/pt/AreasDeNegocios/Siderurgia/CatalogoProdutos/Catalogos2013](http://www.usiminas.com/irj/go/km/docs/prtl_doc/usiminas/pt/AreasDeNegocios/Siderurgia/CatalogoProdutos/Catalogos2013)>
- Wensley, A., 1996. “Corrosion and protect of Kraft digesters”. *TAPPI Journal*, Vol. 79.
- Wensley, A., 2003. “Anodic Protection Against Corrosion and Cracking of Digester Vessels” In *TAPPI Fall Conference & Trade Fair*. Toronto, Canada.

## 8. RESPONSIBILITY NOTICE

The author is the only responsible for the printed material included in this paper.