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Integrating 3D Layered Manufacturing with Photonic Sintering, Precision Machining and Smart Coating Techniques for Rapid Casting Applications

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INTEGRATING 3D LAYERED MANUFACTURING WITH PHOTONIC SINTERING, PRECISION MACHINING AND SMART COATING TECHNIQUES FOR RAPID CASTING APPLICATIONS

by

Hemant Bohra

A dissertation submitted to the Graduate College in partial fulfillment of the requirements for the degree of Doctor of Philosophy Engineering and Applied Sciences Western Michigan University August 2014

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INTEGRATING 3D LAYERED MANUFACTURING WITH PHOTONIC SINTERING, PRECISION MACHINING AND SMART COATING TECHNIQUES FOR RAPID CASTING APPLICATIONS

Hemant Bohra, Ph.D.
Western Michigan University, 2014

Developments in rapid casting technologies have led to a new era of inclusion of 3D printing. Three-Dimensional (3D) printing provides the flexibility and ease of reproducing a sand mold directly from CAD models, eliminating patterning steps, thus reducing the process time for creating prototypes. In addition to minimizing processing steps, 3D printing provides the advantages of higher precision and the ability to produce complex shaped sand molds, but it simultaneously possesses some limitations and concerns related to throughput, safety and logistics.

This study proposes an alternative method for creating sand molds by introducing a hybrid rapid prototyping approach to overcome the limitations observed for conventional 3D printing techniques. By this method, thermosetting resin-coated sand particles can be bonded layer-by-layer after being exposed to a high energy photonic light source, which raises the layer temperatures to a desired range for bonding directly followed by precision machining, to obtain complex shapes.

This study focuses on the determination of the physical, mechanical and thermomechanical properties of the printed layers and process functionality, while using both an additive and subtractive approach to selectively integrate machining with the high photonic curing mechanism and on the comparison of the achieved parameters to the results obtained using conventional sand casting techniques.
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CHAPTER I

INTRODUCTION

Manufacturing technology refers to the processes involved in the production of structures with definite shapes identified by material and geometric characteristics [Grote and Antonsson, 2009]. Various processes are involved in achieving the desired parameters, which can be classified into different groups, forming, cutting, joining, etc. Casting can be considered the backbone of most shaping processes for various applications where metalcasting is a key component. Metal casting [Schleg et al, 2008] can be explained as the process of pouring molten metal into a shaped cavity in order to transform it into a desired shape upon solidification (Figure 1.1). The primary step of this process involves the creation of a shaped cavity, also known as a mold. A mold can be prepared using various heat resistant materials such as sand, which provides the primary advantage of cost effectiveness. The molding processes [Schleg et al, 2008] can be classified into permanent and non-permanent where the mold is destroyed to remove the casted metal structure (e.g. sand molds). This step is referred to as the shakeout process. The casting process offers many advantages over other primary shaping processes, such as freedom of castability over a wide range of metals of desired shapes and versatile mechanical properties. Achieving net near shape and integral castings leads to the reduction in the time and costs associated with any additional machining and assembly requirements. Altogether, the advantages of the process include savings in material and energy use along-with recycling and ecological benefits.
The properties of a casting [Schleg et al, 2008] can primarily be identified by the geometry/shape of the object, chemical composition/characteristics of the casted material, treatment of the molten material prior to casting, material and process parameters involved during mold-making, rate of solidification and the following heat treatment.

The selection of the mold material and mold making process plays a vital role in governing the casting characteristics. Sand being a refractory material [Schleg et al, 2008] can withstand very high metal pouring temperatures, which imparts the required dimensional stability at elevated temperatures. Sand also provides an economical advantage over other comparable refractory materials. Dry sand molding requires the introduction of a binder system to hold the loose sand particles during the mold shaping
process. The finished mold’s physical, chemical and thermo-chemical properties depend on the sand-binder system [Carey, 1998] interactions and the process of creating the mold as well.

The casting molds can be divided into two categories; patterned and pattern-less [Beaudoin et al, 1997]. A patterned mold acquires the desired shape through the deposition of the mold material around a defined shaped object also known as a pattern. The primary concern with patterned mold making is the cost and time involved in creating the pattern with the help of various machining and tooling activities. On the other hand, pattern-less molds can be created directly from a digital drawing/model through the implementation of additive or subtractive manufacturing techniques.

This work focuses on demonstrating the feasibility of introducing a hybrid manufacturing approach, involving both additive and subtractive manufacturing, for pattern-less mold-making in rapid casting applications.
CHAPTER II

LITERATURE REVIEW

Rapid Prototyping and Rapid Casting Technologies

Additive manufacturing [Jang and Ma, 2002], also known as layered manufacturing [Hochsmann, 2011], has attained acceptance in rapid prototyping [Brensons and Mozga, 2011] and rapid manufacturing with the integration of computer aided manufacturing [Bernard et al, 2003] by imparting additional opportunities for attaining complex shapes with higher precision and tolerance levels as compared to conventional molding techniques. Rapid prototyping [Ortiz et al, 2008] refers to the production of prototypes of actual designs. It is used for the product development phase of a production process because it is able to impart the characteristics in close proximity to the finished product, which enables the further investigation and analysis of the product before finalizing the end product features. The rapid casting process defines the integration of traditional metal casting techniques with additive manufacturing approaches to achieve either functional prototypes or end products.

Various rapid casting solutions have been developed during the last few decades to implement concurrent engineering approaches for the development of functional prototypes and customized production of metal castings for applications in various sectors of manufacturing industries. These rapid casting [Chhabra and Singh, 2011] solutions include unique production approaches, such as selective laser sintering, fused
deposition modeling, stereolithography, 3-D printing and rapid tooling.

Selective laser sintering [Levy et al 2003; Kruth et al, 2007] in conjunction with additive manufacturing provides the capabilities to produce sand molds and cores bearing complex shapes or design. The phenomenon of this approach includes fusing binder coated sand particles [Barlow and Veil, 2000] layer-by-layer in desired patterns guided by a CAD solid model using heat energy being generated from a CO₂ laser source (Figure 2.1).

Figure 2.1 Selective laser sintering process [Modified From: www.azom.com].

The investment casting process [Chhabra and Singh, 2011; Cheah et al, 2005]
involves the production of molds by applying ceramic coatings on sacrificial patterns. These sacrificial patterns can be created through the application of various shaping techniques, such as selective laser sintering, fused deposition modeling, stereolithography or rapid tooling. The sacrificial patterns can be either wax based or non-wax based. Fused deposition modeling is an additive manufacturing approach where a material in its solid state is melted and extruded through a nozzle while following the pattern path as directed by CAD file layer by layer to achieve desired shape and dimensions. Stereolithography [Chhabra and Singh, 2011] is also an additive manufacturing process where a pool of liquid photopolymer is cured using a laser source to achieve the desired shape.

The 3-D printing process [Kawola, 2003; Gill and Kaplas, 2009] of pattern-less molds involves the spreading of a layer of dry refractory material particles and to achieve a desired thickness thereafter spraying the desired binder system using inkjet print-head nozzles in the pattern as directed by the CAD model. The printed binder system keeps the loose particles intact to provide the defined shape parameters. These steps are repeated layer-by-layer until the target mold cavity is achieved and the unbound particles in the non-image areas are removed after completion of the final print cycle (Figure 2.2). The resulting mold cavity is thereafter cured to attain the required mechanical strength for handling and metal pouring.
Figure 2.2 3D printing process for prototyping and rapid casting applications [Modified From: www.economist.com].

Hybrid Manufacturing Technique

Hybrid manufacturing [Boivie et al., 2011] refers to integration of additive and subtractive manufacturing technologies to create complex shapes and functional prototypes [Kerschbaumer and Ernst, 2004]. The additive and subtractive layered manufacturing approach has found applications in various areas such as in the rapid pattern manufacturing of sand castings, which implies layer by layer manufacturing along-with the simultaneous machining using chemical wood, where the adhesion of wood slabs over one another refers to the additive approach and milling, and patterning of each slab layer, thereafter corresponds to the implementation of a subtractive approach. The additive and subtractive approach for creating sand casting molds while maintaining the desired strength characteristics and accuracy in the replication of features needs further investigation. The selection of the machining tool is dependent on various
inter-related parameters, such as desired layer thickness, depending on the mechanical bonding strength of material, interaction of cured layer with cutting tool during machining, etc.

Milling and cutting operation performances primarily rely on various input parameters [Kibbe et al, 2009] such as cutting and feed speeds, direction of feed, cutting/milling tool selection, vertical feed rate or depth of the cut, etc. Cutting rates can be defined as being the rate at which a point referenced on a cutter passes a reference point on a work piece in a calculated amount of time. The cutting speed constants are dependent on cutting tool material, work piece material, machine setup, etc. For the machining and milling operations [Kerschbaumer and Ernst, 2004] of sand molds/cores, a ball-end mill tool can be utilized. To obtain optimum results, the cutting speeds should be kept at half to two thirds of the speeds of comparable end mills using different tool sets. The feed rate [Kibbe et al, 2009] can be defined as the rate at which either the material is advanced into the cutter or vice-versa. The horizontal feed rates are also dependent on feed directions being characterized as up milling or down milling. In instance of the material being fed in opposite direction to the rotation of the cutting/ milling tool, is known as conventional or up milling. Whereas, the material being fed in the direction of tool rotation is known as climb or down milling. The selection of horizontal feed direction is highly dependent on the backlash elimination capabilities of machine and achieved material surface finish during the machining operation. The horizontal feed rates are also determined by mechanical properties of material and obtained surface finish, as excessive feed rates can result in very rough or chipped cutting edges. The
vertical feed rates refer to the depth of cut and are governed by the amount of material to be removed from the work piece in a single pass-cycle, power available at spindle, rigidity of work piece material, selection of tool, machine setup, etc. By general rule, in case of soft work piece materials, the depth of cut should not exceed half the diameter of the tool and vertical feed rates should be reduced while making deeper cuts.

The optimum machining parameters for a sand mold/core material, such as cutting speeds, feed rates, diameter of cutting tool, etc., can be determined by conducting a series of machining trials.

Overview of Casting Defects

Casting defects [American Foundrymen Association, 1947] can be defined as undesired irregularities in a metal casting process where occurrence of these irregularities or in other words severity of these irregularities beyond tolerance limits results in additional process steps to either repair or eliminate the irregularities. The presence of these irregularities in a casting can lead to additional tooling/machining of parts and can add high overhead costs associated with these additional process steps while affecting overall process throughput. These defects can be categorized on the basis of their originating parameters such as gas porosity, shrinkage, mold-material, metal pouring and metallurgical defects [British Foundrymen Society, 1963]. The first three defects can be minimized to a great extent through the proper selection of the molding material and casting design, whereas improving the process parameters can minimize the latter two.

The gas porosity [American Foundrymen Association, 1947; British Foundrymen
Society, 1963] defects can primarily be identified as blows/ gas holes, porosity defects, pinholes and blisters. Blows or gas holes are rounded cavities that may be spherical, flattened or elongated and are caused by generation and/or accumulation of gas or entrapped air bubbles. The molding material interfaces showing higher gas evolution rates in conjunction with molten metal interactions most likely impart these defects. These cavities vary in size over a wide range with variations in color imparting dark blue to silver metallic luster. These defects rarely occur in drag surfaces, but most likely are identified as smooth depressions or a series of jagged and irregular depressions on the cope, mostly targeting flat surfaces. Blows can also be caused due to the passage of a stream of gas through the metal, accompanied by inclusion of mold slag. These irregularities or defects sometimes resemble a shrink or dendritic area and hence are mistaken as shrink. In some cases, a portion or entire surface of a casting may be pitted with very small holes known as pinholes, which indicate the presence of subsurface blowholes. Blisters also fall in the same category of casting defects as identified by a shallow blow appearing on the casting surface accompanied by a thin film of metal over it. The major cause of these defects is packing efficiency of granular media to form mold cavities, which is primarily governed by the grain size, shape and distribution. Too closely packed sand grains decrease the mold permeability, hence affecting the venting characteristics by blocking the air passageways at the mold interface. This results in the accumulation of gas/air bubbles at the mold-metal interface.

Erosion scabs (penetration) [ASM International, 2009], expansion scabs (veining) and blacking scabs are some examples of other surface defects primarily originating due
to the improper selection of molding materials. Erosion scabs mostly occur at the mold-metal interface, where molding sand partially erodes away, leaving a solid mass of sand and metal at the particular spot. The eroded sand generally flows to the cope part and is referred to as dirt or inclusions (mold slag). Expansion scabs [American Foundrymen Association, 1947] can easily be identified as rough thin layers of metal followed by partial separation from the body due to the intermediate trapping of a thin layer of sand encompassing a thin vein of metal. In most of the cases the scabs do not get readily chipped off during the shakeout process and appear as indentations on the casting surface, known as buckles. Rattails are minor buckles represented by a small irregular line/lines on the casting surface. Blacking scabs are formed by the expansion of sand resulting in blacking or flaking off and are retained either in or on the surface of metal castings.

Other common metal casting defects [ASM International, 2009; British Foundrymen Society, 1963] include cold shots, shrinkage cavities and depressions, hot tears and cracks, hard spots, misruns and cold shuts, etc. These defects normally exist through a combination of improper selection of mold-material, poor casting design, improper metal pouring procedures and unsuitable metal chemistry, which interfere with the desired solidification parameters. These defects can be minimized to a certain extent by the analysis of the solidification process prior to metal pouring and/or implementing various solidification modeling techniques to detect hot spots and molten metal flow phenomena. Utilizing solidification modeling and simulation techniques [ASM International, 2009], the hot and cold spots obtained during the process can be strategically placed either by modifications to the casting design or by controlling the
heat transfer [Guyer and Ramrattan, 2003] at the metal-mold interface by placing insulating or chill coatings at the mold interface.

**Testing Chemically Bonded Sands**

The bulk density [Schleg et al, 2008] of sand plays a vital role in the estimation of the finished sand mold properties from a handling and logistical prospective. Bulk density is also significant in tracking any lot-to-lot variations in the sand systems employed. The density of a system is representative of mass/volume ratio and can be measured by weighing a known volume of material and thereafter calculating the ratio.

The Grain Fineness Number [Schleg et al, 2008] is indicative of how small or large is the average grain size of a sample of sand. Though smaller grains of sand are capable of imparting better surface finish, due to their closely packed structure, they hinder the venting characteristics of the mold, which can result in various casting defects. The higher the GFN, the smaller the sand grain size. Very low GFN refers to larger sand grain sizes compromising the packing efficiency consisting of larger interstices between the grains and hence resulting in rough casting surfaces. To calculate the GFN, a known sample weight can be placed into an arrangement of sieves, and then be placed in a sieve-testing device with continuous sieve shaking for 15 minutes. Thereafter, the weight of sand retained in each sieve & pan and could be recorded followed by calculating GFN as shown in Table 2.1:
Table 2.1 AFS GFN calculation chart

<table>
<thead>
<tr>
<th>Sieve</th>
<th>Amount Retained (g)</th>
<th>Mesh Opening (µm)</th>
<th>% Retained</th>
<th>Multiplier</th>
<th>Product</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>3,327</td>
<td></td>
<td>0.03</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>1,651</td>
<td></td>
<td>0.05</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>850</td>
<td></td>
<td>0.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>600</td>
<td></td>
<td>0.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>425</td>
<td></td>
<td>0.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>50</td>
<td>300</td>
<td></td>
<td>0.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>70</td>
<td>212</td>
<td></td>
<td>0.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>150</td>
<td></td>
<td>0.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>140</td>
<td>106</td>
<td></td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>75</td>
<td></td>
<td>1.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>270</td>
<td>53</td>
<td></td>
<td>2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PAN</td>
<td></td>
<td></td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>TOTAL</td>
<td></td>
<td></td>
<td>AFS GFN</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The particle size distribution of a sample of sand is determined by the process of screening and hence, also known as screen distribution. In normal practice; the sand systems used in foundries range from three-screen distribution to a highest of five-screen distribution. For determination of particle distribution, a sieve testing could be carried on the sand sample with further calculations to reveal the distribution information.

The pH and Acid Demand value [Schleg et al, 2008], being indicator of the purity of sand system, represents the lot-to-lot variability and hence could be measured prior to further sand testing. The shape and sphericity/ roundness of sand grain can be determined
using an image analysis system.

Permeability [Guo et al, 2000] is a measure of gas flow through a porous medium, such as a sand mold or core. Permeability and MQI tests could be performed to provide a measure of the specimen’s venting characteristics.

The permeability [Pal et al, 2006] of a sand mold or core is affected by several factors including the size, shape, distribution, and the method of compaction of the sand in the mold or core box. Furthermore, permeability is directly affected by the quantity of resin in the sand. Permeability testing is very common in the foundry industry and is part of the sand control tests performed on a regular basis at most foundries. A Gerosa Simpson permeability tester could be used to perform the permeability tests with a specimen holder designed and fabricated at WMU. A special rubber gasket can be used between the specimen and the holder to provide a seal. Additionally, a plug could be used to restrict the airflow in order for the Gerosa Simpson machine to detect the permeability of each specimen.

The Mold Quality Indicator (MQI) test [Ramrattan et al, 1997], which is inversely related to permeability, should also be studied. The MQI number is a measurement of the resulting backpressure developed from the resistance of airflow through a mold or core. The MQI unit equipped with an air pump, air tubing, and a rubber/foam contact head could be connected to the end of the tubing. An MQI unit is typically deployed somewhere along the molding line to perform real time measurements on the molds waiting to receive the molten metal. With some modifications to the original rubber
contact head, this instrument could be utilized with the WMU specimen holder.

The specimen could be secured into a holder, which thereby is fixed to the permeability tester. The test can then be started and the permeability could be measured thereafter removing the holder with the specimen and then attaching to the MQI unit for measurement.

Disc transverse strength tests (DTS) [Iyer et al, 2001] could be used to measure the strength of the sand specimens before and after thermal distortion testing (TDT) [Ramrattan et al, 1997]. The strength properties before TDT relate to the handling of the core/mold material after core/mold production, prior to pouring. The strengths after TDT testing relate to the shakeout/collapsibility characteristics.

The disc-shaped specimen could be fitted into a specimen holder on the testing machine (Dietert Model 490-A) equipped with a disc transverse accessory while being supported on its ends. It can then be subjected to a transverse force by applying a load with a 2.00 mm thick rounded edge blade across its diameter while being loaded at a constant linear load rate. A load-cell electronically senses the specimen failure and digitally displays the results. Thereafter, the maximum load to failure is recorded.

Impact testers could be employed to determine the impact strength of materials. Dynamic tensile behavior being considered as important, the impact test determines the parameters of performance of a material at strain rates closer to some end-use applications. A product being of superior or inferior quality can be judged on the basis of performance index and consistency by analysis of mechanical strength and
variability as well. The impact test conforms the mechanical strength of the product and by consideration of variation in strength properties of the product variability can be determined.

For conducting the impact test, a Tinius Olsen impact tester, with up to 2.825 J or 2,825mJ capacities, could be used to determine the strength disc shaped specimens in terms of breaking energy (Joules). The impact tester can be used in conjunction with a Dynatup energy display, model ETI-220P.

Abrasion resistance defines the property of a material surface to resist wear while in contact with another material. The determination of the abrasion/wear resistance of a cured surface layer plays a vital role in the estimation of the effect on sand mold surface due to handling procedures. This test method encompasses the ability to compare the strength of different sand specimens against scratch or wear caused by handling. A Teledyne Standard Abrasion Tester Model 503 equipped with custom sample holder for disc specimens could be used to determine the friability characteristics.

The 50 mm dia. x 8 mm thick disc-shaped specimens can be weighed and secured onto the sample holder using four screws, one on each corner. The sample holder should be mounted onto the abrasion tester with a ceramic bead pressing against the specimen surface perpendicularly. A desired load (250 g), thereby be applied onto the ceramic bead by mounting corresponding circular weights on top of the abrading assembly. The specimen can then be rotated in clockwise direction maintaining a constant rotational speed for a desired number of cycles/rotations (10-100). To ensure the proper contact
between the ceramic bead and sand specimen surface, a vacuum should be applied continuously to pull away any loose sand particles during the test run. The abrasion/wear resistance of the specimen surface can thereafter be determined by calculating the weight loss or %weight loss.

The thermal distortion tester (TDT) using a disc-shaped specimen is a suitable device to compare chemically bonded sand systems [Iyer et al, 2001]. The temperature is variable and can be set to represent molten metal temperatures for the specific alloy for which the core/mold material it should be used for example aluminum (1400°F, 760°C), copper based alloys (2200°F, 1204°C) and cast iron (2350°F, 1288°C). Different loading pressures can be applied to the specimen to simulate different metallostatic pressures on the core/mold material. After thermal exposure, the test specimen remains intact allowing determination of additional information, which would include the strength, visual analysis for cracks (which in metalcasting process, could result in veining), and weight loss measurement that relates to pyrolysis of binder bridges and the amount of loose, unbounded sand generated at the mold metal interface.

Experiments could be conducted using TDT to expose 50 mm diameter, 8 mm-thick disc specimens to 1400°F (760°C) for up to 90 seconds. The purpose of this investigation would be to test the 3D cured sand system used for rapid prototyping manufacturing and compare with a 3D printed and production grade chemically bonded PUCB (Poly Urethane Cold-box) sand systems [Iyer et al, 2001]. The TDT method could be used as examples of application on foundry mold and core media, and to further
compare the data.

For distortion longitudinal [Rebros et al, 2007; Ramrattan et al, 1997; Iyer at al, 2001] it is possible to differentiate between expansion ($D_E$) and plastic distortion ($D_P$) separately from the thermal distortion curve (TDC). The total longitudinal distortion ($T_D$) can simply be stated as $T_D = \sum D_E + \sum D_P$. The radial distortion ($D_R$) indicating expansion in the radial direction could also be studied using next generation thermal distortion tester (TDTng) developed at WMU.

An image analysis could be conducted thereof to quantify the dimensions of surface cracks after thermal distortion testing using an ImageXpert Analyzer. While using the image analysis method, it is possible to measure the opening (width) and length of visual surface cracks on a given sample.

Gas evolution [Samuels et al, 2011] or generation of gases at the mold-metal interface with flow of molten metal into mold/ core cavities, tend to originate various casting defects as discussed earlier as, the trapped gas always follows the path of least resistance to escape the mold or core. Various experiments [Samuels et al, 2011; Lever et al, 2000; Ahamad et al, 2012] can be conducted for identification of generated gaseous products at mold/core interface when exposed to elevated temperatures. An experimental set-up for conducting identification of these gaseous compounds uses micro scale pyrolysis units, CDS Pyro probe 5250 (CDS Analytical Inc., Oxford, PA) interfaced to a Shimadzu QP-5050A gas chromatograph/mass spectrometer (Shimadzu Corp, Columbia, MD). The test could be performed by packing approximately 0.5 mg of sample between
quartz wool being placed in a quartz tube with a filler rod. Pyrolysis, could be conducted by setting the pyro probe at 1200°C with a hold time of 10s at the maximum heating rate ‘set-point’ of 999 °C/s. The Gas Chromatograph (GC) oven temperature program begins with a 1 minute hold at 40°C followed by heating at a rate 8°C/min to 300°C. The injector and detector temperature could be set at 300°C. The mass spectra could be recorded in electron ionization mode for m/z 14 to 200. Comparing the mass spectra of peaks with standard spectra of other compounds using the NIST library could perform identification of gaseous products.

Surface energy [Kan et. al, 2004; Adamson, 1997] of a surface can be defined, as the energy required for forming a unit area of new surface at the solid-gas interface. Surface energy is an essential component in estimating wettability of a solid surface with a liquid, and hence to characterize the solid-liquid interface interaction properties, such as spreading coefficient. The surface energy of a surface can be estimated by measuring the contact angle of different immiscible liquids on the solid surface. This estimation of the surface energy of a solid surface and surface tension of a liquid can be used in determining the wettability of the liquid on a given surface by calculating the spreading coefficient of the liquid on the surface, as the liquid could spread on the surface of solid only if the spreading coefficient of the liquid on the solid is greater than zero i.e. $S(B/A) > 0$. The spreading coefficient of liquid B on surface A can be expressed by the equation 2.1 [Adamson, 1997].
\[ S_{B/A} = \gamma_A - \gamma_B - \gamma_{AB} \]

Equation 2.1

A positive spreading coefficient is necessarily a non-equilibrium situation. The equilibrium-spreading coefficient is necessarily non-positive, with a negative value corresponding to a nonzero contact angle [Adamson and Gast, 1997].

The contact angle of a liquid on the surface of a surface can be measured using the sessile drop method and hence the surface energy of surface can be estimated [Owens-Wendt, 1969], this value can then be further used to predict the wettability of another liquid of given surface tension on this surface by calculating the (negative) spreading coefficient of the liquid on the surface.

The relationship between the surface energy of mold/core surface and the surface tension [Kan et al, 2004; Adamson and Gast, 1997; Owens and Wendt, 1969] of coating can be proven an essential element of consideration, during selection of optimal refractory coatings.

Initial casting trials can be conducted to study the mold-metal interactions at the mold interface by analysis of casting defects observed. The effect of metallostatic pressures generated on the inner mold/core wall can be studied and thereby requirements of any prior treatment or coating applications can be determined thereof. To conduct the experiment, a sand mold could be developed to deliver molten metal to six disc-shaped specimens simultaneously. Certain disc shaped specimens could experience an external
tangential point load (300g/400g) while the mold is poured to a metallostatic head wetting all surfaces of the specimens. The purpose of this model is to identify the effect of super heat, metallostatic head and external load on chemically bonded disc shaped specimens to determine at what conditions distortion, penetration and/or veining occurs.

The molds containing six symmetrical cavities can be poured with the disc shaped specimens arranged by condition and point loading sequence. This approach could allow the possible variation in casting surface quality (specimen/metal interface) to be assigned to the disc-shaped sand specimens. Molds could be poured manually using a six-inch tall pouring sleeve fitted with a foam filter affixed to the cope as a sprue to deliver the metallostatic head. Castings should be allowed to solidify prior to cooling and shakeout and thereafter be sectioned at the core/metal interface for analysis using an ImageXpert image analyzer to determine the percent coverage of surface defects.

Applications of Refractory Coating in Metal-Casting

Refractory coatings [Guyer et al, 2005] refer to the application of selected refractory materials to resin bonded cores and molds. The applications of these refractory materials [Schleg et al, 2008] serve the purpose to:

(a) Improve the surface finish of mold/ core interface

(b) Control the heat transfer characteristics at metal-mold interface

(c) Alter the venting characteristics of core and mold
(d) Prevent certain defects in casting such as erosion, etc.

In order to impart good surface finish characteristics and minimize/eliminate additional machining/tooling steps, smooth mold and core interfaces are required. The surface smoothness of these interfaces can be altered by the application of refractory coatings. The gas-generated defects could be minimized, closing air/gas passageways located at the mold/core surface by application of refractory coatings. The generated gaseous products could then be forced to escape the system by strategic placement of vents in the mold design.

Identified areas of casting with hindered heat-transfer [Guyer et al, 2005] can be altered by the strategic placement of ‘chill spots’ with application of ‘chill coatings’ imparting better heat transfer and thus faster solidification. The mechanical properties of metal castings can also be altered by application of a chill coating to produce a steeper thermal gradient resulting in harder surfaces. The other applications of refractory coatings is in the placement of insulating materials in areas of casting where faster solidification is not desired in order to impart required mechanical properties along with minimization of certain casting defects such as misrun or cold shut originating from steeper thermal gradients.

The refractory coatings consist of several ingredients [Guyer et al, 2005; Rebros et al, 2007] such as refractory material (pigment), carrying agent (binder), suspension agent (dispersant), rheology modifiers, surfactants, etc. These coatings can be applied by various methods including:
(i) Dipping core and mold sections into coating formulation

(ii) Brushing on the coating

(iii) Spraying coating onto cores and molds

(iv) Strategic placement of coatings onto cores/molds using inkjet heads

The coating rheology plays a vital role in determination of coating process compatible with the desired coating formulation. Rheology [Tanner, 2000; Mezger, 2006] can be defined as the study of the flow and deformation of materials when dealing with forces, stresses, and energy interchanges. The term “rheology” originated from Greek work “rheos” meaning “flowing”, therefore “rheology” refers to the “flow of science”. Deformation can be described three different ways: Reversible, Irreversible and Reversible-Irreversible. Further, a reversible deformation is understood as being elastic, irreversible deformation is flowing or viscous and reversible-irreversible is viscoelastic [Joyce, 2012; Tanner, 2000]. Understanding the micro-geometries of the particles in these materials when forces are applied is crucial in understanding processes in many areas such as polymers, lubricants, coatings, and printing.

Trying to understand rheological aspects of materials is very important. Rheology affects things such as storage capability and shelf life, flow and leveling, smoothness and uniformity, and viscosity and dispersion stability [Joyce, 2012]. By testing coatings or printing inks under shear, the application aspects of flow and leveling become clearer. The concept of this testing is based on fluid dynamics and layer flow. If the shear stress
and the shear rate [Joyce, 2012; Mezger, 2006] of a fluid are known, the viscosity can be calculated. This is useful for tracking viscoelastic properties and transient response of a fluid.

The shear stress is the measure of a force over a specific area and is defined as $\tau = F/A$, where $F$ is the force (shear) (N) and $A$ is the area (m$^2$). The shear rate is the measure of the velocity of a moving piece over a measured gap and is defined as $\dot{\gamma} = \frac{v}{H}$, where $v$ is the velocity of the moving plate (m/s) and $H$ is the gap between the plates. Viscosity of a fluid can be defined as the measure of resistance being offered for the gradual deformation on application of shear or tensile stress. Viscosity can then be calculated by the ratio of the shear stress to the shear rate and is mathematically defined as $\mu = \frac{\tau}{\dot{\gamma}}$ [Joyce, 2012].

Figure 2.3 is a pictorial representation of these terms. It shows how the velocity of the moving plate located on the top, while a stationary plate is located on the bottom is affected by the shear rate. Also seen is the velocity gradient that takes places as a velocity is applied to the top plate. The representation of the arrows that decrease in size shows that the layers within the fluid see decreasing amounts of force created by the top plate.
Testing the rheological properties could also provide information about the behavior of the fluid. A fluid can be Newtonian, similar to water, or non-Newtonian. Newtonian fluids have a constant proportionality between shear rate and shear stress; viscosity is independent of shear rate and shear stress. In a non-Newtonian fluid, viscosity can be effected by either time or shear rate. Time-dependent fluids are considered to be thixotropic if viscosity decreases or rheopectic if viscosity increases at constant shear rate. Time-independent non-Newtonian fluids exhibit behavior that depends on the magnitude of the shear stress and not the time of application. Three types of fluids fall into this group: pseudoplastics, dilatant, and Bingham fluids [Ikoku, 1978]. Pseudoplastics fluids shear thin, dilatant fluids shear thicken; Bingham fluids have a yield
stress. Figure 2.4 is a graphical representation of a thixotropic and rheopectic fluid compared to a Newtonian fluid.

![Graphical Representation of Fluids](image)

*Figure 2.4 Effects of varying time on time-dependent fluids [Modified From: TA Instruments Manual].*

Figure 2.5 shows a graphical representation of the time-independent fluids with varying shear rate.

![Graphical Representation of Fluids](image)

*Figure 2.5 Effects of varying shear rate on time-independent fluids [Modified From: TA Instruments Manual].*

Rheological testing can be conducted using a series of stress controlled/ strain
controlled instruments such as a TA AR 2000. Depending on the equipment used for testing, the test could either be Strain-controlled or Stress-controlled. The AR 2000 is a dynamic Stress-controlled measuring instrument, which applies a torque and reads the rotation of an engraved disk measured by a displacement sensor. Therefore, the applied stress can be measured without contact of the testing equipment, providing very accurate results. Figure 2.6 is an example schematic of a Stress-controlled rheological tester.

![Stress-controlled rheological tester](image)

*Figure 2.6 Stress-controlled rheological tester [Modified from: Vader and Wyss].*

The modern rotational rheometers provide capabilities to fully characterize a given material over a range of temperatures and flow rates [Mavern Instruments] using various test methods such as:
(a) Continuous Flow

(b) Creep Tests

(c) Stress Relaxation

(d) Oscillatory Sweep Tests

Flow curves or flow rate tests are conducted to measure the shear viscosity versus shear rate or shear stress. At sufficiently low shear rates (.001/s) a constant value for the viscosity can be attained, thereafter the zero-shear rate viscosity can be calculated by corresponding model fitting. For polymeric blends the zero shear viscosity depends on the average molecular weight of the polymer and the length of the plateau (how high a rate before the viscosity decreases) and thereby also reflects the width of the molecular weight distribution.

Different geometries can be used to conduct the above-stated tests, however the selection of geometry is made considering the flow parameters of the given sample. There are three different types of rheometer geometries mostly used for these testing purposes: Cone-plate, plate-plate, and couette cell [Mavern Instruments]. Each of the geometries possesses distinct benefits within certain parameters.

The Plate-Plate geometry is best used for highly viscous and higher particle sized fluids. The nature of having two flat parallel plates introduces a non-uniform strain within the fluid but has the ability of having an adjustable gap. Especially, for the specimens to be tested where a gap size of at least 10 times greater than particle size
should be maintained. Geometries can be constructed from a wide variety of materials such as stainless steel, aluminum, acrylic, etc. The geometry being used should ideally be as light as possible to compensate for inherent inertia effects and should possess chemical-compatibility for a wide range of testing specimens to prevent surface corrosion during the test. Usually stainless steel geometries are more preferred, considering the advantages of low thermal expansion and offering chemical compatibility with most test materials. Moreover, it provides the mechanical advantage of being robust enough to withstand heavy use, and to some extent is capable of withstanding handling issues from a less experienced operator. An example of parallel plate geometry configuration is shown in Figure 2.7.

![Figure 2.7 Parallel Plate geometry](Modified From: TA Instruments Manual)

Where, Shear Rate \((s^{-1}) = K_\gamma \omega\), as \(K_\gamma = R/D\) and Shear Stress \((\text{Pa}) = K_\sigma \tau\), as \(K_\sigma = 2/\pi R^3\). \(\tau\) refer to the Applied Torque \((\text{mN-m})\), \(\omega\) as the Angular velocity \((\text{rad/sec})\) and \(K_\gamma\)
& $K_0$ can be defined as the strain & stress constants respectively, and are considered to be geometry dependent factors.

Creep and creep recovery tests [Malkin, 2006; Mainardi and Spada, 2011] are employed to analyze the viscoelastic properties of a fluid as these properties strongly depend on leveling characteristics of a fluid. Figure 2.8 represents the elastic and viscous behavior of a fluid. These tests can be used to evaluate the viscoelasticity of chemically cross-linked polymers [Yen, 2009], dispersions and gels, etc. involving a vide variety of acting physical forces [Le Fèvre, 1953; Atkins and De Paula, 2006] as well as chemically unlinked polymers and solutions [Campbell et al., 2005; Bloomfield, 1992; Chang, 2005].

There are two phases of the creep recovery test, the first being the creep curve, which exhibits the deformation, and the second being creep recovery curve, which depicts reformation of the structure of the fluid/polymer matrix being investigated.

During the first phase of the creep curve, a constant shear stress $\tau_0$ is applied for a defined time interval thereby, carefully monitoring the creeping motion [Petrucci et al, 2002] of the fluid caused by increasing deformation of the fluid structure. In the second phase, the external forces applied on the fluid are removed by application of infinitesimally small shear stress while monitoring the reformation of the fluid structure. The constant stress $\tau_0$ applied during the first phase should not exceed the limiting value in the Linear Viscoelastic Range (LVR) [Yannas, 2004] as when the applied stress exceeds this limiting value, the basic laws of rheology (Newton’s law, Hooke’s law, etc.) [Mezger, 2006] are no longer valid thereby invalidating the application of various
corresponding creep and recovery models (Maxwell, Burgers, Voigt) [Mainardi and Spada, 2011; Karas, 2012]. Generating and analyzing the stress curve by conducting a shear sweep test on the given fluid sample can easily determine the limiting shear stress in the LVR region.

![Elastic and viscous behavior](http://www.helsinki.fi/~serimaa/soft-luentoonsoft-2-viscoelastic.htm)

*Figure 2.8 Elastic and viscous behavior on application of a constant stress. [Modified from: http://www.helsinki.fi/~serimaa/soft-luentoonsoft-2-viscoelastic.htm]*

The ideal-elastic, ideal-viscous (shown in Figure 2.9) or viscoelastic behavior (shown in Figure 2.10) [Roland, 2011] of a fluid can easily be determined by conducting a creep-recovery test. An ideal-elastic material can be defined as the material showing immediate deformation on application of stress followed by immediate and complete reformation on removal of stress. The total deformation energy stored during the deforming step is fully recovered in the reformation phase of the creep-recovery phase. The delayed deformation or reformation experienced by the material/ fluid after application or removal of stress can be termed as retardation.

Fluid/polymer melts, depicting ideal- viscous behavior, reflect continuous
increasing deformation with constant applied stress [TA Instruments] followed by no reformation on removal of stress. These materials do not store energy of deformation by application of load and therefore no reformation is evident after removal of load due to release of stored energy. Behavior of an ideal-viscous fluid is shown below in Figure 2.9.

![Ideal-viscous behavior](Modified From: TA Instruments Manual)

In fluids or polymeric blends, the viscoelastic properties [TA Instruments] can be easily observed in the two phases of the creep-recovery curve, the first phase showing immediate continually increasing deformation on application of constant stress and second phase showing time-dependent partial reformation after the release of applied load. The curve shown below in Figure 2.10 represents an example of a viscoelastic material.
In most of the polymeric samples, the polymer chains at rest have many entanglements and neighboring chains optimizing the minimum energy requirement at rest. On application of shear, creeping motion starts by increasing deformation due to storage of energy followed by change in orientation of polymeric chains in the direction of application of stress. On release of applied stress, the molecules try to return to their original states by slow reformation with release of stored energy.

This viscoelastic behavior of a material and its individual components (elastic and viscous) can easily be predicted by fitting various models to the creep - recovery curve to obtain the best fit with minimal error. There are various models available to predict the viscoelastic behavior of materials such as Burgers Model, Kelvin Voigt Model, Maxwell Model, etc. These model fits can be obtained and hence error can be calculated by

*Figure 2.10 Viscoelastic behavior [Modified from: Johnson, 1985].*
plotting a compliance \(J(t)\) vs time curve. Compliance [Mezger, 2006] can be defined as reciprocal of elastic modulus and defines the stiffness of the material being tested.

The Maxwell element [Mezger, 2006] comprises of a spring and a dashpot element in series (Figure 2.11), representing the two components of viscoelasticity, the elastic modulus and viscous modulus, respectively. This model suggests that when a spring and a dashpot are arranged in a series arrangement then: \(\sigma_{\text{Total}} = \sigma_D = \sigma_S\) and \(\gamma_{\text{Total}} = \gamma_D + \gamma_S\).

\[\gamma(t) = \gamma_0(1 + t/\lambda)\]

\textit{Figure 2.11} Maxwell element and creep of a Maxwell element [Modified From: Oversimplified Viscoelasticity; www.psu.edu]

The creep in the Maxwell element can be defined by equation below:

\[\gamma(t) = \gamma_0(1 + t/\lambda)\]

\textit{Equation 2.2}

Where, \(\gamma\) represents strain, \(t\) represents time and \(\lambda\) represents the relaxation time.
The relaxation time can be explained as the ratio of linear dashpot constant (ratio of force and displacement) and linear spring constant (ratio of force and velocity). The creep shows a linearly increasing pattern in a Maxwell element and the stress relaxation steps of the Maxwell element are shown below in Figure 2.12.

\[ \sigma(t) = \sigma_0 \exp(-t/\lambda), \]

where \( \sigma \) represents stress at a time interval. The oscillating (under damped) case arises when \( G > G_{\text{critical}} \) and non-oscillating (overdamped) case when \( G < G_{\text{critical}} \). \( G_{\text{critical}} = \frac{4\eta^2}{\alpha} \), where \( \alpha = \frac{I F_\sigma}{F_\gamma} \), with \( I \) refering to the measuring system inertia, and \( F_\sigma \) and \( F_\gamma \) refers to proportionality factors between the shear stress and torque, and shear rate and angular velocity, respectively.

The Voigt element [Karas, 2012] as explained by the Voigt model comprises of a parallel arrangement of a spring and dashpot elements (Figure 2.13). Since the Voigt
element contains spring and dashpot elements in parallel arrangement, the total stress and strain relationship can be expressed as $\gamma_{\text{Total}} = \gamma_D = \gamma_S$ and $\sigma_{\text{Total}} = \sigma_D + \sigma_S$. The creep of the Voigt element follows an exponential curve and can be represented by $\gamma(t) = \gamma_\infty [1 - \exp(-t/\lambda)]$ as shown in Figure 2.13.

![Figure 2.13 Voigt element and creep of a Voigt element](Modified From: Oversimplified Viscoelasticity; www.psu.edu).

Jeffery’s model [Mezger, 2006] has two forms being considered as Maxwell and Voigt form as shown in Figure 2.14. These forms show serial and parallel arrangement of an isolated dashpot with a series arrangement of a spring and a dashpot. Equation 2.3 represents the relationship of compliance to viscosity, relaxation time and elastic modulus.
\[ \eta_p(t) = \frac{1}{G} \left( \frac{\eta_p}{\eta_s + \eta_p} \right) \left[ 1 - e^{-t/\tau} \right] + \frac{t}{\eta_s + \eta_p} \]

Equation 2.3

Where, \( \eta_p \) is the viscosity of the dashpot within the Maxwell element, \( \eta_s \) is the viscosity of the isolated dashpot, and \( \tau = (G/\eta_s + G/\eta_p)^{-1} \), where G is the elastic modulus. This represents the Maxwell form of Jeffrey’s model, where the parameters of this arrangement are given by: \( J = (1/G) \left[ \eta_p/(\eta_s + \eta_p) \right]^2 \), \( \eta_V = \eta_s (\eta_s + \eta_p)/\eta_p \) and \( \eta_0 = \eta_s + \eta_p \).

The Jeffrey’s model can be represented in Voigt element form and in this form, \( J \) is the spring compliance, i.e. the reciprocal of its elastic modulus. In terms of the previous form of the model,

\[ \tau + \lambda_1 \frac{\partial}{\partial t} \tau = -\eta_0 \left( \dot{\gamma} + \lambda_2 \frac{\partial}{\partial t} \dot{\gamma} \right) \]
Three constants being the zero shear rate viscosity and two time constants \((\lambda_2\) is called the retardation time) are contained in the equation shown above representing the Jeffrey’s model.

The creep recovery curve [Malkin, 2006; Mainardi, 2011] represents the two phases of the creep-recovery experiment. The first phase refers to the creep phase where a constant stress is applied on each sample as determined from the stress sweep test and the creeping motion of the specimen is carefully monitored for a period of time. The second phase, being the recovery phase, represents the recovery/relaxation of the specimens after the removal of applied stress by applying the minimal possible stress as per the instrument’s limitation. The recovery curve suggests the amount of strain recovered in a period time. This demonstrates the leveling properties of a refractory coating and hence determines its aptness for desired coating process.

After determination of the requirement of the use of refractory coatings from the experimental results as obtained from the casting trials and solidification modeling, the corresponding refractory coating could be identified to impart either chilling or insulating properties or to improve surface smoothness of the core/mold interfaces. A rheology test for the identified/developed refractory coating formulations could be performed in order to determine the suitable coating method. The cured sand specimens can be coated with the application of identified refractory coating with the determined coating method and casting trials could be performed.
Problem Statement

Various major rapid casting techniques have proven to be beneficial during the development phase of a product to obtain a proof of concept, but there are some associated drawbacks for these processes.

Considering the limitations of rapid prototyping and manufacturing, also known as layered manufacturing processes, there has emerged a desire for the development of a hybrid manufacturing system [Boivie et al, 2011; Kerschbaumer and Ernst, 2004; Liou et al, 2006], which combines the advantages of high accuracy, while low build process time for conventional CNC machining with the one-setup process of CAD for rapid prototyping, RP. The hybrid RP systems [Hur et al., 2002] advances the build process from that of layered manufacturing to a next generation additive and subtractive layered manufacturing process, where the integration of CNC machine tooling [Xiong et al, 2008; Keshwamurthy et al, 2004; Liou et al, 2006], and layer deposition techniques provides more precision and accuracy while exponentially increasing production speeds.

The 3D printing techniques [Kawola, 2003; Gill et al., 2009], currently being implemented, have limitations in context of speed, safety and logistics. Because 3D printing involves the printing of a liquid phenolic binder to chemically bond sand particles layer-by-layer in patterns dictated by a CAD file, the thickness of the layers are restricted by the penetration of the binder into sand layer. This limitation increases the process cycle time, resulting in lower throughput. The printed layers must then be cured/conditioned for nearly 24 hours, which increases the process time for a build. As such, a 59 X 29 X 27 inches build envelope would take more than two days to complete.
The other limitations of this process include the safety, storage and transportation of the liquid binder system, which is considered to be highly flammable and hazardous.

The additive and subtractive layered manufacturing [Karunakaran, 2010] approach has found applications in various areas such as in the rapid pattern manufacturing of sand castings, which implies layer by layer manufacturing along-with the simultaneous machining [Frank et al., Iowa State University] using chemical wood, where the adhesion of wood slabs over one another refers to the additive approach and milling [Rooks, 2002; Knights, 2005], and patterning of each slab layer, thereafter corresponds to the implementation of a subtractive approach.

The additive and subtractive approach for creating sand casting molds, while maintaining the desired strength characteristics and accuracy in the replication of features, needs further investigation.

The sand specimen proposed for implementing this approach comprises of thermosetting resin [Okubo, 2007] coated carbon shell sand. The carbon shell sand is used to enhance the heat transfer between the top layer and bottom layer by its high heat absorption properties. Moreover, the sand particles are also easily machinable due to the lubrication properties of graphite, so the cured layers should be more readily machined, allowing for higher precision. However, the machinability [Hentschel et al., 2006; Wetzel, 2012], also depends on the grain size and grain structure so these properties must also be taken into consideration.

The proposed methodology, shown in the schematic (Figure 2.15), represents the step-by-step procedure to be followed in order to create the desired sand molds to obtain

An off-line layer deposition followed by off-line photonic curing [Aijazi et al, 2012] and machining approach [Cheah et al, 2005] could be implemented to achieve desired sand mold/core shapes and dimensions. The process cycle could be terminated upon attainment of desired shape and dimensions of the mold cavity. The optimum layer thickness, curing, and machining parameters required to achieve castability could thereby be determined.

The selection of the machining tool and light source should be governed by various inter-related parameters, such as desired layer thickness, depending on the

*Figure 2.15* Hybrid manufacturing approach-utilizing layer manufacturing with simultaneous machining.
mechanical bonding strength of material, interaction of cured layer with the cutting tool during machining, required cure temperature and cure time for each layer, etc.

Contributions of this Work to Forward the Science in the Field

The hybrid rapid casting technology, upon further development, could be easily adapted by almost every pattern-shop holding capabilities for precision machining. This new rapid casting technique could be employed in the industries such as the Automotive Industry (Eaton, Ford, GM, Chrysler, etc.), Entertainment (Formula one racing), Agriculture industry (John Deer, Caterpillar, Kubota, etc.), Construction industry (Caterpillar, United Tractors Co., Tex Corp., Komatsu, Kubota, etc.), Defense Industry (replacement parts and new weapon prototypes, etc.), Energy Industry (Wind turbine blades, hydraulic turbines, oil and gas pumps, etc.), Aeronautical Industry (Precision Cast-parts, Airbus, Boeing, GE, Rolls-Royce, etc.), Shipping Industry (Propeller blades, anchors, etc.), Medical and Biomedical Industry and others to fulfill their rapid prototyping and rapid casting needs.

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CHAPTER III

INTEGRATION OF CAD/CAM IN A HYBRID TECHNIQUE FOR RAPID MANUFACTURING APPLICATIONS

Summary

A novel hybrid technique, utilizing both additive and subtractive manufacturing techniques, has been demonstrated in this study. This technique finds applications in production of shaped cavities/molds for casted metallic parts for various rapid prototyping and rapid manufacturing application areas by integration of Computer Aided Design (CAD) and Computer Aided Manufacturing (CAM). This study reveals the advantages of integrating CAD/CAM with the hybrid techniques for achieving pattern-less molds to obtain various metal casted parts for a variety of applications. The major advantage of this application being high process throughput, which facilitates production of casted metallic parts from computer generated 3D models, through to finished parts, thus cutting overall process time as compared to other processes being currently available for various rapid prototyping applications.

This experimental work emphasizes producing a finished mold utilizing the hybrid technique to validate the viability of the process for various rapid manufacturing applications.
Introduction

Manufacturing [Grote and Antonsson, 2009] refers to the production of specially designed parts and structures with definite shapes identified by material and geometric characteristics. These parts and structures can be produced by means of the various processes involved in achieving the desired parameters such as, forming, cutting, joining, etc. With the introduction of machine tool automation [Bedworth et al, 1991] about six decades ago, followed by the automation revolution in the manufacturing industry, there now exist almost fully automated manufacturing plants and facilities in every part of the world. The epoch of this development started with the introduction of Computer Aided Design (CAD) offering the capabilities of generating pictorial representations. The designed parts can now be represented with the help of 3D models using various CAD modeling [Bedworth et al, 1991] software, providing the flexibility to incorporate modifications to the design and thorough analysis of prototypes prior to manufacturing. These 3D models can first be simulated based on performance analysis, using various simulations modeling software and the problem areas can easily be determined and improved, even before the initialization of the manufacturing process for the designed parts.

Development of CAD laid down some strong roots for the introduction of Computer Aided Manufacturing (CAM) [Bedworth et al, 1991; Chang et al, 1998], changing the face of industry by enabling the complete automation of the production lines. Building blocks of this automation process can be considered as the introduction of numerical control machines and industrial robots. Computer Numerical Control (CNC)
machines [Bedworth et al, 1991; Chang et al, 1998], provide the major advantage of controlling the machining operations with accurate tool positioning being accompanied by various computer-controlled commands. Developments of robots can be considered as being a natural extension to CNC machines by offering the flexibility of using a wide variety of interchangeable tools and larger build envelopes depending on the reach of robotic arms.

Three-dimensional printers [Bassoli et al, 2007] can also be considered as advanced CNC machines with the capability of moving the printer head in three different directions (XYZ). The accurate positioning of the printer head with the help of computer controlled commands and precise synchronization of the axial movements allow the selective deposition of materials. As a process that involves the layer-by-layer deposition [Kochan et al, 1999; Hochsmann, 2011] of selected material, it is termed additive manufacturing [Jang and Ma, 2002]. This type of manufacturing is being widely used in industry for various rapid prototyping applications [Bernard et al, 2003]. For casted metal prototypes [Ortiz et al, 2008], additive manufacturing offers the advantage of the ability to produce complex shapes by the selective deposition of a binder system on granular media [Kawola, 2003; Gill and Kaplas, 2009]. The granular media get cured and bind together the granular media only in the selective areas where the binder has been printed. This process continues layer by layer to attain the desired shaped cavity and upon termination of the process, the shaped cavity or casting mold is separated from the powdered granular media by vacuuming out the excess powdered material. The process provides the flexibility of building complex shapes directly from CAD models [Bernard
et al, 2003]. However, it simultaneously offers some limitations related to the maximum achievable layer thicknesses in a single pass, hence resulting in overall low process throughput.

Subtractive manufacturing [Basila et al, 2007] refers to the process of building a shaped cavity by selective removal of material using precision machining using computer-controlled machines. The integration of CAM allows machining the cavities with high precision [Hentschel et al, 2006; Wetzel, 2012] and defined dimensional tolerances using numerical control machines. The accurate tool positioning of CNC machines and the capabilities to use interchangeable tooling provide the advantages of achieving better dimensional tolerances. The high horizontal and vertical machining feed rates accelerate the machining process and result in high process throughput; however, the maximum feed rates also vary with the mechanical strength of the material used for creating the shaped cavities.

This study demonstrates a new hybrid rapid manufacturing technique [Karunakaran et al, 2010; Hur et al, 2002; Brensons, 2011], implicating the layered manufacturing process (additive manufacturing) followed by selective removal of material with assimilation of precision machining technologies (subtractive manufacturing). The integration of subtractive and additive manufacturing offers various advantages, such as high process throughput, ability to build complex shapes and undercuts, etc. This technology can be utilized in various rapid manufacturing applications such as for obtaining casted metallic replacement parts and prototypes for automotive, entertainment, agriculture and construction industries, etc.
Experimental

Methodology

The process flowchart, shown in Figure 3.1, represents the step-by-step procedure followed in order to create the desired sand molds to obtain functional prototypes and replacement parts, following the hybrid rapid manufacturing technique.

*Figure 3.1* Process flowchart showing the hybrid manufacturing technique.
Schematic of the Assembly

The schematic of the assembly, as shown in Figure 3.2, would be able to fulfill the requirements to build shell sand casting molds [Beandoin et al, 1997], using the hybrid rapid manufacturing technique by implementation of additive and subtractive layered manufacturing.

Figure 3.2 Schematic of the model for the inline machine assembly.
The schematic shows a movable build platform and a moving tool assembly, where the tool assembly is comprised of different tool head combinations. The XY directional movements of the tool assembly perform the layer build and tooling operations. The z-directional downward movement of the build platform governs the layer thickness, and the fixed sidewalls provide the closed build envelope for sand curing and machining.

The wall structure is comprised of four thin expandable metal walls around the build platform. The metal walls are comprised of heating elements to maintain the cured and machined layers at elevated temperature to accelerate curing of new layers of granular media. The z-directional downward movement of the platform controls the maximum fill volume of the sand on top of the build platform, hence controlling the required layer thicknesses. For obtaining the desired layer thickness of the sand (suppose 8 mm) on top of the build platform, a resin-coated sand in powder form is delivered and spread using a sand dispenser enclosed in the tool assembly. Thereafter, so as to obtain an even layer thickness, excess sand is removed using a sand-sweeping bar or blading system which rides on the top edge of the wall structure. The layer curing and patterning is performed with the help of the XY directional movements of a high intensity pulsed light source and machining tool head, as enclosed in the tool assembly. The components of the tool assembly arrangement for this layered manufacturing approach are shown in Figure 3.3:
The sand blading system comprises of a pneumatic blading system, as used in various gravure printing or coating processes. The pneumatic blading system is able to maintain blade contact and apply blade pressure only at the time when sand is dispensed from the sand dispenser. The light source is turned ‘ON’ in synchronization with the dispensing and blading of the sand. The movement of the blading system is restricted to only one direction (X dir.) and the dimensions of the screening blade remain the same as the Y directional dimensions of the wall structure. After obtaining a cured layer of sand of desired layer thickness (additive approach), the desired pattern is obtained by machining (subtractive approach). The cutting tool assembly for machining is mounted on a pneumatic/spring loaded mount platform with an arrangement such that the tool is able to establish contact with the sand layer only at the time of machining. The cutting tool assembly is comprised of an arrangement with z-directional movement capability to define the machining of the complex shapes by small step increments in depth.

The selection of the machining tool and light source is governed by various inter-related parameters, such as desired layer thickness, depending on the mechanical
bonding strength of material, interaction of cured layer with cutting tool during machining, required cure temperature and cure time for each layer, etc.

**Experimental Procedure**

An off-line layer deposition followed by off-line photonic curing and machining approach was implemented to achieve the desired sand mold. The sand specimen utilized for implementing this approach was comprised of a foundry grade thermosetting resin coated shell sand system [Beaudoin et al, 1997; Barlow and Veil, 2000]. The shell sand system used for this study comprised of resin-coated [Okubo et al, 2007] carbon sand. This specially engineered sand provides advantages such as enhanced heat transfer and ease of machinability over the conventional shell sand systems. A layer-by-layer deposition followed by simultaneous machining [Karunakaran et al, 2010] as shown in Figure 3.4 was utilized to achieve the shaped cavity.
Various physical and mechanical tests [Ramrattan et al, 1997; Ramrattan et al, 2013] were performed on the cured sand mold material to ensure that these parameters met the recommended benchmark to qualify for castability. Carbon shell sand particles offer the advantages of ease of machinability, due to the self-lubricating properties of the graphite. The cured layers can be easily machined with high precision; however, the machinability also depends on the grain size and grain structure of the sand. After performing mechanical strength analysis, the tool path on the CNC machine was generated utilizing the CAD model for the targeted proof of concept part shown in Figure 3.5

*Figure 3.4* Schematic representing the various sequential processes involved in the hybrid manufacturing technique [Ramrattan et al, 2013].
The targeted shape was comprised of some level of complexity by including curvilinear surfaces on the top half of the shown part. This part was selected to obtain a corresponding mold/cavity, as this mold produced using the hybrid technique demonstrates the capabilities of producing curvilinear shapes. After generating the machining tool path, the uniform layer-by-layer deposition of the powdered material was followed by the light curing and precision machining using the generated tool to attain the desired mold cavity.

*Figure 3.5 CAD model of the proof of concept part.*
Results and Discussion

A sand mold was produced using the hybrid manufacturing technique involving additive and subtractive manufacturing. The resin coated powder granular media were cured layer-by-layer using a light source. Performing the machining operation simultaneously with layer-by-layer deposition and curing enabled the curvilinear shaped cavity shown in Figure 3.6 to be achieved.

![Figure 3.6 Finished mold cavity for the proof of concept part.](image)

The sand mold produced using the hybrid technique was easily able to meet the castability benchmark by exhibiting the desired physical and mechanical characteristics [Bohra et al, 2014; ASM International, 2009]. The physical characteristics exhibited by the molds include good venting properties to subdue the defects originating from gas evolution and good abrasion resistance to minimize sand inclusion defects [American Foundrymen Association, 1947; British Foundrymen Society, 1963]. Sufficient mechanical strength was achieved by the sand mold to withstand head pressure exerted by the molten metal as well as the ability to accommodate the ease of shakeout.
The surface smoothness of the mold/cavity was found to be satisfactory, could be improved upon by the strategic placement of a ‘smart coating’ interface [Guyer et al, 2005; Guyer et al, 2006]. The application ‘smart coating’ at the mold-metal interface [Ramrattan et al, 2006; Ramrattan et al, 2008] also provides several other advantages apart from smoother casted surfaces, which reduce additional tooling costs during the finishing operations.

Conclusions

A sand mold or shaped cavity with defined level of complexity including curvilinear shapes and surfaces was successfully produced using the hybrid technique, involving additive and subtractive manufacturing with successful integration of CAD and CAM technologies. The process capabilities to attain complex shapes have been clearly demonstrated, which qualifies the process to be employed in various rapid prototyping and rapid manufacturing applications [Chhabra and Singh, 2011].

Acknowledgements

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CHAPTER IV
EVALUATION OF A 3D LIGHT CURED SAND FOR RAPID CASTING TECHNOLOGY

Summary

Development in rapid casting technologies has led to a new era in recent years by the inclusion of various 3D print manufacturing techniques. These techniques provide the flexibility and ease of reproducing a sand mold directly from CAD models, hence eliminating laborious pattern making steps, thus reducing the total time required for prototyping.

This study emphasizes on an alternate methodology of developing 3D cured sand molds by introducing a hybrid rapid prototyping approach [Hur et al, 2002; Boivie et al, 2011; Brensons et al, 2011]. Resin coated sand particles can be bonded layer-by-layer after being exposed to a light source, which raises the layer temperatures to a desired range for curing followed by precision machining to obtain complex shapes. Hybrid rapid prototyping techniques [Frank et al, 2009, Xiong et al., 2008; Kerschbaumer et al., 2004] have been employed in the past, however, the methodology of developing sand molds by integrating light curing with the use of the proposed light curing compatible materials for this process is new. The purpose of this study was to characterize the post cured molding materials with tests designed to measure the physical, mechanical, thermo-mechanical and chemical properties as required for castability.

This work represents the physical, mechanical, thermo-mechanical and chemical properties obtained for 3D light cured sand specimens with single 8 mm layers. Additional work could be done to alter the thicknesses in single pass to determine the
effect of various thicknesses that could be cured, while allowing the simultaneous machining of the mold. Machining to achieve desired patterns will enable the industry to identify optimum machining parameters, analyze molten metal-mold interactions and characterize the properties of metal castings obtained thereof.
Introduction

The additive and subtractive layered manufacturing [Karunakaran, 2010; Hochsmann et al., 2011; Jang et al., 2002] approach has found applications in various areas such as rapid pattern manufacturing for traditional sand castings [Keshavmurthy et al., 2004], which implies layer by layer manufacturing along-with simultaneous machining using wood. Adhesion of wood slabs over one another following this process refers to the additive approach and milling and patterning of each slab layer, thereafter, corresponds to the implementation of a subtractive approach.

Selective laser sintering has been applied in the shell-mold process to form sand casting molds using resin coated sand [Okubo et al., 2007; Barlow and Veil, 2000] and a blend of thermosetting sand. Thermoplastic resin coated sand has also been recommended to obtain the required strength characteristics by accurate patterning. The additive and subtractive approach for creating sand casting molds while maintaining desired strength characteristics and accuracy in the replication of features needs further investigation.

The sand specimen proposed for implementing this approach was comprised of a thermosetting resin coated carbon shell sand, to enhance the heat transfer between the top and bottom layers. Due to its high heat absorption, the carbon shell sand when exposed to the high intensity light enabled the sintering and curing of 8-mm thick layers in a single pass. The ability to cure thicker layers can lead to much higher throughputs for current 3D printing techniques, where the layer-by-layer deposition of sand-binder system is restricted to comparatively much lower layer thicknesses in a single pass.
The advantages of using a shell sand system with a thermosetting binder [Freedman, 1963; Carey et al, 1998] have been well known for several decades. But, almost 60% of sand mold applications in the U.S., the majority being in the automotive industry, utilize conventional (PUCB) sand systems. Carbon shell sand particles are easily machinable due to the self-lubricating properties of the graphite. The cured layers are also easily machined with high precision; however the machinability also depends on the grain size and grain structure of the sand.

Materials and Methods

The procedure consisted of 3 major steps:

1) Preparation of 50.0 mm diameter x 8.0 mm thick disc-shaped specimens.


3) Analysis and interpretation of results.

Note: All specimens were tested in the Metal Casting Laboratory at Western Michigan University (WMU) where ambient conditions were controlled at 22±1°C and relative humidity of 50±2%.

Preparation of 3D Cured Disc Shaped Specimen

The materials comprise of resin coated carbon shell sand and the properties are shown in Table 4.1.
An in-house laboratory scale assembly was built for preparing and curing specimens using an Infrared (IR) light source. A 50 mm diameter mold cavity was adjusted and filled with resin coated sand particles to achieve 8 mm thick disc specimens. The excess sand was screened off to obtain a uniform layer, which was exposed under the light source for 90 seconds.

![Diagram](image)

**Figure 4.1** Steps to produce 3D light cured specimens.

**Specimen Weight and Thickness**

The disc-shaped specimens were weighed prior to conducting any tests using a four place digital balance and the weights recorded. The thicknesses of the specimens were measured and recorded with a digital caliper. For testing of specimens on the basis of physical, mechanical, thermo-mechanical and chemical properties, a sample size of 15 specimens was selected, however the results reported in the study are based on an average of 5 specimens.

Table 4.1 Properties of sand

<table>
<thead>
<tr>
<th>Process</th>
<th>Sand Type</th>
<th>AFS/ GFN</th>
<th>Shape</th>
<th>% Resin</th>
<th>Roundness/ Sphericity (Krumbein)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D Light Cured</td>
<td>Carbon</td>
<td>64</td>
<td>Subangular</td>
<td>3%</td>
<td>0.5/0.7</td>
<td>7.4</td>
</tr>
</tbody>
</table>

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Permeability and MQI Testing

Permeability and MQI tests were performed to provide a measure of the specimen’s venting characteristics. The light cured shell carbon sand disc-shaped specimens were testing using a Gerosa Simpson permeability tester (Figure 4.2), Disa George Fisher Mold Quality Indicator (MQI) (Figure 4.3). A specimen holder designed and fabricated at WMU (Figure 4.4) was used to mount the specimens.

Permeability is a measure of gas flow through a porous media, such as a sand mold or core. It was calculated for each specimen by use of equation 4.1.

\[ P_{AFS} = \frac{V \times H}{P \times S \times T} \]

Equation 4.1

Where, \( P_{AFS} \): Permeability (cm\(^4\)/gm-min). \( P_{AFS}^{m^4/gm-s} = 1.66 \times 10^{-10} \) \( P_{AFS} \) (Refer to Chapter V for relationship of \( P_{AFS} \) with Darcy’s coefficient of permeability).

V: Percolated volume (ml)

H: Height of test sample (cm)

P: Air pressure (gm/cm\(^2\))

S: Area of sample (cm\(^2\))

T: Time in minutes

The permeability of a sand mold or core is affected by several factors including the size, shape, distribution, and method of compaction of the sand in the mold or core box. Furthermore, permeability is directly affected by the quantity of resin in the sand. Permeability testing is very common in the foundry industry and is part of the sand control tests performed on a regular basis at most foundries. A Gerosa Simpson
permeability tester (Figure 4.2) was used to perform the permeability tests conducted in this experiment. However, the specimen holder was designed and fabricated at WMU. A special rubber gasket was used between the specimen and the holder to provide a seal. Additionally, a plug was used to restrict the airflow in order for the Gerosa Simpson machine to detect the permeability of each specimen.

![Permeability tester with accessory attached.](image)

*Figure 4.2 Permeability tester with accessory attached.*

The Mold Quality Indicator (MQI) test, which is inversely related to permeability, was also studied. The MQI number is a measurement of the resulting backpressure developed from the resistance of airflow through a mold or core. The MQI unit (Figure 4.3) was equipped with an air pump, air tubing, and a rubber/foam contact head connected to the end of the tubing. An MQI unit is typically deployed somewhere along the molding line to perform real time measurements on the molds waiting to receive the molten metal. With some modifications to the original rubber contact head, this
instrument was utilized with the WMU specimen holder.

*Figure 4.3* MQI with accessory attached.

The specimen was secured into a holder (Figure 4.4), which was then fixed to the permeability tester. The test was then started and the permeability measured. The holder with the specimen was then removed and attached to the MQI unit for measurement.

*Figure 4.4* Specimen in gasket within holder assembly.
Disc Transverse Test

Disc transverse strength tests (DTS) were used to measure the strength of the sand specimens prior to the thermal distortion test (TDT), and after the TDT. The strength properties before TDT relate to the handling of the core/mold material after core/mold production, prior to pouring. The strengths after TDT testing relate to the shakeout/collapsibility characteristics. A sand strength machine (Dietert Model 490-A) equipped with a disc transverse accessory (Figure 4.5).

The disc-shaped specimen was fitted into a specimen holder on the testing machine and was supported on its ends. It was then subjected to a transverse force by applying the load with a 2.00 mm thick rounded edge blade across its diameter. Loading was performed at a constant linear load rate. A load-cell electronically sensed the specimen failure and digitally displayed the results. The maximum load to failure was recorded.

Figure 4.5 Images before (left) and after (right) DTS test.
Abrasion Test

Abrasion resistance defines the property of a material surface to resist wear while in contact with another material. The determination of the abrasion/wear resistance of a cured surface layer plays a vital role in the estimation of effect on sand mold surface due to handling procedures. This test method encompasses ability to compare strength for different sand specimens against scratch or wear caused by handling. Teledyne Standard Abrasion Tester Model 503 equipped with a custom sample holder for disc specimens (Figure 4.6).

![Figure 4.6 Abrasion tester.](image)

The 50 mm dia. x 8 mm thick disc-shaped specimens were weighed and secured onto the sample holder using four screws, one at each corner. The sample holder was mounted onto the abrasion tester with a ceramic bead pressing against the
specimen surface perpendicularly as shown in Figure 4.6. A desired load was applied onto the ceramic bead by mounting corresponding circular weights on top of the abrading assembly. The specimen was then rotated in clockwise direction maintaining a constant rotational speed for a desired number of cycles/rotations. To ensure the proper contact between the ceramic bead and sand specimen surface, a vacuum was applied continuously to pull any loose sand particles during the test run, Figure 4.7. Reweighing the specimen and calculating the weight loss or % weight loss then determined the abrasion/wear resistance of the specimen surface.

Figure 4.7 Top view of secured 3D light cured specimen.

\[ c = a - b \]

Equation 4.2

\[ \%c = \frac{c}{a} \times 100 \]

Equation 4.3

Where, 

- a: Initial weight (g) of specimen
- b: Final weight(g) of specimen after test
c: Total weight loss(g)

%c: %Weight loss

The sand specimens were tested for 10 cycles/rotations with a load of 250 g on the ceramic bead and thereafter calculating weight loss and %weight loss as defined in equations 4.2 and 4.3.

**Thermal Distortion Testing (TDT)**

To operate the TDT the temperature control was adjusted to 1000°C (1832°F) to represent the cast iron-sand mold interface. To simulate the force of molten metal to a 6 in. (15.24 cm) head height for cast iron with a density of 0.25 lb/in³ (6.92 g/cm³) providing a head pressure of 1.50psi (0.01MPa) (Head Height*Metal Density), actuator on the TDT was adjusted to a predetermined load of 331 g. The predetermined load was chosen for the test on the basis of the weight calculated to represent a 6 in. (15.248 cm) cast iron head height (Contact Area of TDT Hot Surface*Head Pressure), representing a head pressure typical of a medium sized iron casting.

The temperature at the hot surface was controlled using a K-type thermocouple and controlling, monitoring and plotting graphs of temperature/time versus distortion being performed by using an integrated computer peripheral and data acquisition system. The disc shaped specimen was mounted onto a pivoting holder (as shown in Figure 4.8) and the specimen was automatically raised to achieve a symmetrical contact with the 2.00 cm (0.787 in.) dia. hot surface. A linear voltage displacement transducer (LVDT) was engaged at this point, which simultaneously engaged a laser to measure the distortion in longitudinal and radial directions. The distortions versus time/temperature curves
were generated using the integrated data acquisition system. The thermal distortion tests were performed over a 90 second interval, being based upon the recommendations from a committee of foundry experts.

For the longitudinal distortion it is possible to differentiate between expansion (\(D_E\)) and plastic distortion (\(D_P\)) separately from the thermal distortion curve (TDC). In this investigation, the authors chose to record the total distortion (\(T_D\)) and simply state \(T_D = \Sigma D_E + \Sigma D_P\). Further, the distortion radial (\(D_R\)) indicating expansion was monitored using a high-speed laser micrometer-scanning sensor (resolution of 0.05\(\mu\)m). Detailed procedure for the TDT has been defined in several AFS transactions [Oman et al, 2013; Iyer et al, 2001; Ramrattan et al, 1997; Guyer et al, 2005].

*Figure 4.8* Thermal distortion tester.

---

80
Prior to TDT, each specimen was weighed. Following TDT the surface of the specimen was blown with 20psi (0.14MPa) air pressure to remove any loose sand grains. The specimens were then again weighed, and the percent change in mass was recorded. Following weighing, the specimens were visually examined looking for signs of thermally induced cracking of the surface, loss of sand where contact was made with the hot surface, and any other discolorations or visual changes. If the core/mold media breaks down, this may be indicative of the tendency to produce cuts and washes, erosion/inclusion type defects. In interpreting these data, it is critical to identify the components causing the change in mass. The percent change in mass was calculated based upon the weight before and after as a percent of the weight before.

Gas Generation and Analysis

Various gaseous products generated at mold-metal interface were identified by the help of experiments being conducted using a microscale pyrolysis unit, CDS Pyroprobe 5250 (CDS Analytical Inc, Oxford, PA) interfaced to a Shimadzu QP-5050A gas chromatograph/mass spectrometer (Shimadzu Corp, Columbia, MD). Approximately 0.5 mg of sample was sheared off from the light cured disc shaped specimen sample and packed between quartz wool in a quartz tube with a filler rod. Pyrolysis proceeded by setting the pyroprobe at 1200°C with a hold time of 10s at the maximum heating rate set point of 999 °C/s. The GC used an Agilent CP-Porabond Q column; 25mm x 25mm with a 3 µm film thickness. The column gas flow was 1 cm/s with a split ratio of 1:100 so as to not overwhelm the mass spectrometer. The GC oven temperature program began with a 1 minute hold at 40°C followed by heating at 8°C/min to 300°C. The injector and
Detector temperature was set at 300°C. The mass spectra were recorded in electron ionization mode for m/z 14 to 200. Comparing the mass spectra of the peaks with standard spectra of other compounds using the NIST library to obtain the most probable identification matches of compounds. Pure compounds (Sigma-Aldrich Co., St Louis, MO) were then used to confirm the peak identities based on matching of retention times and mass spectra.

**Casting Trial**

For this experiment a green sand mold was developed to deliver molten metal to six chemically bonded disc shaped specimens simultaneously. Certain discs shaped specimens experience an external tangential point load (400 g) replicating an addition to 18-inch aluminum head pressure, when the mold is poured to a metallostatic head wetting all surfaces of the specimens (Figure 4.9). The purpose of this model is to identify the effect of super heat, metallostatic head and external load on chemically bonded disc shaped specimens to determine at what conditions distortion, penetration and/or veining occurs.

*Figure 4.9 Thermo-mechanical point loading on specimen.*
The mold making procedure consisted of the following steps: Green sand cope and drag mold halves were fabricated according to an experimental pattern. The drag mold with six specimens set on core-prints is shown in Figure 4.10. Two disc-shaped carbon shell specimens were uncoated (identified as no. 1 and 2 in the open mold), two were coated with an insulation coating (Diatomaceous Earth) identified as no. 3 and 4 in final casting) and two were coated with a chill coating (Zircon) (identified as no. 5 and 6 in final casting) (see Table 3.1 for details on carbon sand). The two coatings were applied to specimens 3-6, using brush coating. A six-inch tall pouring sleeve was affixed to the cope as a sprue to deliver the metallostatic head where one of each specimen type received a point load (Figure 4.11).

*Figure 4.10 Drag mold with specimens.*
The molds contained six symmetrical cavities and were poured where the chemically bonded disc shaped specimens were arranged by material and point loading sequence. This approach allowed for possible variation in casting surface quality (specimen/metal interface) to be assigned to the chemically bonded sand specimens. The sand-to-metal ratio for all molds was 4:1.

Molds were manually poured and the aluminum was (average pouring time = 10 sec., temperature at pour 732°C (1350°F)) delivered through a pouring sleeve fitted with a foam filter. All molds were produced at a 6-inch (15 cm) head-height.

Castings were allowed to solidify prior to cooling and shakeout. The casting
was sectioned at the core/metal interface and images for the casted mold/metal interface were captured using an ImageXpert image analyzer for visual analysis of the surface defects.

Results and Discussion

Specimen Weight

The weight for each disc shaped specimen was recorded prior to conducting further testing to keep track of any variability in each sample set.

Table 4.2 Average weight of disc-shaped specimens

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Weight</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D Light Cured</td>
<td>18.35</td>
<td>1.13</td>
</tr>
</tbody>
</table>

Permeability and MQI Testing

Higher mold and core venting (permeability) is desirable in the foundry industry, since some gas related casting defects can be averted. However, the venting characteristics of the chemically bonded molds and cores are difficult to measure. An inverse relation between the permeability and MQI has long been established in the green sand foundry industry. So, the purpose here was to investigate how these two tests track the venting of chemically bonded sand.

3D light cured disc shaped specimens tested using the custom-made specimen holder and plug had a permeability number of 159±3 and MQI of 170±4. It is important to note that the specimen holder and plug has a permeability number of 221±3 with a flow rate of 274 cc/sec. The permeability and MQI data collected for the 3D cured
specimens are shown in Table 4.3.

Table 4.3 Permeability and MQI data for disc shaped sand specimen

<table>
<thead>
<tr>
<th>Specimen</th>
<th>3D Cured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Permeability</td>
<td>159±3</td>
</tr>
<tr>
<td>MQI</td>
<td>170±4</td>
</tr>
</tbody>
</table>

**Abrasion**

The abrasion test is an indicator of friability and hence determines the shelf life, handling and storage parameters of molding materials. The results obtained from abrasion test conducted for the various disc shaped specimens are provided in Table 4.4.

Table 4.4 Abrasion losses for disc-shaped specimen

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Weight Loss</th>
<th>% Weight Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D Light Cured</td>
<td>0.56 g</td>
<td>3.07</td>
</tr>
</tbody>
</table>

**Disc Transverse Strength (DTS)**

The DTS represents the mold strength of a material, as well as shakeout parameters. Higher strengths are desired for better handling and storage of the molds, while on the other hand, very high mold strengths make the separation of the mold cavity with casting upon solidification challenging. The disc transverse strength results for the specimens are shown in Table 4.5.
Thermal Distortion Testing (TDT)

The sand system was tested at elevated temperature over a 90 second interval; TDT curves and picture information related to the system are presented in Table 4.6 and Figure 4.12 and 4.13 respectively.

The TDT curves for all three systems showed undulations that indicate thermo-mechanical and thermo-chemical changes in these systems at elevated temperature. The longitudinal distortion curves showed an initial expansion (upward movement of TDC) before plastic deformation (downward movement of TDC). Note that TDT curves for longitudinal distortion and radial distortion depicts an average for 5 specimens tested (Figure 4.12). The 3D light cured specimens experienced longitudinal plastic deformation for approximately 90 seconds and radial expansion for approximately 55 seconds.

There are many heat induced thermo-chemical reactions occurring in all three sets of specimens as evident from the hairline surface cracks found on tested specimens and percent change in mass values (Table 4.6). Expansion cracks were macroscopically evident on all specimens tested. Significant mass losses were not evident with 3D light cured specimens.

All specimens tested remained intact and showed minimal change in mass after TDT and after the application of the 20 psi (0.14 MPa) air pressure (Figure 4.13). Every

<table>
<thead>
<tr>
<th>Specimen</th>
<th>DTS (lb)</th>
<th>psi</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D Light Cured</td>
<td>14.28±1.5</td>
<td>92.12±9.67</td>
</tr>
</tbody>
</table>

Table 4.5 Disc transverse strength for disc-shaped specimen
specimen tested had minimal losses of sand and possessed cracks that increased in size with the duration of the test. Observations from the heat-affected zone on the surface of tested specimens revealed that all the specimens had visible minimal sand losses and crack propagation. The hot surface/specimen interface generally shows a crater with discoloration due to binder degradation, the discoloration was not present on the opposite side of the 3D light cured specimens. In addition, sand binder losses were not evident at the hot surface/specimen interface where binder bridges pyrolyzed and sand grains broken loose.

Table 4.6 Physical and thermo-mechanical properties of the disc specimens

<table>
<thead>
<tr>
<th></th>
<th>Results of Thermal Distortion Testing @ 331 g for 90 sec.</th>
<th>Obs. During Elevated Temp. Testing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Type</td>
<td>Blow Pressure (psi)</td>
</tr>
<tr>
<td>3D Light Cured</td>
<td>N/A</td>
<td>3.0</td>
</tr>
</tbody>
</table>
As compared to conventional silica or PUCB sands, the carbon shell sand behaves differently as observed during the TDT. It can be inferred from the TDT observations for the carbon shell sand that the radial distortion ($D_R$) decreased significantly, on the other hand an increase in longitudinal distortion was also observed. The heat transfer to the backside of the specimens (as shown in Figure 4.14) at elevated temperature (1000°C)
did not affect the backside temperature as much as observed in conventional silica sand systems [Oman et al, 2013], suggesting the carbon shell sand systems acting as a ‘chill’ material. Due to this observed characteristic, much heat is not being transferred radially; hence decreasing the overall radial distortion implying confined expansion/contraction of the sand binder system.

**Figure 4.14** Temperature versus time plots between the hot surface and back of specimens.

**Gas Evolution Test**

By conducting the gas evolution test using a GCMS, various compounds are detected in the gaseous products evolved as being represented by the corresponding pyrogram (Figure 4.15), when the light cured specimens were exposed to high temperatures as per mentioned in test procedure.
The gaseous compounds as detected were mainly comprised of the compounds from the phenol family and the gaseous byproducts evolved as a result of the combustion of phenol-based compounds. The presence of phenol-based compounds in the gases evolved from the light cured specimen can be justified as having originated from the combustion reaction of the phenol-based thermosetting binder system.

Observations from Casting Trial

It is important to reiterate that the 8 mm disc-shaped specimen cores used for casting were poured from a 6 inch (15 cm) head height at 732°C (1350°F) while TDT was conducted at 1000°C (1832°F) using 6 inch (15 cm) of head. In the casting trial all specimens support the thermo-mechanical stresses involved in filling and point loading through solidification. However, there was macroscopic distortion observed at the mold/metal interface. The refractory coating investigated did not prevent distortion, but did improve the interfacial as-cast surface. It should be pointed out that the refractory
coatings were brushed-on and striation marks from the coating application were apparent on the interfacial surfaces as well.

Observations were made from the disc-shaped specimen core/metal interface surface of the casting (Figure 4.16). Macroscopic observations revealed the two coated core/metal interfaces were comparatively clean and possessed minimal casting defects regardless of the load applied. The uncoated 3D light cured carbon shell sand system had the greatest amount of specimen-metal interfacial defects (penetration and veining); this was augmented with point loading. The coated 3D light cured carbon shell sand specimen-metal interfacial was reasonably clean, but did suffer penetration with load. The authors recognize the thermo-chemical reaction for carbon sand and aluminum, and thus the incompatibility. The use of 3D light cured carbon shell sand for the aluminum prototypes applications will require the use of refractory coatings at the mold and core-metal interface.
<table>
<thead>
<tr>
<th>Specimen</th>
<th>400g Load</th>
<th>No Load</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uncoated</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
</tr>
<tr>
<td>Insulation Coated (Diatomaceous Earth)</td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
</tr>
<tr>
<td>Chill Coating (Zircon)</td>
<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
</tr>
</tbody>
</table>

*Figure 4.16 Images showing core/metal interface.*
Conclusions

This paper has shown that there is a difference in both room and elevated temperature behavior for the sand binder system studied. The 3D light cured carbon shell sand gained strength in a manner similar to thermosetting resins.

The approach was to develop a 3D light cured shell mold with low sand to metal ratio where these elevated temperature strength properties are beneficial.

3D light cured carbon shell sands possess the physical and mechanical properties suitable for core and mold development also at room temperature. The 3D light cured carbon sands are thermally stable. However, carbon shell sand is not compatible with some molten metal chemistries; this was revealed in the casting trial.

A longer shelf life of carbon shell sand considering the longer shelf life possessed by shell sand systems, ease of machinability and safety and logistical advantages can be considered as add-on for the suitability of the molding material for applications in rapid casting.

When 3D light cured carbon sands are used for a rapid casting process with alloys such as aluminum, cast iron, etc., a suitable refractory coating interface is required. It is therefore recommended that casting trials with refractory coatings applied to 3D light cured carbon shell sand molds and cores be studied in detail for better castability.

Proof-of-concept casting with moderate complexity level was successfully obtained through casting trials conducted at WMU. This shows the capability of the process to produce castings with moderate complexity, however, the process capabilities
to produce higher complexity castings with internal geometry features is under investigation.

Acknowledgements

The authors gratefully acknowledge Glenn Hall, Matt Stoops, and Peter Thannhauser, WMU and Brian Guyer, H.A. International, for their technical support. Thanks to Mr. Kelley Kernes, Fairmount Minerals for supplying the granular media used in this study.

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CHAPTER V

LAYERED MANUFACTURING UTILIZING NEW LIGHT CURED MEDIA FOR CAST PROTOTYPES

Summary

Continuously increasing rapid casting demands in the production of functional or test prototypes have stirred the recent developments reported in this work. These technological advancements help to reduce the time till casting by introducing new techniques to produce pattern-less molds. Three-dimensional (3D) printing [Hochsmann, 2011] offers the flexibility and ease of production of sand molds directly from CAD models. The new technology offers the advantages of minimal processing steps and higher precision; it also provides the capabilities to readily produce complex shaped sand molds. However, wide industrial use of this technology has been suppressed by some limitations and concerns associated with this technology such as low process throughput, environmental impact concerns, and increased logistic costs. An alternate methodology of developing 3D cured sand molds has been proposed in this study through the introduction of a hybrid rapid prototyping [Hur et al., 2002; Brensons et al., 2011] approach to overcome these limitations. The resin coated sand particles are bonded layer-by-layer after being exposed to a high intensity pulsed light source, to enable complex shapes to be obtained by precision machining [Karunakaran et al., 2010; Basila et al., 2007; Hentschel et al., 2006] which follows the curing cycle. Although hybrid rapid prototyping techniques have been employed in the past, the methodology of developing sand molds through the integration of light curing and granular media is new. The purpose of this study was to evaluate the physical, mechanical and thermo-mechanical properties of
single and multiple layers using the cured molding media for castability. This study draws the comparison between single and multiple layers of cured sand in terms of physical, mechanical and thermo-mechanical properties possessed.
Introduction

Manufacturing technology [Grote and Antonsson, 2009] refers to the processes involved in the production of structures with definite shapes identified by material and geometric characteristics. Various processes are involved in achieving the desired parameters, which can be classified into different groups, forming, cutting, joining, etc.

Casting can be considered the backbone of most shaping processes for various applications where metalcasting is a key component. Metal casting [Schleg et al, 2008] can be explained as the process of pouring molten metal into a shaped cavity in order to transform it into a desired shape upon solidification. The primary step of this process involves the creation of a shaped cavity, also known as a mold. A mold can be prepared using various heat resistant materials such as sand, which provides the primary advantage of cost effectiveness. The molding processes can be classified into permanent and non-permanent where the mold is destroyed to remove the casted metal structure (e.g. sand molds). This step is referred to as the shakeout process. The casting process offers many advantages over other primary shaping processes, such as freedom of castability over a wide range of metals of desired shapes and versatile mechanical properties.

Achieving net near shape and integral castings leads to the reduction in the time and costs associated with any additional machining and assembly requirements. Altogether, the advantages of the process include savings in material and energy use along-with recycling and ecological benefits.

The selection of the mold material and mold making process plays a vital role in governing the casting characteristics [ASM International, 2009]. Sand, being a refractory
material, can withstand very high metal pouring temperatures, which imparts the required
dimensional stability at elevated temperatures. Sand also provides an economical
advantage over other comparable refractory materials. Dry sand molding requires the
introduction of a binder system to hold the loose sand particles during the mold shaping
process. The finished mold’s physical, chemical and thermo-chemical properties depend
on the sand-binder system [Carey et al, 1998] interactions and the process of creating the
mold as well.

Additive manufacturing [Beaudoin, 1997; Jang and Ma, 1997], also known as
layered manufacturing [Hochsmann et al., 2006], has attained acceptance in rapid
prototyping [Kochan et al., 1999; Cheah et al., 2005] and rapid manufacturing [Wetzel,
impacting additional opportunities for attaining complex shapes with higher precision and
tolerance levels as compared to conventional molding techniques. Rapid prototyping
[Ortiz et al, 2008] refers to the production of prototypes of actual design. It is used for the
product development phase of a production process because it is able to impart the
characteristics in close proximity to the finished product, which enables the further
investigation and analysis of the product before finalizing the end product features. The
rapid casting [Chhabra and Singh, 2011] process defines the integration of traditional
metal casting techniques with additive manufacturing approaches to achieve either
functional prototypes or end products.

Various rapid casting solutions have been developed during the last few decades
to implement concurrent engineering approaches for the development of functional
prototypes and customized production of metal castings for applications in various sectors of manufacturing industries. These rapid casting solutions include unique production approaches, such as selective laser sintering, fused deposition modeling, stereolithography, 3-D printing and rapid tooling [Levy et al, 2003; Gill and Kaplas, 2009; Barlow and Veil, 2000; Kruth et al, 2007; Kawola, 2003].

Experimental Procedure

The procedure consisted of 4 major steps:

1) Characterization of granular media.

2) Preparation of 50.0 mm diameter x 8.0 mm thick disc-shaped specimens with variable layer thicknesses.

3) Testing of the various properties of the specimen.

Characterization of Granular Media

Resin coated [Okubo et al, 2007] shell sand with 3% binder loading will be evaluated and characterized for density, Grain Fineness Number, pH, shape and sphericity or roundness (Refer to Table 5.1).

The bulk density of sand plays a vital role in the estimation of the finished sand mold properties from a handling and logistical prospective. Bulk density is also significant in tracking any lot-to-lot variations in the sand systems employed. The density of a system is representative of mass/volume ratio and is measured by weighing a known volume of material and thereafter calculating the ratio.

The Grain Fineness Number [Schleg et al, 2008] is indicative of how small or
large is the average grain size of a sample of sand. Though smaller grains of sand are capable of imparting better surface finish, due to their closely packed structure, they hinder the venting characteristics of the mold, which can result in various casting defects. The higher the GFN, the smaller the sand grain size. Very low GFN refers to larger sand grain sizes compromising the packing efficiency consisting of larger interstices between the grains and hence resulting in rough casting surfaces. To calculate the GFN, a known sample weight was placed into an arrangement of sieves, which was then placed in a sieve-testing device with continuous sieve shaking for 15 minutes. Thereafter, the weight of sand retained in each sieve and pan was recorded followed by calculating GFN as shown in Table 5.2:

Table 5.1 Properties of resin coated sand

<table>
<thead>
<tr>
<th>Shape</th>
<th>Roundness/Sphericity (Krumbein)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sub-angular</td>
<td>0.5/0.7</td>
<td>7.4</td>
</tr>
</tbody>
</table>

Table 5.2 AFS GFN calculations for the resin coated sand

<table>
<thead>
<tr>
<th>Sieve</th>
<th>Amount Retained</th>
<th>%</th>
<th>Multiplier</th>
<th>Product</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>0</td>
<td>0</td>
<td>0.03</td>
<td>0</td>
</tr>
<tr>
<td>12</td>
<td>0</td>
<td>0</td>
<td>0.05</td>
<td>0</td>
</tr>
<tr>
<td>20</td>
<td>0.01</td>
<td>0.01</td>
<td>0.1</td>
<td>0.00</td>
</tr>
<tr>
<td>30</td>
<td>1.94</td>
<td>1.95</td>
<td>0.2</td>
<td>0.39</td>
</tr>
<tr>
<td>40</td>
<td>3.99</td>
<td>4.01</td>
<td>0.3</td>
<td>1.20</td>
</tr>
<tr>
<td>50</td>
<td>15.64</td>
<td>15.71</td>
<td>0.4</td>
<td>6.28</td>
</tr>
<tr>
<td>70</td>
<td>26.14</td>
<td>26.26</td>
<td>0.5</td>
<td>13.13</td>
</tr>
<tr>
<td>100</td>
<td>33.08</td>
<td>33.23</td>
<td>0.7</td>
<td>23.26</td>
</tr>
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</table>
### Table 5.2 - continued

<table>
<thead>
<tr>
<th>Sieve</th>
<th>Amount Retained</th>
<th>%</th>
<th>Multiplier</th>
<th>Product</th>
</tr>
</thead>
<tbody>
<tr>
<td>140</td>
<td>16.01</td>
<td>16.08</td>
<td>1</td>
<td>16.08</td>
</tr>
<tr>
<td>200</td>
<td>2.57</td>
<td>2.58</td>
<td>1.4</td>
<td>3.62</td>
</tr>
<tr>
<td>270</td>
<td>0.17</td>
<td>0.17</td>
<td>2</td>
<td>0.33</td>
</tr>
<tr>
<td>PAN</td>
<td>0</td>
<td>0.00</td>
<td>3</td>
<td>0.00</td>
</tr>
<tr>
<td></td>
<td>Total = 99.54</td>
<td></td>
<td></td>
<td>AFS GFN = 64.30</td>
</tr>
</tbody>
</table>

#### Preparation of Light Cured Disc-Shaped Specimen

A laboratory scale assembly designed at Western Michigan University and integrated with the light source was used for preparing specimens. A 50 mm diameter mold cavity was adjusted and filled with resin coated sand particles to achieve 8 mm-thick disc specimens. The excess sand was screened off to achieve a uniform layer and exposed under the light source (Xenon Sinteron 2000 photonic light curing) for 90 sec. duration. For 2-pass 8mm thick specimens, the mold cavity was adjusted to 4 mm thickness, filled and exposed for 45 sec. Thereafter, the first layer was dropped down to 4 mm from the mold reference point so as to again obtain the 4 mm thick cavity and was filled and exposed with the same procedure as followed for the first layer.
Physical Properties

The thicknesses of the specimens were measured and recorded. Disc-shaped specimens were weighed prior to conducting any tests using a digital balance and the thicknesses were measured using a digital caliper.

The porosity of a material can be defined, as the measure of total void volume present, as such is dimensionless. Darcy’s Equation [Pal et al, 2006] defines the relationship between permeability and flow rate as shown in Equation 5.1:

\[ Q = \frac{k A \Delta P}{\eta L} \]

Equation 5.1
Where, \( k \) = Permeability Coefficient (Area)

\[ A = \text{Area of cross-section} \]

\[ L = \text{Thickness of Material} \]

\[ \Delta P = \text{Pressure drop} \]

\[ Q = \text{Flow rate (Volume/s)} \]

\[ \eta = \text{Viscosity} \]

AFS Permeability is a measure of gas flow through a porous medium, such as a sand mold or core. The relationship between AFS Permeability and Darcy’s coefficient of permeability has been defined in Equation 5.2 [Starobin, 2012]:

\[
k = \frac{\mu_{\text{air,RT}}}{g} P_{\text{AFS}}' \times 10^3
\]

Equation 5.2

Where, \( k \) being expressed in \( m^2 \), \( P_{\text{AFS}}' \) in \( m^4/gm\text{-sec} \) (Refer Chapter IV), \( \mu \) in Pa-sec or kg/sec-m and \( g \) in m/s\(^2\).

Permeability and MQI tests were performed to study the venting characteristics of specimens as good venting parameters play a major role in elimination of casting defects originating due to gas evolution at the mold-metal interface. A Gerosa Simpson permeability tester was used to measure AFS permeability. MQI was measured using Disa George Fisher Mold Quality Indicator.

**Mechanical Properties**

Disc transverse strength is a measure of transverse tensile strength of bonded sand particles. The disc transverse strength is used in the foundry industry to study the
parameters required for mold or core against transverse tensile stresses acting on mold-metal interface being resultant of the hydrostatic pressure. Therefore, a disc transverse strength test (DTS) was used to measure the strength of the disc shaped sand specimens using a disc transverse testing machine (Dietert Model 490-A) and was supported on its ends. During the test run, the specimen is subjected to a linearly increasing load and the maximum load to failure is then determined.

*Figure 5.2 DTS tester.*

**Thermo-mechanical Properties**

Thermal Distortion Testing (TDT) [Ramrattan et al, 1997; Iyer et al, 2001; Rebros et al, 2007] was used in this experiment to expose 50 mm diameter, 8 mm-thick disc specimens to 1000°C for up to 90 seconds. The purpose of this investigation was to test the 3D cured sand system used for rapid prototyping manufacturing. The TDT method was used for application on foundry mold and core media, and to further compare the data.
The longitudinal distortion of specimens can be differentiated vide expansion ($D_E$) and plastic distortion ($D_P$), considering the observations from the thermal distortion curve. The total longitudinal distortion ($T_D$) can be represented as the sum of expansion and plastic distortion components ($T_D = D_E + D_P$). The radial distortion ($D_R$), indicating expansion in the radial direction, will be studied further using a next generation thermal distortion tester (TDTng) developed at WMU.

Each disc shaped specimen was weighed prior to the thermal distortion testing and was blown with 20 psi (0.14 MPa) air pressure to remove any loose sand grains at the completion of the distortion testing. The change in mass of the specimens before and after thermal distortion testing was recorded.

**Casting Trial**

For conducting casting trials, so as to evaluate the castability of molding media, a 56 mm thick and 50 mm diameter disc shaped specimen was prepared using the layer-by-layer approach with each layer thickness being 8 mm. Two holes with approximately 13 mm diameter were drilled into the specimen and refractory coating [Guyer et al, 2006] was thereafter applied on one using the dip coating method to analyze the effect of refractory coating application on castability. Aluminum was thereafter melted in a small crucible and poured at a pouring temperature of 760°C. A manual shakeout procedure was followed after the solidification of the casting to separate the metalcasting from the mold media.
Results and Discussion

The weight for each disc shaped specimen was recorded prior to conducting further testing to keep track of any variability in each sample set.

Table 5.3 Average weight of disc shaped specimens

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Weight (g)</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D Cured 1-Layer</td>
<td>16.73</td>
<td>1.07</td>
</tr>
<tr>
<td>3D Cured 2-Layers</td>
<td>15.95</td>
<td>1.02</td>
</tr>
</tbody>
</table>

Light cured single pass 8 mm thick disc specimens, tested using the custom made specimen holder and plug, had a permeability number of 157±3 and MQI of 170±3; while 2-pass 8mm thickness specimens had a permeability number of 149±5 and 173±3 respectively (Figure 5.3). An inverse relationship between permeability and MQI has already been established in the foundries for other sand systems such as green sand. The results from permeability and MQI tests suggest that the proposed sand binder system also follows an inverse relationship trend (Refer Figure 5.3)
The 3D cured disc-shaped specimens prepared with 1-Layer and 2-Layers were tested at elevated temperature over a 90 second interval for thermal distortion testing. The TDT curves for both specimens (Figure 5.4) showed thermo-mechanical and thermo-chemical changes in these systems at the elevated temperature. The longitudinal distortion curves showed expansion (Dₑ) followed by plastic deformation (Dₚ). The TDT curves for longitudinal distortion depict an average for 3 specimens.

The 3D light cured 1-layer specimens expanded for approximately 46 seconds followed by plastic deformation for approximately 44 seconds whereas 3D light cured 2-layer specimens expanded for ~48 seconds followed by plastic deformation for the remaining ~ 42 seconds of the test. Heat induced thermo-chemical reactions [Guyer et al., 2005] occurring in both sets of specimens were evident from the surface cracks found on

Figure 5.3 MQI and Permeability data for disc shaped specimens.
tested specimens and percent change in mass values (Table 5.4).

Table 5.4 Thermo-mechanical properties of the disc shaped specimens

<table>
<thead>
<tr>
<th>Sample</th>
<th>Blow Pressure (psi)</th>
<th>( (D_p) ) Longitudinal (mm)</th>
<th>( (D_p) ) Longitudinal (mm)</th>
<th>( (T_D) ) Total Dist. (mm)</th>
<th>% Change in Mass</th>
<th>Cracks % Fractures</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-Layer</td>
<td>20</td>
<td>0.049</td>
<td>0.041</td>
<td>0.090</td>
<td>0.64</td>
<td>present</td>
</tr>
<tr>
<td>2-Layer</td>
<td>20</td>
<td>0.028</td>
<td>0.020</td>
<td>0.048</td>
<td>0.64</td>
<td>present</td>
</tr>
</tbody>
</table>

*Figure 5.4* TDT curves for 3D Light cured sand specimens.

The observations from the casting trial (*Figure 5.5*) show the evidence of presence of surface casting defects [American Foundrymen’s Association, 1947; Institute of British Foundrymen, 1963], such as penetration with the absence of the refractory coating applications. The casted metal surface obtained with the application of refractory coating [Guyer et al., 2006] imparted better surface finish and did not incorporate any surface defects subject to penetration and veining.
Figure 5.5 Coated and uncoated mold cross-sections is shown on the left and metal casting obtained from corresponding surface is shown on the right.

Conclusions

The observations from the experimental analysis suggest that the proposed media for the light cured system possess the desired physical, mechanical and thermo-mechanical properties for castability. These light cured sand specimens are quite stable at elevated temperatures, as revealed from the thermal distorting testing. Since, the proposed sand binder system may not be compatible with certain casting alloy chemistries; a refractory coating interface may be required.

Acknowledgements

The authors gratefully acknowledge Glenn Hall, Matt Stoops (Western Michigan University), and Xenon Corporation for their technical support.

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CHAPTER VI

ANALYSIS OF THERMAL CURE CHARACTERISTICS FOR CHEMICALLY BONDED SHELL SAND SYSTEMS

Summary

Shell molding technologies have been widely used by the foundry industry over the past decades for the production of casted metal parts; for a variety of application areas. These technologies are preferred due to various advantages; such low molding costs while imparting higher strength characteristics; dimensional stability and high surface smoothness on casted surfaces. These characteristics of shell molding technologies lead to attainment of near net shaped castings, minimizing the time and costs involved in secondary machining operations to attain the desired shape and dimensions.

Thermo-setting binder systems are most widely used for shell molding technologies. The resin-coated shell sand system, when being exposed to elevated temperatures forms a bonded cured surface, which possesses the required strength characteristics. However, in the event of being exposed to the elevated temperatures in excess of the cure temperature, there lies a negative impact on the strength parameters.

In current industry practices, the level of cure of a shell sand system is determined by visual comparison of the cured shell sand to the developed shell sand conundrum. This induces variability due to the human factor involved and the comparison is limited only to a qualitative analysis. This study suggests a methodology to identify the optimum cure parameters and demonstrates the use of color measurement technologies; for quantitative analysis of cure levels by making the comparisons using the measured color values.
Introduction

Over the decades shell molding and core-making has been predominantly used for foundry applications due to various perceived advantages such as higher shell/mold/core strengths [Andrews L. S. R, 1963], higher level of dimensional stability and precision [McIntyre S., 2008], high surface smoothness for better castability, etc.; which allows attainment of near net shape castings. A variety of thermoplastic or thermosetting binder systems have been widely used in the shell molding process. The properties of these binder systems such as melting point, cure rate, degree of cross-linking, shelf-life, etc. can be altered by making adjustments to the amount of various additives added to the formulation; to impart desired binder characteristics.

The binder systems [Andrews L. S. R, 1963; Kagaya et al., 1992; Qureshi, P. S., 2001; Waitkus et al., 2003; Gupta et al., 1988; Fink et al., 2005, Companer et al. 2009] used in the shell molding process comprises of a heat-sensitive binder that attains the bonding strength when being exposed to elevated temperatures. The sand system comprises of the binder coated free-flowing particles of sand in dry form, where a dried layer of binder surrounds each particle. Upon exposure to elevated temperature, due to the induced thermal effects, the binder softens and flows in-between these particles. Upon cooling the binder solidifies and hence creates a bond between these particles of sand.

Thermoplastic [Andrews L. S. R, 1963] binder systems possess solid state at the room temperature and melts to attain a viscous liquid state when being exposed to high temperatures. The heating and cooling cycles of a thermoplastic binder system are being considered as reversible. Whereas, thermosetting [Andrews L. S. R, 1963; McIntyre, S.,
2008; Gupta et al., 1988] resins when exposed to elevated temperature convert to a liquid state from the solid state. The viscosity gradually increases and thereafter it acquires a solid state due to a cross-linking/polymerization reaction, even at the higher temperature.

The cross-linking rate of a thermosetting binder system can be controlled by the amount of cross-linker being added to the formulation. Thermosetting binder systems are most predominantly used in the foundry industry for the shell molding applications. Hexamethylenetetramine [McIntyre S., 2008], commonly known as ‘hexa’ has been the most widely used cross-linking agent by the industry in conjunction with the novolac resin (phenol formaldehyde) systems. These systems are known to possess almost indefinite shelf life under controlled environment conditions.

The cure temperature play a vital role in the shell molding process to impart desired mechanical strength due to the characteristics of the binder system and additives added. The optimum cure temperature can be defined as the cure temperature at which the cross-linking/polymerization reaction has been fully completed; and the binder system has been converted from a liquid state to a solid state by the means of cross-linking/polymerization reaction.

Excessive cure temperatures adversely affect the strength characteristics of these systems; therefore continuous monitoring of the cure temperatures plays a vital role in obtaining optimum castability parameters. With the lot-to-lot variation in the composition and amount of added additives, it becomes important to periodically determine the optimum cure conditions.
Experimental

Determining optimum cure characteristics plays a vital role in selection of molding material suitable for castability. The resin system is desired to impart rapid softening to a consistency favorable for flow and adhesion by subsequent instantaneous curing and solidification without too much drop in viscosity. The viscosity of a resin system after reaching softening is considered very important to minimize peel-back and maximize internal bonding strength. The melt-index [McIntyre S., 2008] provides a polymerization index of the resin system, higher melting points are indicative of more advanced polymerization reactions [McIntyre S., 2008] taking place. Low-melting point resin systems limit the cure speed, thereby causing delamination of soft/uncured mold parts generally termed as ‘peel-back’ [McIntyre S., 2008] and higher melt-point resin systems limit the internal bonding strength thereby, limiting the bridging on the vertical walls [McIntyre S., 2008] of shell-mold system. Melt or softening index can be determined by implementation of differential scanning calorimetry. A Differential Scanning Calorimeter (DSC) [Waitkus et al., 2003] as shown in Figure 6.1, identifies the phase transformation peaks by plotting heat flow vs. temperature curve. By use of DSC, the melt-index for 8 different shell sand specimens was determined. Tests were performed by packing 3-5mg of powdered resin-coated sand into a small aluminum pan, which was sealed using a closed lid system, and thereafter loaded into the DSC.
A non-isothermal scan plot of the given sample was generated for the heating cycle by conducting a temperature scan from 40°C to 300°C at a heating rate of 5°C/min. A cooling curve was also generated for the temperature range 300°C to 40°C at a cooling rate of 5°C/min. The peaks identified on a heat flow vs. temperature plot are representative of phase-transformations taking place in the binder system during heating and cooling cycles.

After determination of the melt-index using the differential scanning calorimeter, the cure temperature range was obtained from the data and optimum cure temperature determined for the 8 shell sand specimens, using a cure temperature indicator (CTI) as

*Figure 6.1* Differential scanning calorimeter (DSC).
A cure temperature indicator is comprised of a 50mm diameter and 8mm deep cavity. This cavity is divided into four temperature zones, where four different temperatures can be selected respectively, to identify the optimum cure temperature. The cavity is thereafter filled with resin-coated sand and excess sand is thereafter screened off to fill the 50mm diameter and 8mm deep cavity. Comparing the scuff resistance characteristics of cured layer further identifies the optimum cure temperature. A Dietert Core Hardness tester [McIntyre S., 2008] was used to determine the scuff resistance of cured specimens, where a hardness index of 100 indicated no penetration whereas; an index of 0 refers to .25 mm or .1 inch penetration of concentric reference surface [Beeley P., 2001]. A color chart or shell cure conundrum designed for shell sand systems, is used in current industry practices to identify the level of cure for the sand system. A visual color match by the operator is used to decide the level of cure,
however the color of sand can vary from lot-to-lot, depending upon the impurities and amount of additives present. However, in the event of dark specimens (as shown in Figure 6.3), the color variation could not always be perceived with the naked eye and thus cure levels could not be determined.

Therefore, an alternate methodology test to quantify the level of cure and thereafter to determine the optimum cure temperature was developed, which employed the use of a core hardness (also termed as scratch hardness) tester in conjunction with the cure rate indicator, as shown below in Figure 6.4.

*Figure 6.3* Cure conundrum for shell sand systems.
Color theory can be applied for quick determination of cure characteristics rather than relying on human perception of color in current industry practices. The color of a cured specimen can be measured using CIE LAB, a device independent color model [X-Rite Corporation, 2007]. A CIE LAB plot represents color values on three different scales ‘L’ (dark to light), ‘a’ (Red to green) and ‘b’ (blue to yellow). As shown Figure 6.5, the z-axis represents the Lightness scale (0-100) where 100 represent the lightest (white) and 0 represents the darkest (black) point. The x-axis represents ‘a’ values where negative values represent shades of red and positive values correspond to shades of green. The y-axis is representative of ‘b’ values where negative values correspond to shades of blue and positive values refer to the shades of yellow color.

Figure 6.4 Cure temperature indicator (CTI) used in conjunction with core hardness tester to evaluate strength properties of cured disc-shaped specimens.

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Generating a CIE LAB index for each sand system at different cure temperatures and comparing it to the measured specimen values can determine the cure level. The CIELAB values were measured by using an X-Rite 530 Spectrophotometer using the disc-shaped specimens being cured at four different temperatures from the Cure Rate Indicator (CTI).

Results and Discussion

Figure 6.6 represents the melt-index for the provided sand samples. This heat flow vs. temperature plot represents the phase transformation of the binder system being depicted by different peaks at different temperatures for different samples. Sand is considered a refractory material and is able to withstand temperatures in excess of 1500°C, therefore the peaks shown in the plot are representative of thermal effects on the resin system.
The resin system is a two-component system, which comprises of wax as the first component being represented by the first peak in mostly all of the samples at approximately 65°C. The other peak is representative of the other component of the resin system and varies sample-to-sample due to different added additives in different samples. A temperature range from 224°C to 274°C has been selected for the CTI to accommodate these different resin-coated sand samples.

*Figure 6.6* Heat flow vs. temperature plot obtained from DSC.
Four different temperature zones 224°C(435°F), 241°C(465°F), 257°C(495°F) and 274°C(525°F), respectively were selected and set for all four quadrants as per the results obtained for the melt-index. The strength of the disc-shaped specimens cured at different temperatures is represented in Figure 6.7.

![Figure 6.7 Core hardness values representing core/mold strength properties.](image)

The optimum cure temperature for each sand specimen was obtained by determining the maximum core/scratch hardness values. Results obtained from Analysis of Variance (Table 6.1) using 95% confidence interval for Core/Scratch hardness vs.
Cure Temperature suggests a strong dependence of the core hardness on cure temperature (as shown in Figure 6.8). Hence, the determination of the optimum cure temperature for each sample by use of CTI and core hardness testing played a vital role in the determination of cured core/mold strength characteristics.

Table 6.1 ANOVA for core/scratch hardness

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Adj. SS</th>
<th>Adj. MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cure Temp.</td>
<td>3</td>
<td>312.03</td>
<td>312.03</td>
<td>104.01</td>
<td>15.89</td>
<td>0.000</td>
</tr>
<tr>
<td>Error</td>
<td>20</td>
<td>130.91</td>
<td>130.91</td>
<td>6.55</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>23</td>
<td>442.93</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

S = 2.55838  R-Sq = 70.45%  R-Sq (adj.) = 66.01

*Figure 6.8 Main effects plot for core/scratch hardness.*
The digital color measurements for determining the cure level of the shell sand systems show a significant dependence on ‘L’ values. A set of 6 different cured shell sand specimens were used for this study. The cured carbon shell sand system was excluded thereof, due to dark color and negligible color changes observed with varying cure temperatures. Hence, the observations for only 5 different shell sand systems are reported. An average of 15 observations for each cured sand specimen at different cure temperatures suggests the dependence of ‘L’ values on cure temperature, as shown in Figure 6.9.

![Figure 6.9](image_url)  
*Figure 6.9* Plot representing dependence of L values vs. cure temperature.

The lower the cure temperature, the higher the ‘L’ value, or lighter the cured
sample. For most cases, this represents under-cured specimens as per current foundry practices. The ANOVA results and main effects plot for ‘L’ values are shown in Table 6.2 and 6.10, respectively. The higher the cure temperature, the darker the colors of the samples, representing a state of over-cured sand specimen by lower ‘L’ values. For each sand specimen, after generating a thermal cure index comprising of measured ‘L’ values at each cure temperature, it is very easy to quantify the level of cure by comparing the given and measured ‘L’ values.

Table 6.2 ANOVA for ‘L’ values

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Adj. SS</th>
<th>Adj. MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cure Temp.</td>
<td>3</td>
<td>14297.6</td>
<td>14297.6</td>
<td>4765.9</td>
<td>248.09</td>
<td>0.000</td>
</tr>
<tr>
<td>Error</td>
<td>296</td>
<td>5686.3</td>
<td>5686.3</td>
<td>19.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>299</td>
<td>19983.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S = 4.38296</td>
<td></td>
<td></td>
<td>R-Sq = 71.55%</td>
<td></td>
<td>R-Sq (adj.) = 71.26</td>
<td></td>
</tr>
</tbody>
</table>
Conclusion

This study demonstrated an alternate methodology for determining accurate cure parameters for various shell sand systems. It showed the correlation of various tests in the study to the core/scratch hardness, which defines the strength characteristics of the mold/core suitable to attain better castability.

This study also illustrated the application of color measurements for certain shell sand systems and developed a thermal cure index for each sample to quickly verify the level of cure by making color comparisons. Color comparisons made using color measurement instruments provided the ability to quantify the color and hence determine the cure levels while eliminating variability. Eliminating the need for the human perception of color, which differs from person to person, this new methodology can eliminate variability in the testing of cured sand samples.

Figure 6.10 Main effects plot for ‘L’ values.

Conclusions
These color measurement systems and test conducted thereof are applicable only for certain shell sand system where a strong dependence of ‘L’ values on cure temperatures can be observed. For the shell sand systems, such as carbon shell system used for the 3D light cured rapid casting applications, where the color of sand system is dark (black); these color measurement techniques cannot be applied. In such instances, the cure levels can be determined using a Thermal Cure Indicator (CTI) test in conjunction with Core/Scratch Hardness tester to identify the optimum cure characteristics.

Acknowledgements

The authors gratefully acknowledge Glenn Hall, Peter Thannhauser, Nick Dixon and Nick Miskovitch, Western Michigan University for their support.

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CHAPTER VII
RHEOLOGICAL EVALUATION OF REFRACTORY COATINGS FOR 3D CURED CARBON SAND SYSTEM

Summary

Refractory coatings are employed in various metal casting applications to impart enhanced surface characteristics by suppressing the mold-metal interfacial defects. The refractory coatings act as intermediate interfacial layer at the mold-metal interface to impart a variety of functional characteristics. These coatings boost the performance of mold/core in casting applications by improving the interfacial surface finish, hinder the thermal-transfer characteristics, improve venting characteristics to reduce gas defects and protect the mold-metal interface.

Therefore it is important to characterize these coatings on various parameters such as wettability, adhesion, flow ability, leveling, etc. These parameters could be analyzed by employing a variety of tests. A wide range of instruments has been utilized to estimate the wetting characteristics and to analyze the rheological characteristics of the coatings developed for carbon shell sand system.
Introduction

Refractory coating [Guyer et al, 2005] is used in the foundry industry to impart desired functional characteristics to mold/core. Refractory coatings refer to the application of selected refractory materials [Schleg et al, 2008] to resin bonded cores and molds. These coatings are applied with the objective to improve the surface finish of mold/core interface, control the heat transfer characteristics at metal-mold interface, alter the venting characteristics of core and mold and prevent certain defects in casting such as erosion, etc.

In order to impart good surface finish characteristics and minimize/eliminate additional machining/tooling steps, smooth mold and core interfaces are required. The surface smoothness of these interfaces can be altered by the application of refractory coatings. The gas-generated defects could be minimized closing air/gas passageways located at the mold/core surface by application of refractory coatings. The generated gaseous products could then be forced to escape the system by strategic placement of vents in the mold design.

These refractory coatings consist of several ingredients [Guyer et al, 2005; Rebros et al, 2007] such as refractory material (pigment), carrying agent (binder), suspension agent (dispersant), rheology modifiers, surfactants, etc. These coatings can be applied by various methods including dipping core and mold sections into coating formulation, bushing on the coating, spraying coating onto cores and molds and strategic placement of coatings onto cores/molds using inkjet heads.

The coating characteristics such as wettability, leveling, flow phenomenon, etc.
can be determined by implementation of various surface phenomenon and rheological studies. Surface energy [Bohra et al., 2008] of the core/mold surface plays a vital role in determination of wettability characteristics; the surface characteristics in terms of interactions with liquid phases. The polar and dispersive components of surface energy are employed so as to estimate the interaction of a substrate with polar and non-polar liquids.

Rheology [Tanner, 2000; Mezger, 2006] can be defined as the study of the flow and deformation of materials when dealing with forces, stresses, and energy interchanges. Deformation can be described three different ways: Reversible, Irreversible and Reversible-Irreversible. Further, a reversible deformation is understood as being elastic, irreversible deformation is flowing or viscous and reversible-irreversible is viscoelastic [Joyce, 2012; Tanner, 2000]. Rheology affects things such as storage capability and shelf life, flow and leveling, smoothness and uniformity, and viscosity and dispersion stability [Joyce, 2012]. By testing coatings or printing inks under shear, the application aspects of flow and leveling become clearer.

**Experimental Methodology**

**Surface Energetics**

Surface energy [Kan et al., 2004; Adamson et al., 1997] of a substrate can be defined as the energy required, for forming a unit area of new surface at the solid-gas interface. Surface energy is an essential component in estimating wettability of a solid surface with a liquid, and hence to characterize the solid-liquid interface interaction properties, such as spreading coefficient [Kan et al., 2004; Adamson et al., 1997]. The
surface energy of a substrate can be estimated by measuring the contact angle of different immiscible liquids on the surface of the substrate. This estimation of the surface energy of a substrate and surface tension of a liquid can be used in determining the wettability by application of rule of thumb used by printing industry for the decades. This rule suggests that in order to obtain optimum wettability and adhesion characteristics, the surface energy should be at least 10 points higher than the surface tension of the observed liquid. The contact angle (Figure 7.1) of a liquid on the surface of a substrate can be measured using the sessile drop method [Adamson et al., 1997] and hence the surface energy of substrate can be estimated [Owens et al., 1969] by measuring the contact angle of different immiscible liquids on the surface of mold/core.

![Figure 7.1 Contact angle measurement of a liquid (water) on the surface of mold/core.](image)

The surface energies of these cured mold/cores were estimated by measuring contact angles of two different liquids (water and methylene iodide). In order to estimate surface energy, the Owens-Wendt method [Owens et al., 1969], which decomposes the total measured surface energy into two components, i.e. dispersive and polar components, was used. The contact angles of the above mentioned liquids on these coated sheets were
measured using FTA Dynamic Contact Angle measurement device (Figure 7.2).

![FTA Dynamic Contact Angle measurement device](image)

*Figure 7.2 FTA 200 dynamic contact angle measurement device.*

**Coating Characterization**

The two developed coatings, one for light-metal casting applications such as aluminum, zinc, etc.; and the other for heavy-metal casting applications such as iron, were adjusted to the viscosity levels suitable for dip coating applications. The viscosities of these coatings were evaluated at different RPMs using a Brookfield viscometer. The Thix (thixotropic) index for these coatings was calculated as per current industry practices by taking the ratio of measured viscosities (cps) at two different RPM [McGregor, 2013], 10 and 20 RPM respectively using spindle number 3. The solid content of these coatings upon dilution to desired viscosity levels was determined and recorded using a moisture/solids microwave unit.
Rheological properties such as leveling and flow phenomena were studied utilizing Hercules DV-10 HI-Shear Viscometer and TA AR 2000 Stress-Controlled Rheometer. The leveling index was measured using the Hercules DV-10 HI-Shear Viscometer and obtaining a rheogram representing rotational speed in revolutions per minute (RPM) vs. torque measured in dyne-cm. The leveling index can thereby be calculated as per the method described in the literature [Triantafillos, Kaltec Scientific Inc.], where viscosities at two different RPMs (one at peak and one at midpoint) are determined for both ‘up curve’ and ‘down curve’ on the rheogram. Further calculations for the leveling index are done following the equations as per defined in Appendix D [Triantafillos, Kaltec Scientific Inc.]. Leveling index can be defined as a dimensionless ratio of thixotropic coefficient to the peak plastic viscosity, a high leveling index refers to a higher thixotropic coefficient and a lower plastic viscosity, whereby a value above 0.3 is preferred for various low-speed coating applications.

Various rheological tests were conducted using the coating samples to better understand the flow phenomenon and leveling characteristics of the coatings. To understand the viscoelastic behavior of these refractory coatings, a constant shear stress is applied using TA AR2000 rheometer and thereby, continuous monitoring as the deformation progresses by continually recording strain vs. time. The couette geometry was used on the rheometer to conduct the test. The constant shear stress was determined from the stress limits for Linear Viscoelastic Region (LVR) by running a stress-sweep test at a frequency of 1Hz prior to conducting the test. A frequency sweep test was also performed using the oscillation stress value as determined for the LVR region. Thereafter, the
applied stress was released by application on minimum stress as per being specified by instrument limits and plotting strain vs. time curve continually monitored reformation.

After conducting the creep-recovery test various model fits were performed by plotting Compliance versus Time and the best fit was determined based on the standard error minima.

![Hercules DV-10 Hi-shear viscometer and TA AR 200 dynamic stress-controlled rheometer.](image)

*Figure 7.3 Hercules DV-10 Hi-shear viscometer and TA AR 200 dynamic stress-controlled rheometer.*

**Casting Trial and TDT**

For this experiment a chemically bonded shell sand mold was developed to deliver molten metal to six chemically bonded disc shaped specimens simultaneously.
Certain disc shaped specimens experience external tangential point loads (300g and 400g) replicating an addition 14 and 18-inch aluminum head pressures, when the mold is poured to a 6-inch metallostatic head wetting all surfaces of the specimens (Figure 7.4). The purpose of this model is to identify the effect of super heat, metallostatic head and external load on chemically bonded disc shaped specimens to determine at what conditions distortion, penetration and/or veining occurs.

*Figure 7.4* Thermo-mechanical point loading on specimen.

The mold making procedure consisted of the following steps: shell sand cope and drag mold halves were fabricated according to an experimental pattern. The drag mold with six specimens set on core-prints is shown in Figure 7.5. Three disc-shaped 3D light cured carbon shell specimens (refer chapter iv and v) were uncoated (identified as no. 1, 2 and 3 in the final casting) and three were coated with the light-metal coating being developed for Aluminum casting applications (identified as no. 4, 5 and 6 in final casting).
The coating was applied to specimens 4-6, by implementation of dip coating process utilizing a robotic arm. The robotic arm [Abdelwahab et al., 2009] (as shown in Figure 7.6) was used to dip coat the samples in order to minimize the sample-to-sample variability, by maintaining constant speeds during the dipping and draining processes for each sample. The sidewalls of the sample were covered using a Teflon tape and the coated samples were allowed to dry at in oven at a temperature of 105°C for 24 hours.
After preparation of the coated specimens, these specimens were then arranged in the drag mold half as discussed earlier. A six-inch tall pouring sleeve was affixed to the cope as a sprue to deliver the metallostatic head where one of each specimen type received a point load (Figure 7.7). The molds contained six symmetrical cavities and were poured where the chemically bonded disc shaped specimens were arranged by material and point loading sequence. This approach allowed for possible variation in casting surface quality (specimen/metal interface) to be assigned to the chemically bonded sand specimens. The sand-to-metal ratio for all molds was 4:1. Molds were manually poured and the aluminum was (average pouring time = 10 sec., temperature at
pour 732°C (1350°F)) delivered through a pouring sleeve fitted with a foam filter.

Thermal Distortion Testing (TDT) was also conducted using these coated specimens to analyze the thermal transfer characteristics and interactions at mold-metal interface at elevated steady-state temperature of 1000°C and an acting head pressure to simulate the force of molten metal to a 6 in. (15.24cm) head height for cast iron with a density of 0.25lb/in³ (6.92g/cm³) providing a head pressure of 1.50psi (0.01MPa). A detailed procedure of this test has been explained in Chapters IV and V.

![Figure 7.7 Poured mold with point loads.](image)

Castings were allowed to solidify prior to cooling and shakeout. The casting was sectioned at the core/metal interface and images for the casted mold/metal interface were

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captured using an ImageXpert image analyzer for visual analysis of the surface defects.

Results and Discussion

Observations for Surface Energetics

The contact angles as measured for water on the surface of cured disc-shaped carbon shell sand specimen represents hydrophobicity of the specimen. The average contact angle for water and methylene iodide on the surface of the disc-shaped specimens as shown in Figure 7.8 were determined to be 108.13±0.5 and 61.57±2.17. The surface energy of the specimen calculated using Owens-Wendt method was estimated to be 28.11 dyne/cm². The dispersive and polar components of the surface energy were calculated to be 28.11 dyne/cm² and 0 dyne/cm² respectively.

*Figure 7.8* Graph representing contact angle measurement values for water and methylene iodide for carbon shell sand disc-shaped specimens.

Table 7.1 represents the density and interfacial tension (IFT) of the observed coatings. Comparing IFT with the surface energy suggests the wettability and
adhesion characteristics of these coatings on the surface of carbon shell sand system. The light-metal coatings

Table 7.1 IFT and contact angles of coatings on the surface of the specimens

<table>
<thead>
<tr>
<th>Coatings</th>
<th>Density (g/cm³)</th>
<th>IFT (dyne/cm)</th>
<th>Contact Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heavy-metal</td>
<td>1.39±0.2</td>
<td>33.54 ± 0.6</td>
<td>79.83 ± 2.4</td>
</tr>
<tr>
<td>Light-metal</td>
<td>1.33±0.2</td>
<td>25.22 ± 2.1</td>
<td>64.82 ± 2.7</td>
</tr>
</tbody>
</table>

The Brookfield viscosities measured at three different RPM and thixotropic index calculated thereon are represented in Table 7.2. The solid content values measured for the two coating specimens at these viscosities are also in the table.

Table 7.2 Brookfield viscosities and solid content of the coatings

<table>
<thead>
<tr>
<th>Coatings</th>
<th>10 RPM</th>
<th>20 RPM</th>
<th>100 RPM</th>
<th>Thixotropic Index</th>
<th>Solid %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heavy-metal</td>
<td>364 cps</td>
<td>210 cps</td>
<td>124 cps</td>
<td>1.73</td>
<td>42.62%</td>
</tr>
<tr>
<td>Light-metal</td>
<td>400 cps</td>
<td>234 cps</td>
<td>128 cps</td>
<td>1.71</td>
<td>41.39%</td>
</tr>
</tbody>
</table>

Leveling index was calculated for the two coating specimens by determining the torque values corresponding to 2200 and 5400 RPM for the ‘up curve’ and ‘down curve’ from the rheogram shown in Figure 7.9. The apparent viscosity versus shear rate rheograms as obtained for the two coatings from the Hercules DV-10 Hi-shear viscometer are shown in Figure 7.10.
Figure 7.9 Rheogram obtained from Hercules DV-10 Hi-shear viscometer.

Figure 7.10 Viscosity versus shear rate plot obtained from the Hercules DV-10 Hi-shear viscometer.
The torque values as determined from the RPM versus torque rheograms and calculated values of the leveling index thereon for the coating samples are shown in Table 7.3. The results obtained from Hi-shear viscometer suggest poor leveling performance for both of the coating specimens, however the leveling performance of the light-metal coating was found to be completely unsatisfactory. Sometimes, depending on the method of coating application, the leveling index results for the low-shear application methods using the Hi-shear viscometer appear deceptive as per the instruments capabilities, therefore a creep-recovery test using the TA AR 200 rheometer are conducted. Based on the observations, adjustments to the coating formulations to improve the leveling characteristics are mandatory to improve the surface smoothness of the coated specimens.

Table 7.3 Leveling index of the coating specimens

<table>
<thead>
<tr>
<th>Coatings</th>
<th>Up-Curve</th>
<th>Down-Curve</th>
<th>Leveling Index</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Torque (T1) @ 2200 RPM</td>
<td>Torque (T2) @ 5400 RPM</td>
<td>Torque (T1') @ 2200 RPM</td>
</tr>
<tr>
<td></td>
<td>kilodyne-cm</td>
<td>kilodyne-cm</td>
<td>kilodyne-cm</td>
</tr>
<tr>
<td>Heavy-metal</td>
<td>260</td>
<td>575</td>
<td>200</td>
</tr>
<tr>
<td>Light-metal</td>
<td>220</td>
<td>525</td>
<td>180</td>
</tr>
</tbody>
</table>

By conducting a stress sweep test for the two refractory coating samples, the limiting stress values in linear viscoelastic region was determined and thereby was used as input for creep test at constant stress values. The observations revealed that the limiting oscillation stress for the two coatings was 0.2 Pa. The results
obtained from the stress sweep test are shown in the Figure 7.11 below:

![Stress sweep plot](image)

*Figure 7.11 Stress sweep plot for the two coating samples.*

After determination of the limiting oscillation stress for the LVR region, the limiting value was used to conduct a frequency sweep test. This test was conducted to determine elastic modulus, viscous modulus and coefficient of viscoelasticity. Higher values of elastic modulus represent better reformation of coating structure upon removal of the applied stress, whereas higher viscous modulus represents higher permanent deformation upon application of applied stress. Figures 7.12, 7.13 and 7.14 correspond to the elastic modulus (Figure 7.12), viscous modulus (Figure 7.13) and coefficient of viscoelasticity (Figure 7.14) respectively. The elastic modulus represents the elastic behavior of coatings and suggests that there is no significant difference in the elastic behavior of the two coatings. The viscous modulus shows the viscous or plastic behavior.
of coatings and is representative of the deformation on application of stress. The graph reveals that the coating formulated for heavy metals shows a higher level of deformation as compared to the other coating sample. The coefficient of viscoelasticity represents the ratio of viscous modulus to the elastic modulus. The graph for viscoelasticity shows a higher value viscoelasticity for the heavy-metal coating sample, which suggests the dominance of viscous nature over the elastic properties.

*Figure 7.12* Frequency sweep plot for the two coating samples representing the elastic modulus.
Figure 7.13 Frequency sweep plot for the two coating samples representing the viscous modulus.

Figure 7.14 Frequency sweep plot for the two coating samples representing the coefficient of viscoelasticity (tan δ).
The creep and recovery test was conducted to determine the leveling characteristics. The creep phase of the test was run at a constant oscillation stress of 0.2 Pa as determined from the stress-sweep for a period of approximately 600 seconds. The recovery phase curve was obtained by the application of constant oscillation stress 0.00656 Pa determined by the limiting minimum oscillation stress application capabilities of the instrument. The recovery after application of negligible stress for light-metal coating was observed to be 21.74% of the total deformation after a period of 600 seconds during the recovery phase. Whereas, the reformation for the heavy-metal coating was observed to be 15% of the total deformation caused during application of 0.2 Pa stress.

*Figure 7.15* Strain versus time plot depicting the creep and recovery phases.

Shear compliance [Mezger, 2000] can be defined as the reciprocal value of shear
modulus and refers to the rigidity of the coating against leveling. $J_0$ corresponds to the instantaneous shear compliance. The higher the $J_0$ value, correlates to a higher deformation resulting in a slower reformation. $J_e$ is defined as the compliance after an infinitely long period. Lower values of $J_e$ represent higher reformation or recovery at an infinitely long time interval. Retardation can also be explained as the delay of elasticity, and is representative of the delayed response of the coating to the applied stress. The ratio of $J_t/J_{t-1}$ after time interval $t$ represents the recovery in the given time interval. After an infinitely long period of time for the two coatings being identified as light metal and heavy metal on the plot, the recovery was determined to be 93% and 92.35% respectively.

![Figure 7.16 Compliance versus time plot representing the creep and recovery phases.](image-url)
Observations from TDT and Casting Trial

It is important to reiterate that the 8 mm disc-shaped specimen cores used for casting were poured from a 6 inch (15 cm) head height at 732°C (1350°F). In the casting trial all specimens support the thermo-mechanical stresses involved in filling and point loading through solidification. However, there was macroscopic distortion observed at the mold/metal interface. The refractory coating investigated did not prevent distortion, but did improve the interfacial as-cast surface. It should be pointed out that the refractory coating was dip coated and the coating drip marks from the dip coating application were apparent on the specimen and interfacial surfaces as well.

Observations were made from the disc-shaped specimen core/metal interface surface of the casting (Figure 7.17). Macroscopic observations revealed the three-coated core/metal interfaces were comparatively clean and possessed minimal casting defects, which was augmented with point loading. The uncoated 3D light cured carbon shell sand system had the greatest amount of specimen-metal interfacial defects (penetration); this was also augmented with point loading. It is evident that the thermo-chemical reaction for carbon sand and aluminum and thus the incompatibility, however the application of the coating developed for light-metal alloys may result in better casting performance with improved surface finish and reduce surface defects.
<table>
<thead>
<tr>
<th>Specimen</th>
<th>Uncoated</th>
<th>Coated</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Load</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
</tr>
<tr>
<td>300g Load</td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
</tr>
<tr>
<td>400g Load</td>
<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
</tr>
</tbody>
</table>

*Figure 7.17* Images showing core/metal interface.
The coated and uncoated disc-shaped specimens were tested at elevated temperature over a 90 second interval; TDT curves and picture information related to the system are presented in Table 7.4 and Figure 7.18.

The TDT curves for all three specimens showed undulations that indicate thermo-mechanical and thermo-chemical changes in these systems at elevated temperature. The longitudinal distortion curves showed a plastic deformation (downward movement of TDC). Note that TDT curves for longitudinal distortion and radial distortion depicts an average for 3 specimens tested (Figure 7.18).

There are many heat induced thermo-chemical reactions occurring in all three sets of specimens as evident from the hairline surface cracks found on tested specimens and percent change in mass values (Table 7.4). Expansion cracks were macroscopically evident on all specimens tested. Significant mass losses were evident with 3D light cured uncoated and coated specimens (Light metal coating).

All specimens tested remained after TDT and after the application of the 20 psi (0.14 MPa) air pressure. Every specimen tested had losses of sand and possessed cracks that increased in size with the duration of the test. Observations from the heat-affected zone on the surface of tested specimens revealed that all the specimens had visible sand losses and crack propagation. The hot surface/specimen interface generally shows a crater with discoloration due to binder degradation, the discoloration was not present on the opposite side of these specimens. Sand binder losses were evident at the hot surface/specimen interface where binder bridges pyrolyzed and sand grains broken loose.
Table 7.4 Thermo- mechanical properties of coated and uncoated specimens

<table>
<thead>
<tr>
<th>Type</th>
<th>Blow Pressure (psi)</th>
<th>$D_E$ Longitudinal (mm)</th>
<th>$D_P$ Longitudinal (mm)</th>
<th>$T_D$ Total Dist. (mm)</th>
<th>$D_R$ Radial (mm)</th>
<th>% Change in Mass</th>
<th>Cracks &amp; Fractures</th>
</tr>
</thead>
<tbody>
<tr>
<td>Un- Cured</td>
<td>20</td>
<td>0.00</td>
<td>0.11</td>
<td>0.11</td>
<td>0.085</td>
<td>1.59</td>
<td>Minimal</td>
</tr>
<tr>
<td>Light Metal Coated</td>
<td>20</td>
<td>0.00</td>
<td>0.08</td>
<td>0.08</td>
<td>0.075</td>
<td>2.43</td>
<td>Significant</td>
</tr>
<tr>
<td>Heavy Metal Coated</td>
<td>20</td>
<td>0.00</td>
<td>0.07</td>
<td>0.07</td>
<td>0.050</td>
<td>.46</td>
<td>Minimal</td>
</tr>
</tbody>
</table>

Figure 7.18 TDT Curves showing Longitudinal and Radial Distortions.
As compared to conventional silica or PUCB sands, the carbon shell sand behaves differently as observed during the TDT. It can be inferred from the TDT observations for the carbon shell sand that the radial distortion ($D_R$) decreased significantly, on the other hand an increase in longitudinal distortion was also observed. The radial and longitudinal distortions were however, surpassed by application of these refractory coatings. The heat transfer to the backside of the specimens (as shown in Figure 7.19) at elevated temperature (1000°C) did not affect the backside temperature as much as observed in conventional silica sand systems [Oman et al, 2013], suggesting the carbon shell sand systems acting as a ‘chill’ material. However, the thermal transfer characteristics were hindered by application of these refractory coatings, as being observed for the coated specimens. This observation suggests the ‘insulating’ behavior of the coatings as being expected from the coating formulations.

![Figure 7.19](image-url)  
*Figure 7.19* Temperature versus time plots representing the heat transfer.
Conclusions

This study has shown revealed the surface energetics and flow phenomenon of the two coating samples being developed for carbon shell sand systems. The surface energy estimations for the carbon shell sand specimens suggests the hydrophobic nature of these specimens and refer to achievement of poor wettability using aqueous coating systems. However, wettability can be improved to a certain extent by making adjustments to the level of surfactants added in the coating formulation. To obtain good wettability conditions, the interfacial tensions of the coating should be less than the surface energy of the carbon shell sand specimens, which can be reduced to a limit by increasing the level of surfactants in the formulation.

The rheological characterization of the coating specimens suggests poor leveling performance of these coatings. The creep and recovery test results suggest a longer recovery or reformation referring to the poor leveling performance as well. The coating formulation needs to be adjusted to impart better flow ability and leveling characteristics to improve the surface smoothness of the coated disc-shaped specimens.

The TDT results reveal the insulating behavior of the tested refractory coatings, which suggests the application of these coatings in smart coating application areas, where thermal transfer properties can be hindered strategically. Overall, thermal distortion and mass losses have been reduced after application of these coatings.

The casting trial reveals reduction in surface defects as achieved on the mold-metal interface with the application of the light-metal coating for aluminum casting applications, which cannot be neglected. The coatings definitely impart better interfacial
characteristics thereby reducing surface defects such as penetration and eliminating veining. However, the application of this light-metal coating can intensify the distortions at mold-metal interfaces. Therefore a detailed experimental analysis of the thermal distortion with the coated samples is required.

Acknowledgements

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References


CHAPTER VIII
CONCLUSION

A sand mold or shaped cavity with defined level of complexity including curvilinear shapes and surfaces was successfully produced using the hybrid technique, involving additive and subtractive manufacturing with successful integration of CAD and CAM technologies. The process capabilities to attain complex shapes have been clearly demonstrated, which qualifies the process to be employed in various rapid prototyping and rapid manufacturing applications.

The qualification of the carbon shell sand system used for the mold development utilizing the hybrid manufacturing approach suggests that there lies a difference in both room and elevated temperature behavior for the sand binder system studied. The 3D light cured carbon shell sand gained strength in a manner similar to thermosetting resins.

The target is to develop a 3D light cured shell mold with low sand to metal ratio where these elevated temperature strength properties are beneficial. 3D light cured carbon shell sands possess the physical and mechanical properties suitable for core and mold development also at room temperature. The 3D light cured carbon sands are thermally stable. However, carbon shell sand is not compatible with some molten metal chemistries; this was revealed in the casting trial.

A longer shelf life of carbon shell sand considering the longer shelf life possessed by shell sand systems, ease of machinability and safety and logistical advantages can be considered as add-on for the suitability of the molding material for applications in rapid casting.
When 3D light cured carbon sands are used for a rapid casting process with alloys such as aluminum, cast iron, etc., a suitable refractory coating interface is required. It is therefore recommended that casting trials with refractory coatings applied to 3D light cured carbon shell sand molds and cores be studied in detail for better castability.

Proof-of-concept casting (as shown in Fig 7.1) with moderate complexity level has been successfully obtained through casting trials conducted at WMU. This shows the capability of the process to produce castings with moderate complexity, however, the process capabilities to produce higher complexity castings with internal geometry features is under investigation.

*Figure 8.1* Images showing a complex mold cavity and a casted part obtained thereof using the hybrid rapid casting approach.

The layered experimental analysis suggests that the proposed media for the light cured system possess the desired physical, mechanical and thermo-mechanical properties.
for castability. These single and double-layered light cured sand specimens are quite stable at elevated temperatures, as revealed from the thermal distorting testing.

An alternate methodology for determining accurate cure parameters for various shell sand systems has been demonstrated. It shows the correlation of various test implied in the study to the core/scratch hardness which defines the strength characteristics of the mold/core suitable to attain better castability.

It is illustrated from the study that the application of color measurements for certain shell sand systems can be employed. These color measurements can be utilized for developing a thermal cure index for each sample to quickly verify the level of cure by making color comparisons. Color comparisons made using color measurement instruments provide the ability to quantify the color and hence determine the cure levels while eliminating the variability. Eliminating human’s perception of color, which differs from person to person, can eliminate the variability in this event.

These color measurement systems and test conducted thereof are applicable only for certain shell sand system where a strong dependence of ‘L’ values on cure temperatures can be observed. For the shell sand systems, such as carbon shell system used for the 3D light cured rapid casting applications, where the color of sand system is dark (black); these color measurement techniques cannot be applied. In such instances, the cure levels can be determined using a Thermal Cure Indicator (CTI) test in conjunction with Core/Scratch Hardness tester to identify the optimum cure characteristics.

This study has revealed the surface energetics and flow phenomenon of the two
coating samples being developed for carbon shell sand systems. The surface energy estimations for the carbon shell sand specimens suggest the hydrophobic nature of these specimens and refer to achievement of poor wettability using aqueous coating systems. However, better wettability can be achieved by adjustment to the level of surfactants added in the coating formulation. To obtain good wettability conditions, the interfacial tensions of the coating should be less than the surface energy of the carbon shell sand specimens, which can be reduced to a limit by increasing the level of surfactants in the formulation.

The rheological characterization of the coating specimens suggests poor leveling performance of these coatings. The creep and recovery test results suggest a longer recovery or reformation referring to the poor leveling performance as well. The coating formulation needs to be adjusted to impart better flow ability and leveling characteristics to improve the surface smoothness of the coated disc-shaped specimens.

The casting trial reveals reduction in surface defects as achieved on the mold-metal interface with the application of the light-metal coating for aluminum casting applications, which cannot be neglected. The coatings definitely impart better interfacial characteristics thereby reducing surface defects such as penetration and eliminating veining. However, the application of this light-metal coating could reduce the amount of thermally induced distortions at mold-metal interfaces. Using these coating, the thermal transfer characteristics could be altered, as these coatings being acting as insulating material. Hence, these coatings can be used for smart coating applications where strategic placement of insulation material is required to impart better castability.
APPENDICES

Appendix A

Additional carbon shell sand test results

Loss on Ignition test for Carbon Shell Sand system

The LOI test for carbon shell sand was conducted by studying two different sand samples, out of which one was used as reference. The LOI for the carbon shell sand was calculated by comparing the weight loss for the two samples after exposure to very high temperatures. The reference sample used for the study comprised of uncoated carbon sand, whereas the other sand system studied during this test comprised of a resin coated carbon shell sand. 15g sample size of each sand were weighed in small crucibles and oven dried at 105°C for 24 hours to drive-off the moisture present in the sand systems.

After drying the samples were weighed again, and negligible wt. loss was determined. These samples were thereafter exposed to a temperature of 1000°C for 6 hours using a muffle furnace. These samples were taken out of the furnace and cooled for 5min using a desiccator, and re-weighed to determine the wt. loss. Wt. loss for the reference carbon sand sample was observed to be 5.1g, whereas for the carbon shell sand system the wt. loss was determined to be 5.5g.

The LOI for carbon shell sand was thereafter calculated by dividing the difference in wt. loss by the sample wt. The LOI for the carbon shell system was thereby calculated to be 2.66%.

Mechanical Properties of Carbon Shell Sand system

Impact testers are used to determine the impact strength of materials. Dynamic
tensile behavior being considered as important, the impact test determines the parameters of performance of a material at strain rates closer to some end-use applications. A product being of superior or inferior quality can be judged on the basis of performance index and consistency by analysis of mechanical strength and the variability as well.

Impact test was conducted using a Tinius Olsen impact tester, with up to 2.825J or 2,825mJ capacities, in order to determine the strength disc shaped specimens in terms of breaking energy (Joules). The impact tester was used in conjunction with a Dynatup energy display, model ETI-220P as shown in Figure A.1. The observations from the experiment for single and double layer 8mm thick disc-shaped specimens revealed similar impact strength for both the specimens. The impact strength for single and double layers was found to be $213\pm41.22$ and $199.2\pm33.36$ mJ, respectively.

Figure A.1 Tinius Olsen Impact Tester equipped with Dynup Energy Display.

Shell Thickness Indicator (STI)
This test was employed to study the maximum layer thickness, which can be achieved at optimum cure temperature in a given period of time. The test equipment comprises of three 50mm diameter temperature controlled cavities with 8mm, 12mm and 16mm depths respectively, as shown in Fig A.2.

These cavities were set to the desired temperature control determined by the optimum cure temperature using Cure Rate Indicator (refer Chapter VI). The optimum cure temperature for carbon shell sand system was determined to be 255°C and was used for further experimental analysis. The test was conducted by heating up and the cavities to the set temperature and filling the resin-coated carbon shell sand in these cavities and the excess sand was screened off. The sand specimen was cured for the selected time cycle of 1 minute and thereafter the specimens were ejected. A maximum shell thickness of 8mm was determined for the carbon shell sand at 255°C during 1 minute.
Appendix B

Rheological characterization of coatings formulated for carbon shell sand system

To understand the viscoelastic behavior of three different refractory coating samples (marked as Alumina (Al₂O₃) {IFT 22.25mN/m} being solvent based and Diatomaceous Earth (DE) {IFT 15.13mN/m} & Zircon {IFT 19.9mN/m} being water based), a constant shear stress is applied using TA AR2000 rheometer and thereby, continuous monitoring as the deformation progresses by continually recording strain vs. time. Thereafter, the applied stress was released by application on minimum stress as per being specified by instrument limits and plotting strain vs. time curve continually monitored reformation. The couette geometry was used on the rheometer to conduct the test. The constant shear stress was determined by determining the stress limits for LVR by running a stress-sweep test at a frequency of 1Hz prior to conducting the test. After conducting the creep-recovery test various model fits were performed by plotting Compliance versus Time and the best fit was determined based on the standard error minima.

By running a stress sweep test on all three different refractory coating samples, the limiting stress values in linear viscoelastic region was determined and thereby used as input for creep test at constant stress values. The observations revealed that the limiting osc. stress values for DE and Zircon coatings were 0.2 and 1.0 Pa respectively. However, a LVR region for Alumina coating could not be obtained even after trying various test geometries and different test method such as applying a pre-shear to the sample. An oscillation stress value of 0.1 Pa was used to run the creep-relaxation experiment as the
minimum input value for the relaxation experiment is 0.00656 Pa. The results obtained from the stress sweep test are shown in the Table B.1

Table B.1 Stress sweep test results for the different coating samples.

<table>
<thead>
<tr>
<th>Coating</th>
<th>Limiting Osc. Stress Value</th>
<th>LVR region</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina Coating</td>
<td></td>
<td>0.1 Pa</td>
</tr>
<tr>
<td>Diatomaceous Earth Coating</td>
<td></td>
<td>0.2 Pa</td>
</tr>
</tbody>
</table>
The creep recovery curve shown in Table B.2 represents the two phases of the creep-recovery experiment. The first phase refers to the creep phase where a constant stress is applied on each sample as determined from the stress sweep test and the creeping motion of the specimen is carefully monitored for approximately 600 seconds. The second phase being recovery phase represents the recovery/relaxation of the specimens after the removal of applied stress by applying the minimal possible stress as per the instrument’s limitation. The recovery curve suggests the amount of strain recovered in a period of 600 seconds. As being observed from the curve, the Alumina coating reflects an ideal-viscous behavior being depicted by zero strain recovery. The Diatomaceous Earth coating however, shows a strange behavior as the strain keeps on increasing even on the removal of applied stress, which makes difficult to predict this behavior. On the other hand, Zircon coating shows a perfect viscoelastic behavior with partial recovery of approximately 30% of total strain experienced after removal of applied stress.
Table B.2 Creep recovery test results for the different coating samples.
To obtain best fit and thereby to determine the elastic modulus and approximate the coating behavior under applied stress various model fits were applied to the creep-recovery curve. For applying the model fits the variables on the curve were changed from strain vs time to compliance vs time. The model fit with minimum standard error for each specimen were selected and shown in Table B.3. The applied model fits reveals the elastic modulus values to be 3.067E-3 Pa for Alumina, 28.13 Pa for Diatomaceous Earth and 29820 Pa for Zircon coatings respectively. Table B.4 represents the retardation times calculated from discrete retardation spectrum for each spectral point and corresponding compliance value from which in the recovery phase the recovery time can be studied, however the method of calculation is not completely understandable in the case.
Table B.3 Creep recovery test results demonstrating the model fit.

<table>
<thead>
<tr>
<th>Coating</th>
<th>Model Fit</th>
<th>Standard Error</th>
<th>End Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina Coating</td>
<td>Jeffrey's Under-damped Ringing</td>
<td>201.7</td>
<td>Finished normally</td>
</tr>
<tr>
<td>Diatomaceous Earth Coating</td>
<td>Jeffrey's Under-damped Ringing</td>
<td>98.48</td>
<td>Finished normally</td>
</tr>
<tr>
<td>Zircon Coating</td>
<td>Maxwell Under-damped Ringing</td>
<td>172.3</td>
<td>Finished normally</td>
</tr>
</tbody>
</table>
Table B.4 Creep recovery plot demonstrating discrete retardation spectrum.

Alumina Coating (Solvent Based)

Diatomaceous Earth Coating (water based)

Zircon Coating (water based)
Considering the results obtained from the creep-recovery experiment the conclusion can be drawn easily that Alumina and Diatomaceous Earth coatings are not a good fit for refractory coating applications. As under the application of stress during the coating process, it is not able to recover back showing poor leveling characteristics, which can thereby result in achieving poor surface finish at the coated interface. The Zircon coating appears to be the best fit out of these three given specimens showing better recovery of approximately 30% in a time period of 600 seconds which suggests the possibility of longer recovery period. The comparison from the elastic modulus for these coatings suggest the that Zircon coating shows the best leveling performance out of three given samples, as the higher the elastic modulus, the better the recovery and better leveling characteristics.