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# **Toughening of Ceramic Shell Mould with Rice Husk Fiber** (CSm-RH) to Improve Strength Property and Mould Performance

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**Abstract:** For ages, ceramic shell mould (CSm) have been extensively applied in investment casting industry. The formation of CSm requires multiple steps of dipping, layering drying and firing stages. The later steps are very crucial as the solidification thin layer CSm that consist of loose ceramic particles easily cracks when exposed to the higher thermal effect. The inclusion of fiber or any reinforces phases is able to enhance fired ceramic body and also strengthen the green ceramic structure. Thus, the feasibility of rougher NaOH treated rice husk fiber (RHT) prior embedded into composited structure has shown a significant CSm improvement by induced a better adhesion properties and larger bonding area with brittle ceramic matrix, resulted in increased green strength (1.34 MPa) and fired body strength (4.32 MPa). Owing to the decomposed of lignin layer in CSm with untreated rice husk fiber (CSm-RHU) exhibited a higher porosity that provide a better permeation paths of air flow during molten metal pouring as increased 30 % from the standard CSm permeability, giving an enormous benefit for investment casting cooling process. Overall, the incorporation of RHT fiber in a CSm mould.

Keywords: Sustainability, rice husk fiber agricultural waste, ceramic shell mould, fracture surface, strengthening

#### 1. Introduction

For ages, due to the outstanding chemical stability and high thermal resistance at high temperature, most ceramic material was chosen to be applied in the high-temperature investment casting industry. Investment casting technique produce dimensionally accurate CSm component with cost-efficiency compared to other modern techniques, i.e. die casting, sand casting, forging etc. [1–5]. Generally, a good CSm requirement involved an adequate strength and mould permeability to be operating at higher casting temperatures [2, 4, 6–8]. Ceramic material applications are limited due to their inherent brittle nature that simply induces the catastrophic failure, particularly during the handling and processing stage [9]. The brittle CSm can be improved by strengthening method of green loose CSm and densification of fired CSm. The most promising approach in strengthening and improving the fracture toughness of brittle CSm is forming a ceramic–matrix composite by introducing the toughening phase; particles [10], whiskers and fibers [11–13] to the base material. Fiber is the most recognized as a promising reinforcement with large aspect ratio respected to their versatile toughening mechanisms [4, 14–18] by introducing energy dissipating phenomena.

Basically, the strength of unfired CSm is depending on refractory particles bonded with colloidal silica which are in loose particles form. The most significant strengthening technique is incorporating organic fibers into the slurry, which acts as a composite reinforcing agent [1]. Usually, cracking mechanism easily occurred in CSm structure during the autoclave wax removal, which can be avoided via fiber-matrix incorporation. Inasmuch, green CSm strength also increases [4, 11, 12, 19]. High permeability also affectedly by the fibers addition, due to porosity networks from the fiber burned out. Carbon fiber and nylon fiber with excellent mechanical stabilities [18] disclosed a promising in CSm green strength [2, 6] but lack off fiber-matrix interfacial bonding due to smooth nature of fiber surface [16]. As a result, CSm endured weak body with only slight energy to pull out the fiber from the fracture CSm matrix. Thus, it has hindered these fibers utilization as reinforced material in ceramic matrix. Therefore, it may postulated that fiber chemical treatment can be conducted to produce fibers with much rougher surfaces that will allow for mechanical interlocking and improved the adhesion of fiber-matrix crystallinity. Similar phenomenon was proved by other researcher, as enhancement of composites strength can be achieved by improve the interfacial matrix-fiber bonding via chemical modifications [20]. Meanwhile, the fired CSm strength is depends on the firing time and temperature, bonded refractoriness particles, and binder sol gelation concentration [21]. Therefore, sintering is the most important and crucial part in consolidation of ceramic structure [22]. During sintering, diffusion mechanism promotes the interaction of pores and grain boundaries which leads to the grain growth. This phenomenon stimulated newly denser grain structure and also prompted the metallurgical bonds. Hence, the strength of the sintered CSm body is increased due to the bonding and particles necks growth. Numerous advantages have been foreseen in studies incorporated fiber in ceramic matrix for the fired strength advancement; i.e. carbon fibers [15] and SiC-graphite [23].

In the present work, similar behavior can be achieved with green waste fiber that is abundantly available, rice husk fiber (RH) an agriculture sources. Herein, we report the fabrication of CSm incorporated with RH fiber, CSm-RH. This organic fiber is biodegradable and known as a non hazardous material. RH not only can reduce the cracking of CSm but also can reduce the polution emission level that correlated to the industrial waste problem. Indeed, fired CSm with the existance of silica element in RH fiber able to generate very low thermal expansion coefficient, very rigid and strong at high temperature. In relation to this fact, higher silica content allow the mould to be operated at higher temperature. Nonetheless, new phase of zircon (ZrSiO<sub>4</sub>) was found initiated from fused silica phase based-RH fiber as reported elsewhere [24]. Ceramic shells composition are found to be influenced by these factors and eventually enhanced their mechanical strength.

#### 2. Methodology

Prior to the fiber treatment process, RHs were immersed in 5 % NaOH solution for 24 hours then rinsed with distilled water and dried in drying oven for overnight at 80 °C. RHs were sieved to get uniform size of 4 mm and then were mixed in slurry composition by 3 wt-% of slurry. Three types of shell system were prepared; CSm without fiber (CSm-WF) as a control sample, CSm with untreated rice husk fiber (CSm-RHU), CSm with treated rice husk fiber (CSm-RHT). The CSm was prepared by slurry coating followed by alumina and zircon sand coating via rainfall sanding technique by sprinkle it after wax pattern immersion in the modified slurry. The steps were repeated up to 5 layers with a controlled drying and finally subjected to dewaxing and sintering for final CSm matrix consolidation [24–26]. The surface modification of RHs and surface fracture of composites shell mould was characterized via Scanning Electron Microscopy (SEM, Jeol JFM-6380 LA, Japan). The mechanical strength of CSm were examined by three-point bending test via 5 kN load cell Instron according BS 1902, with fixed 70 mm outer span. The interconnected porosity of the CSm are measured by the gas membrane permeation testing unit. This test is generally according to the ceramic testing guidebooks from Investment Casting Institute.

# 3. Results and Discussions

# 3.1 Surface Modification of Rice Husk

The morphology of RHU and RHT fibers were characterized via SEM as shown in Fig. 1(a) and 1(b). RH fiber outer and inner surfaces images give a plain idea of the chemical treatment technique as a potential fiber surface modification. Noticeable, NaOH-RHT fiber acquired more surface modifications perfections owed to the rougher surface created rather than smoother RHU fiber surface. The hairs (trichomes) was observed on the RHU outer epidermis and detached for the RHT fiber [27]. Moreover, a ruptured protuberances and clear parallel grooves between the protuberances appeared on the RHT fiber surface suggested the effects of NaOH physical and chemical interaction.

Usually, the inner surface of RHU is quite smooth with wax and natural fats attached on its surface [28]. The treatment could have diminished these impurities for better adhesion properties. The surface roughness of RHT was significantly changed with some cracking particles suffered due the alkaline treatment. Obviously, the pores on the RHT surfaces giving a larger exposed surface area for the good interaction bonding with the shell matrix. Surface impurities seems to be extinct apart from the RH fibers, thus indirectly increases the surface roughness of the fibers or particles that give effects of the better surface contact area between fiber and matrix in the CSm. In fact, the similar surface modifications of natural fiber purposely carried out to improve the adhesion properties [28, 29]. Commonly the rough nature of the RHT surface associated towards the CSm strength respected to shell matrix-RH fiber interfacial bonding during the application of mechanical force.



Fig. 1 - SEM surface morphology of rice husk; (a) RHU and (b) RHT

#### **3.2 Toughening of Green Ceramic Shell Mould Matrix**

Fracture surface morphology of CSm samples was shown in Fig. 2 to analyse RH addition effects towards CSm matrix structure property. A scheme of the fracture surface condition of green CSm-WF is depicted in Fig. 2(a), showing the shell system is in brittle mode fracture that simply cracked with a low energy absorption. Indeed, cracks behaviour may spread rapidly accompanied by very little plastic deformation for green brittle CSm. Such cracks are unstable and crack propagation easily occurred consecutively, without an increase in the applied stress magnitude. There was a significant difference in the fracture mode when the matrix is reinforced with the RH fibers. As shown in Fig. 2 (b) and 2(c) (representing fracture surface for CSm-RHU and CSm-RHT, respectively), the fractures mode are in the mix condition of ceramic brittle and plastic state. The mechanisms of fracture resistance showed that fiber pull out from the CSm matrix due to mechanism of fracturing in toughening ceramic matrix.

The different in treatment process of the fiber surface also leads to the different fracture surface morphology. The incorporated RH fiber adhesion able to enhance the properties of the composites CSm as an ultimate binder or reinforcing agent within shell matrix by increasing the resistance friction between RH fiber and shell matrix. Therefore, more energy are needed for the fiber pull-out from the shell matrix thus the green strength of reinforced shell-fiber can be improved significantly as compared to the brittle CSm-WF [1]. A poor interaction of CSm-RHU matrix related to the condition of smooth surface occupied by the RHU surface indirectly weakens the fracturing strength. Also, the intact RHU shape and the holes created due to fiber pull-out suggested that RHU fiber unable made a strong interfacial contact with shell matrix and consequently cannot hold the brittle structure. Thus, contributed to eases the shell matrix fracture process in the brittle mode fracture. This limitation has been overcome by the rougher NaOH-RHT surface provided in significant yield fiber tearing showing the crack bridging within the CSm matrix, owed to strong interfacial bonding of RHT-CSm matrix. Moreover, due to this mechanism, the bonding formation induced greater strength during the mechanical fracture. CSm-RHT possess a better fiber-matrix interfacial due to the adhesion mechanism of CSm-RHT which resulted

in the interlocking mechanism between fibers and consequently increase the resistance in the fracturing process. Therefore, the fracture surface of CSm-RHT shows some plastic areas that indicate the obstacles in fracturing process. Rough surface of RHT fibers are fully connected the interfaces and become a tougher pathway for the crack propagation in ceramic matrix. The occurrence of crack debonding along RH fiber in the shell matrix is governed by the ratio of fracture energies of the fiber-matrix interface. Therefore, shell mould fracture toughness was believed enhanced by the toughening effect of RH fiber embedded in CSm matrix.



Fig. 2 - Morphology of green fracture surface and fiber-matrix adhesion of CSm system; (a) CSm-WF, (b) CSm-RHU and (c) CSm-RHT

# 3.3 Ceramic Shell Mould Strength and Mould Performance

Green and fired strength test of CSm are reported in Fig. 3(a) and 3(b). The green strength of CSm-WF, CSm-RHU and CSm-RHT were given by 1.16 MPa, 1.20 MPa and 1.34MPa, respectively. Greater green strength of reinforced shell moulds were influenced by the fracturing resistance in the ceramic matrix mainly in the embedded fiber zone (fiber bridging). The green fracture surface with embedded fibers in the CSm network suggested more energy is required to remove or break the fibers in the composite system which indirectly giving a greater strength of the composite matrix. Microstructure observation of the CSm-RHU fracture surface showed that most of the fibers are easily pulled out from the ceramic structure which represent by the smooth surface, indicates only a small energy needed to pull the RHU fibers out from the matrix. Thus, low green strength is recorded in the CSm-RHU.

A maximum green strength was obtained by CSm-RHT, which increases by about 15.52% from the standard CSm. The effective binding area correlated to rougher surface of RHT, giving a stronger interlocking fiber-matrix for better enhancement strength. During the resistance process of stress loading, a necking or tearing fiber behavior was observed as confirmed by SEM image and schematically illustrated in Fig. 2(c) which indicates failure delayed in CSm-RHT matrix. Microstructure examination of the CSm-RHT fracture surface which is in mixed mode condition with brittle and plastically pulled out indicates that some energy is required to pull out the fibers at the beginning that is represented by the plastic condition. Thus, this has ascertained that the incorporation of RHT in CSm body able to enhanced green strength by preventing the direct transferring loading to the shell structure. Whereas, 3.59 MPa, 3.78 MPa and 4.32 MPa for the fired strength of CSm-WF, CSm-RHU and CSm-RHT. The data indicates that fired body has higher CSm strength than the green body, respected to the existence of diffusion mechanism in sintering stage as fully matrix densification occurred and involves the development of new dense ceramic structure. Typically, a compact loose particles will undergoes the diffusion mechanism between each particle which leads to the stacking and binding of particles together resulted a develop strength. The capability of the RH fiber reinforcement was supported with the fired body strength enhancement as both reinforced fiber has a slightly higher strength than the unreinforced sample. The difference of fired strength between the CSm-RHU and CSm-RHT is correlated to the present of more pores in the sample with RHU fiber. It is therefore in agreement with Harun et al. [25, 30, 31], the un-removed lignin and others organic substance in RH fibers will easily burnt off and create more pores in the CSm fired body. A higher percentage of porosity distribution can drive off the strength of CSm-RHU body. Meanwhile, the existence of SiO<sub>2</sub> phase generated from RH ash able to harden and strengthen the fired reinforced mould [32]. In fact, a study has proved the addition of fly ash RH to slurry composition makes the slurry more hardened [4, 21]. Indeed, highest MOR value indicates the possibility of SiO<sub>2</sub> bonding in the fired CSm structure as supported by [24], proved that the existence of RHs fiber are not only purposely as reinforced agent in green body but existed SiO<sub>2</sub> is indirectly influence the properties of fired CSm.

Fig. 3 (b) shows the effect of RH addition in respect to the CSm permeability. CSm-WF possess the lowest permeability value as compared to the CSm-RH. The incorporated of RHU fibers dramatically improved the 30% of fired permeability of CSm-RHU (as compared to the standard CSm) by formed a linked porosity networks [33], displaced a burnt off RHU volatile material. The formation of sufficient pore structure is very significant during the pouring and casting process for hot air (molten gas) immediately and consecutively leaving the cavity to prevent unwanted trapping gases during the displacement of liquid metal as demonstrated in Fig. 3 (c). Thus, these results suggest that the filling process of molten metal can be improved by introducing reinforced fiber in CSm system accordingly to the increased porosity. Therefore, the distributed porosity not only contributes to the permeation mechanism but also can reduce miss-run and out-fill defects related to the air entrapment within the porous CSm.



Fig. 3 - (a) Flexural strength; (b) permeability of ceramic shell mould system of CSm-WF and CSm-RH; (c) schematic diagram of basic principle of metal investment casting process

# 4. Conclusion

This paper reports on preparation and characterization of CSm-RHs system with enhanced mechanical strength and permeability shell performance. The superior result of mechanical strength properties owing to good bonding properties also leads to the fewer coats layer that can be applied in the shell system. Toughening mechanism in CSm with fibers were proved as pull-out of RH fiber in CSm-RHU, crack bridging and interlocking mechanism in CSm-RHT. The presented work shows RH fiber has potential to improve the fracture toughness of green and fired body of CSm system and produce strong CSm system to suit in investment casting applications.

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