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ABSTRACT

The University of Missouri-Rolla identified materials that permit the safe, reliable and economical operation of combined cycle gasifiers by the pulp and paper industry. The primary emphasis of this project was to resolve the material problems encountered during the operation of low-pressure high-temperature (LPHT) and low-pressure low-temperature (LPLT) gasifiers while simultaneously understanding the materials barriers to the successful demonstration of high-pressure high-temperature (HPHT) black liquor gasifiers. This study attempted to define the chemical, thermal and physical conditions in current and proposed gasifier designs and then modify existing materials and develop new materials to successfully meet the formidable material challenges.

Resolving the material challenges of black liquor gasification combined cycle technology will provide energy, environmental, and economic benefits that include higher thermal efficiencies, up to three times greater electrical output per unit of fuel, and lower emissions. In the near term, adoption of this technology will allow the pulp and paper industry greater capital effectiveness and flexibility, as gasifiers are added to increase mill capacity. In the long term, combined-cycle gasification will lessen the industry's environmental impact while increasing its potential for energy production, allowing the production of all the mill's heat and power needs along with surplus electricity being returned to the grid. An added benefit will be the potential elimination of the possibility of smelt-water explosions, which constitute an important safety concern wherever conventional Tomlinson recovery boilers are operated.

Developing cost-effective materials with improved performance in gasifier environments may be the best answer to the material challenges presented by black liquor gasification. Refractory materials were selected/developed that either react with the gasifier environment to form protective surfaces in-situ; and were functionally-graded to give the best combination of thermal, mechanical, and physical properties and chemical stability; or are relatively inexpensive, reliable repair materials. Material development were divided into 2 tasks:

Task 1, Development and property determinations of improved and existing refractory systems for black liquor containment. Refractory systems of interest include magnesium aluminate and barium aluminate for binder materials, both dry and hydratable, and materials with high alumina contents, 85-95 wt%, aluminum oxide, 5.0-15.0 wt%, and BaO, SrO, CaO, ZrO_2 and SiC.

Task 2, Finite element analysis of heat flow and thermal stress/strain in the refractory lining and steel shell of existing and proposed vessel designs. Stress and strain due to thermal and chemical expansion has been observed to be detrimental to the lifespan of existing black liquor gasifiers. The thermal and chemical strain as well as corrosion rates must be accounted for in order to predict the lifetime of the gasifier containment materials.

TABLE OF CONTENTS

LIST OF GRAPHICAL MATERIALS

INTRODUCTION

The Tomlinson recovery boiler is the conventional technology for recovering cooking chemicals and energy from black liquor. As a potential replacement for the Tomlinson recovery boiler, black liquor gasification (BLG) technology has garnered much interest over the last two decades in the papermaking industry. The BLG technology has higher energy efficiency and generates far more power with overall lower cost than conventional technology. It improves safety by reducing the risk associated with smelt-water explosions. It reduces the wastewater discharges and harmful emissions into the environment. BLG systems recover sodium and sulfur as separate streams that can be blended to produce a wide range of pulping liquor compositions [Stigsson (1998)]. As a technique that is still under development, it has problems including refractory failure during operation due to a combined effect of chemical reaction and thermomechanical stress [Brown and Hunter (1998), Dickinson, Verrill and Kitto (1998)]. The objective of this study is to investigate the failure behavior of refractory lining under chemical and thermomechanical loading by using an analytical model.

High temperature black liquor gasifiers are generally cylindrical in shape as shown in Figure 1. The height ranges from 1.5 m to 25 m and diameter ranges from 0.5 m to 5 m. In the gasifier reactor vessels, there are usually 2-6 coaxial layers of component lining [Taber (2003)]. Refractory lining is used to protect the exterior metallic part of the gasifier vessel. A dense refractory material layer is designed to be exposed to the highest temperature environment. The second “safety” layer is usually made of a similar material. Subsequent layers are used to provide insulation and allow for expansion. The steel shell is used to provide reaction space and confinement. The gasifier generally operates at temperature ranging from 950 to 1000 ^{o}C.

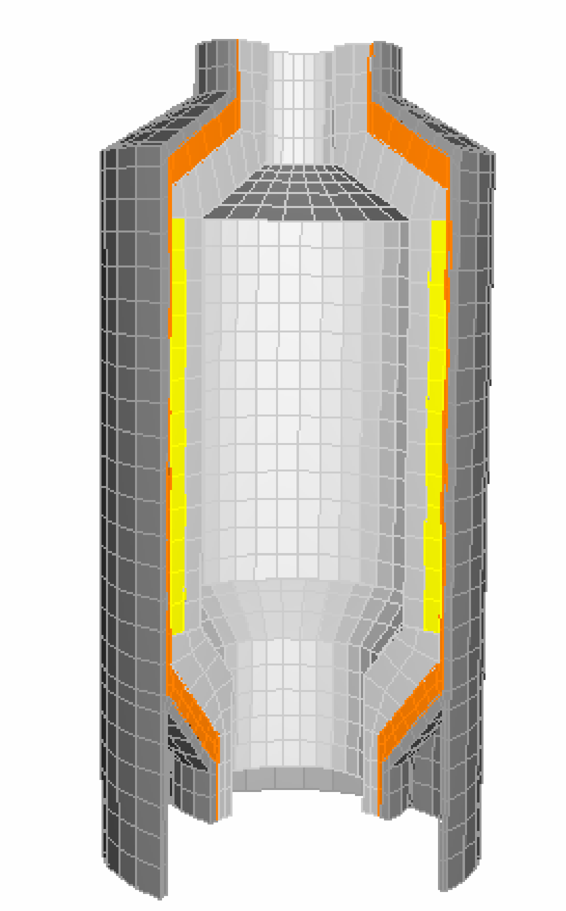

Figure 1 Schematic construction of a typical high temperature gasifier

The commercial high temperature black liquor gasifier was developed by Kvaerner Chemrec. A pilot plant first started running in 1994 at a pulp mill near Karlstad, Sweden [Larson, Consonni and Katofsky (2003)]. The first commercial size Chemrec system (75-100 tons of dry solids/day) was built at the AssiDomän mill in Frövifors in 1991. This air blown gasifier has performed well and been proven to be easy to operate and maintain. The first commercial Chemrec system in North America started operation in 1996 at Weyerhaeuser's New Bern, SC, USA [Brown and Hunter (1998)]. It was an atmospheric, air-blown, entrained bed gasifier operating between 950-1000 ^oC with a capacity of 350 ton black liquor solids per day. However, this system was shutdown in January 2000 due to failure of the stainless steel shell [Brown and Landalv (2001)].

Black liquor gasification converts the organic components into combustible fuel gas and leaves inorganic components as smelt to generate high-quality green liquor for regenerating pulping chemicals [Kelleher and Kohl (1986)]. The combustible gas contains carbon monoxide (CO), hydrogen (H_2), carbon dioxide (CO_2), methane (CH_4), nitrogen (N_2), water vapor (H_2O) and hydrogen sulfide (H_2S). The smelt drops are mainly sodium carbonate (Na_2CO_3) and sodium sulfide (Na_2S). Some of the smelt drops form a thin layer of smelt flowing along the reactor wall.

The current refractory materials for the BLG reactor vessel lining are not deemed adequate. The combination of high temperature and alkalinity produces an aggressive environment for the reactor lining. Chemrec has used several refractory materials in the pilot units and the commercial atmospheric units. The refractories last from 1 to 18 months, with a replacement cost of up to 1 million dollars and several weeks of downtime. Severe refractory thinning occurred and several bricks were found lost from the upper part of the gasifier vessel during operation. The refractory lining is subjected to the penetration of sodium and subsequent reactions with alkali-rich molten smelt, such that the refractory undergoes significant volume change and strength degradation. Several refractory samples have been studied after immersion in molten smelt by Peascoe, Keiser, Hubbard, Brady and Gorog (2001). The results of their study are summarized below. For mullite based refractories, molten smelt first attacks mullite and forms sodium aluminum silicates. This reaction is accompanied by a volume change. A significant surface expansion occurs during immersion testing in smelt. Furthermore, a liquid phase can develop in the mullite refractory as Na_2O concentration increases. Surface expansion coupled with the loss of structural integrity lead to the spalling of the lining. $MgAl_2O_4$ spinel based refractories react with the smelt to form $NaAlO_2$ and MgO, with an associated expansion of 2.1% to 13%. For α/β-alumina refractories, expansion was accommodated partly through spalling and a significant radial expansion of the gasifier's lining. The alumina refractories show the least corrosion, the chemical expansion of alumina samples is from 0 to 0.7%. Due to this reason, fused cast alumina which is expansive and sensitive to thermal shock is being used in the most recent commercial high temperature black liquor gasifier at New Burn, SC, USA, [Brown, Leary, Gorog and Abdullah (2004)].

Computer simulation of existing materials will accelerate the development of these new materials. Compared to experimental characterization, computer simulation is much faster and more economical. Finite element modeling of damage evolution in refractory linings exposed to high temperature and aggressive chemical environment was presented.

EXECUTIVE SUMMARY

Black liquor gasification is a high potential technology for production of energy which allows substitution for other sources of energy. This process uses a waste of the pulp and paper industry as black liquor to produce synthetic gas and steam for production of electricity; therefore development of this technology not only recovers the waste of the paper industry but also decreases dependency on fossil fuel.

Today one of the main obstacles in the development of this technology is the development of refractory materials for protective lining of the gasifier. So far the materials used for this application have been based on alumino-silicate refractories but, thermodynamics and experience shows that these materials are not sufficiently resistant to black liquor under the harsh working conditions of Black liquor gasifiers. Consequently development of cost-effective materials with improved performance in gasifier environments to answer the material challenges presented by black liquor gasification (HTHP, HTLP) was the objective of this project. Refractories provided by in-kind sponsors were tested by cup testing, density/porosity determinations, chemical analysis and microscopy. Magnesia and spinel based gunnable refractories and mortars were developed in this project. They show great promise and should improve the current status of high temperature gasification.

Computer simulation of existing materials will accelerate materials research in developing these new materials, and it is less costly and time consuming. Finite element modeling was conducted in this study.

EXPERIMENTAL

Tests are being performed as described in earlier reports and ASTM standards for Task 1.4 – Submit for Industrial Trial. In addition, all components replaced in industry are being modeled by the finite element method to predict failure mode, stresses and eventually lifetime.

RESULTS AND DISCUSSION

Project is progressing according to plan as shown in Table 1. Task 1.3 is currently complete, additional materials will be tested under this task if requested by manufacturers or end-users. Task 1.4 and 2.0 are nearing completion. An additional gunnable magnesia material was received in June and has been tested. It is the fifth iteration of a material being produced as a hot repair for the current lining and as a future lining. This material has great promise as it is a spinel matrix magnesia trowlable, castable and hot gunning mix.

In Task 1.4, Materials were tested and modeled for the pulse combustors at Big Island. The MorcoCast AZ10 developed, tested and recommended appears to be performing adequately. It was reported by MORCO that Big Island has ordered additional material to replace the current refractory in the remaining pulse combustors.

Currently a spinel lining is being used in New Bern, although there is no strong connection between the selection of the spinel lining and this project. It is of note that spinel is one of the materials strongly suggested by this project. Mortar developed is being tested by Oak Ridge National Laboratory, ORNL. If this mortar performs better than other mortars, it is anticipated that it will be used as the mortar for existing and future gasifiers.

Table 1 Gantt Chart

	2002	2003				2004				2005		
Task Name	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3
1.0 Refractory Determination												
1.1 Perform Thermodynamic Modeling												
1.1.1 Compile Material Data Table												
1.1.2 Compile Reactions												
1.1.3 Compile Gibb's Free Energies												
1.1.4 Compile Phase Diagrams												
1.2 Measure Properties												
1.3 Conduct Simulative Corrosion												
1.4 Submit for Industrial Trial												
2.0 Model Development												
3.0 Project Management												
3.1 Task 1.1 Report					12/31 ◊							
3.2 Task 1.2 Report							6/30 ◊					
3.3 Task 1.3 Report										3/31 ◊		
3.4 Task 1.4 Report												10/31 ◊
3.5 Task 2.0 Report												10/31 ◊
3.6 Task 3.0 Report												12/31 ◊
3.7 Quarterly Reports			7/31 ◊	10/31 ◊	1/31 ◊	4/30 ◊	7/31 ◊	10/31 ◊	1/31 ◊	4/30 ◊	7/31 ◊	10/31 ◊
3.8 Continuation Report			7/31 ◊				7/31 ◊					

Task 1.4

Refractory materials are being evaluated and modeled to be used in the pulse combustors, shown in Figure 2, that are failing at Big Island and Trenton.

Figure 2 Picture of failed pulse combustor tube sheet.

An enlargement showing the critical delamination at a depth of 5-6” into the 11” thick by 5’ diameter panel is shown in Figure 3. The delamination eventually would lead to blockage of the heat exchanger tubes and necessitates refractory replacement. Material properties of two possible replacement materials have been measured and a finite element model of the tube sheet was developed.

Figure 3 Enlargement showing critical delamination failure at 5-6" in depth and accompanying transverse cracking.

The AZ-10 material installed at Big Island failed in 2 pulse heaters due to improper installation and in 2 pulse heaters due to attack by gases generated by the surrounding Pligun LWI28 insulating castable. It is currently believed that the AZ10 is not strong enough for use in the pulse heater and that chemical attack from other refractory materials is leading to failure in the tube sheet. MORCO is redesigning the AZ10 to have higher strength and better flow characteristics. Optional materials for this installation are 90+ % alumina dense refractories. It is important that all refractory materials be compatible.

Task 2.0

Models of the pulse combustor tube sheet were completed. It was determined that there is a minimal change in stress distribution with different anchor shapes. The wavy V anchor was the best at distributing the stress along the length of the anchor. Maximum principal stress in the refractory with hook occurs at 62.9 hours of heating when hot face temperature is 1223°C; Maximum principal stress in the refractory with anchor occurs at 66.9 hours of heating when hot face temperature is 1302°C.

Table 2 Stress comparison for anchor types

	Maximum principal stress (MPa)	Maximum radial stress (MPa)	Maximum axial stress (MPa)
Refractory with hook	365	200	326
Refractory with V anchor	344	153	342

CONCLUSION

Samples provided by in-kind sponsors were tested using cup testing. The best performing materials in the cup testing were fused cast materials. Magnesia brick and castables performed very well and should be moved into industrial trials.

Computer simulation of existing materials will accelerate materials research in developing these new materials, and it is less costly and time consuming. Finite element modeling was conducted for the damage analysis in this study. Both and HTLP gasifier and the pulse combustor used for LTLP gasification were studied. MorcoCast AZ-10 refractory has been recommended as a replacement of existing materials based on the model and thermo-mechanical properties measured and is currently in industrial trial.

This study presented continuum damage mechanics based analytical model for predicting the failure behavior of refractory lining in cup testing. The damage model accounts for the chemical expansion in addition to mechanical and thermal expansion. A comparison of predicted damage patterns for BLG refractory material with the observed damage pattern in the cups used indicates that this model could be used to evaluate failure behavior of refractory linings in black liquor gasifier.

Chemical reaction and thermal expansion with improper constraints causes the most compressive damage in the refractory structure. Layered damage occurred in the refractory structure due to the tensile damage. Expansion allowance affects the damage of the refractory structure. Tensile damage could be reduced by allowing for larger expansion.

No systematic experimental work has been done so far to characterize the failure behavior of refractory materials in black liquor gasifier. Experimental work is needed to validate the models presented here.

Gasifier end-users are still experiencing major refractory problems. Refractories are one of many issues still slowing use of this energy saving technology. Without continued assistance from DOE black liquor gasifiers currently in use may be shut down and future work abandoned.

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