



Development and Characterization of Foundry Refractory Coating And Validation Through Factorial Design of Experiment

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Abstract

Foundry Refractory Coatings are used to improve the surface finish of grey iron castings and reduces the post cleaning and re-work cost to eliminate surface defects. Present research was concentrated on the qualitative and quantitative analyses of commercial coatings through X-ray Diffraction and Energy Dispersive Spectroscopy, respectively. A number of coating compositions were developed by the addition of refractory fillers, binders. The effect of particle size, which is the most important parameter was validated via Design of Experiment protocol. Ultimately, three types of coating formulation were prepared and characterized using X-ray Diffraction. Fe₂O₃ and SiO₂ were determined to the major phases in all coating samples. Particle size of 75 microns was selected as optimum to produce smooth surface finish. With these refractory coatings, the Gray iron castings were produced without inducing any significant effect on the mechanical properties and microstructural features. Finally, based on the experimental results, the formulation of a refractory coating is introduced that satisfied the required performance criteria.

Keywords: Foundry Refractory Coatings, Design of Experiment, Casting, Grey Iron.

1. Introduction:

In manufacturing industry, the surface finish of the cast components is an important parameter to assure quality and aesthetics. In sand casting process the surface finishing depends on the properties of sand and other factors i.e. component geometry, melt and-pouring temperature, pouring speed, time and component section size [1].

Mold or core coating is being extensively used in CO₂ and green sand molding for the production of high quality castings. The unavailability of proper sand grade and poor refractoriness of the mold and core surfaces confer the extensive use of Foundry Refractory Coating (FRC), which covers the voids

between the sand grains on drying and also provides the superior surface finish of intricate casting. The FRC provides a barrier to molten metal penetration to mold/core wall and to minimize surface defects i.e. metal penetration, veining/fining, mold erosion, metal-mold reaction, carbon pick up and blow holes etc. [2-3]. The application of FRC could improve overall casting quality through enhanced surface finish, reduced fettling and scrap cost [4]. Nwaogu et al. [5] concluded in their study that the refractory fillers in sol-gel refractory coating have considerable effect on the casting surface finish by filling the pores between sand grains thus exhibiting fair thermal stability.

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Two types of coatings are extensively used in foundries namely water and alcohol based. Most of the foundries preferred alcoholic based coatings due to quick drying time and elimination of mold drying before pouring. The preference of using alcohol based coating instead of water is also due to the reaction between the moisture and chemical binder of the core, which may lead to reduce the strength of core materials under tensile and flexural loading as reported elsewhere [6]. Harveth et al. [7] also depicted the importance of spirit based and compared with water-based coatings by comparing the color change versus moisture content on drying. Although the importance has given to continual improvement to casting shop floor but development in the core making, molding, pouring and cleaning processes is also necessary. Number of experiments, time, and overall development cost can be reduced by using the factorial design. Many researchers applied factorial design for the development of various processes in diverse applications [8-9].

In the developing world, the foundry sector faces many challenges i.e. sustainable supply chain and sudden change in the import policies. Therefore, the local development of FRC should be initiated. The local demand for refractory coating in the foundries has increased up to 50% in the last decade. However, there is no FRC available that could meet the intended properties i.e. low rejection of castings due to improvement in the surface finish and minimization of post-casting treatments that could save money, time and efforts. This research work aims to develop FRC at low cost that has improved performance and properties as demanded by various industries.

2. Experimental Work:

In this study three foundry refractory coating (FRC) samples of different origins were collected from various industries and designated as A1, A2 and A3. Fresh samples were in the form of suspensions, which were placed in an oven at 110°C to evaporate liquid carrier. Keeping in view, the high temperature of the mold/core, the dried A1, A2 and A3 samples were heated at 1000°C in a muffle

furnace for 3 hours. The dried FRC powder samples were designated as B1, B2, and B3 respectively.

The structural properties of FRC samples A1, A2 and A3 were studied using X'PERT PRO PANalytical X-ray diffractometer (XRD) equipped with Cu-K α 1 ($\lambda = 1.540\text{\AA}$) operated at 40 kV, 40 mA. For determining the particle morphology and chemical nature of ingredients, Scanning Electron Microscopy (SEM) (S-3700) coupled with Energy Dispersive Spectroscopy (EDS) was employed to analyze surface morphology and chemical compositions of the samples. The variation in the structure of coated samples before and after heat treatment was also evaluated by XRD analyses.

The XRD analysis were done on the powder samples containing all the constituents and mixed in the desired amount. Briefly the powder samples were ground in a ball mill for 8 hours to reduce particle size and for uniform mixing of constituents. The average particle size was determined by sieve analysis.

For the development of coating, design of experiment was performed using mini tab software to select the best combination of coating formulation. Factorial design of experiment was used to minimize the total number of experiments to attain the best coating combination. Numbers of features influence the process i.e. quantity of binder, filler and particle size distribution are studied. To change the amount of each variable is tedious and time consuming so factorial design of experiment can minimize these difficulties by optimizing all the other parameters. A total 16 combinations were tested on the basis of particle size, quantity of binder and fillers as given in Table 1. Additives and suspension agents were also included in such combinations and on the basis of binder type, 13 combinations were rejected. Only three coating formulations were selected after conducting design of experiment that was developed and practically capable of implementation in the grey iron foundry [10].

2.1 Practical implementation of Coating:

There are many advanced technologies for metal casting but the sand casting process is one of the

most widely used and conventional process. The advantage of using sand casting process is the low cost of raw materials, application to wide variety of casting with respect to size, composition and the possibility of recycling the molding sand. The green molding sand was composed of silica sand (86.8%), sodium based bentonite clay (10.3%), bituminous coal dust (2.4%), yellow dextrin (0.45%) and water as balance. This composition is analogous to the one reported by Carnin et al. [11] and actually applicable to wide variety of castings produced in many foundries. Green strength, compactness and hardness were determined according to DIN (Deutsches Institut für Normung) standards. The green strength of molding sand was measured to be 14.2 psi at 50% compaction whereas the minimum requirement for green strength and compactness is suggested as 11-15 psi and 45-52, respectively [2]. The hardness measured at the bottom of mold and

walls was 80 and 45, respectively.

Three recipes of the FRC were prepared and applied through spraying on the core surface and mold cavity of intricate 85kg casting. Grey iron scrap, coke, limestone and iron-silicon alloy were processed in a 5 ton capacity cupola furnace. The molten grey iron was poured into the green sand mold at 1400 °C in 19 seconds. After complete solidification and cooling to 630°C, the mold was sent to the casting shakeout section to clean the surface.

3. Results and Discussion:

The FRC was applied at the mold/core surfaces and dried in air. These coatings experienced thermal excursion at 1400°C when molten metal was poured. The coating filled the pores of the mold surface. The appearance of the surface became uniform and refine.

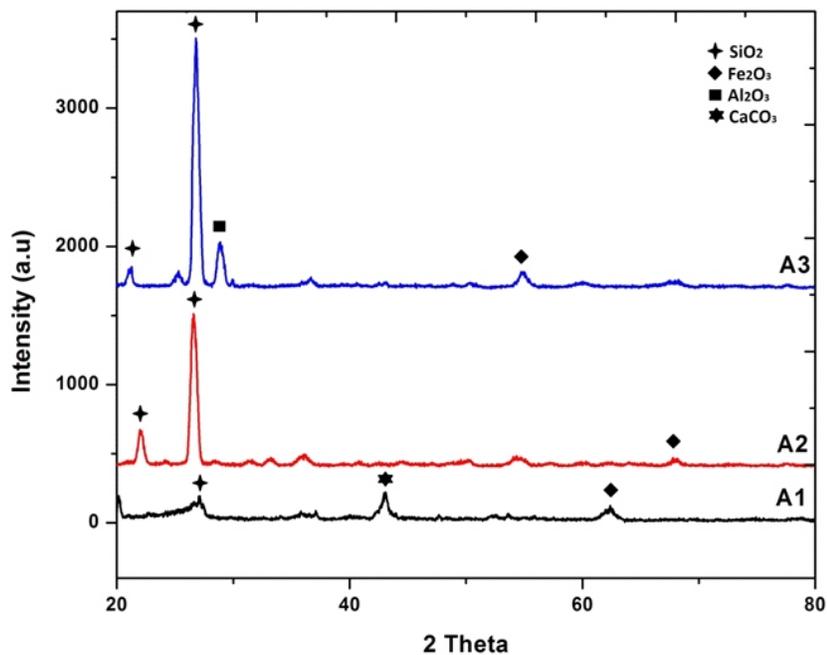


Figure 1: XRD pattern of the dry refractory coating

The X-rays Diffraction (XRD) pattern of dried FRC, labeled as A1, A2 and A3 are shown in Fig. 1. A sharp peak of SiO₂ was observed at 27° in these FRC which is also reported by Pavloviæ et al. [12]. The XRD spectrum of A1 (black curve) exhibited the presence of CaCO₃, whereas Al₂O₃ peak was originated in sample A3 (blue curve). The XRD

patterns of these FRC reflected the presence of Fe₂O₃ peaks associated with different crystallographic planes. Energy Dispersive Spectroscopy (EDS) results also confirmed the existence of Al, Si, O and Fe in these FRC and the results were comparable to the literature values [13].

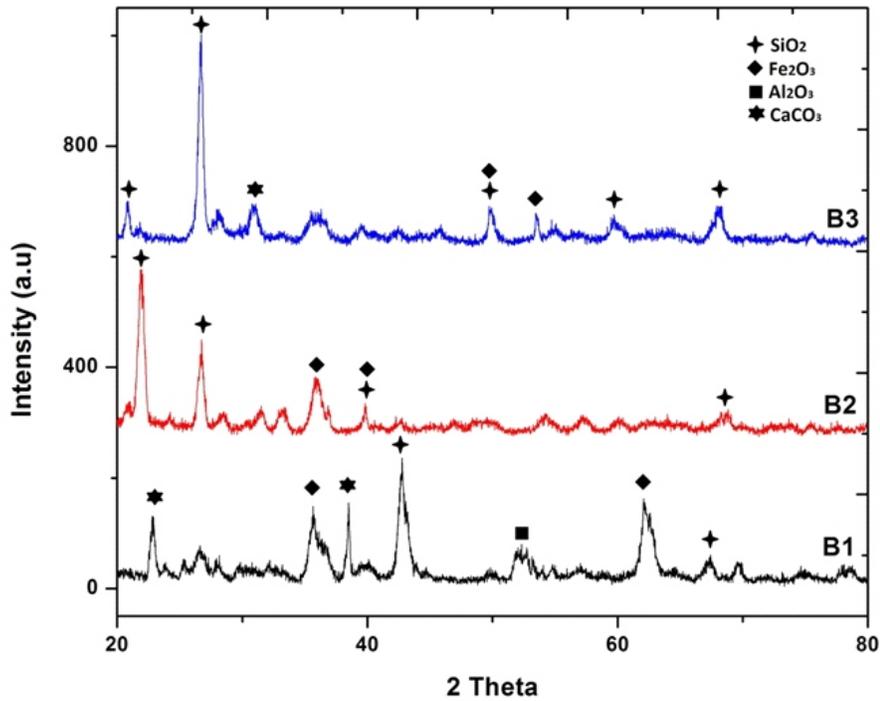
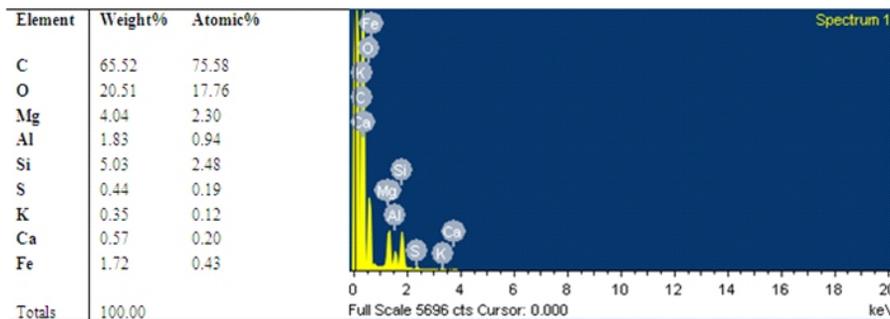


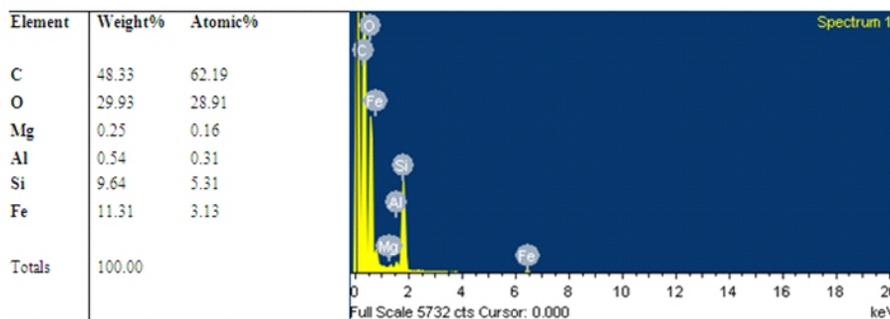
Figure 2: XRD pattern of the heat treated (1000°C) foundry refractory coating

Fig. 2 showed the XRD pattern of the ash produced from the A1, A2 and A3 FRC after exposure to 1000°C are designated as B1, B2 and B3, respectively. The sharp peaks of SiO₂ were observed in these heat treated FRC. In addition to such phases, all samples also revealed the presence of

Fe₂O₃ and Al₂O₃ corresponding to various crystallographic planes as indicated by diffraction peaks originated at various diffraction angles (Fig. 2). These phases could withstand high temperature leading to produce high temperature refractoriness [14].



(a)

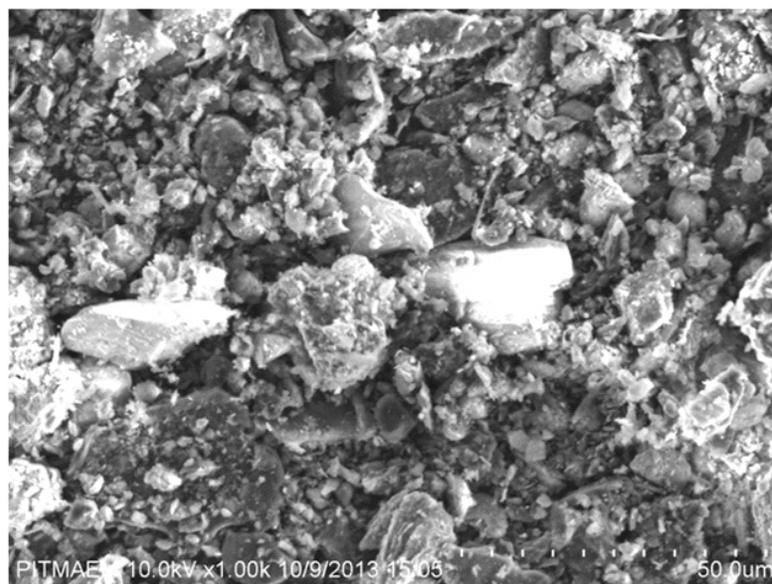


(b)

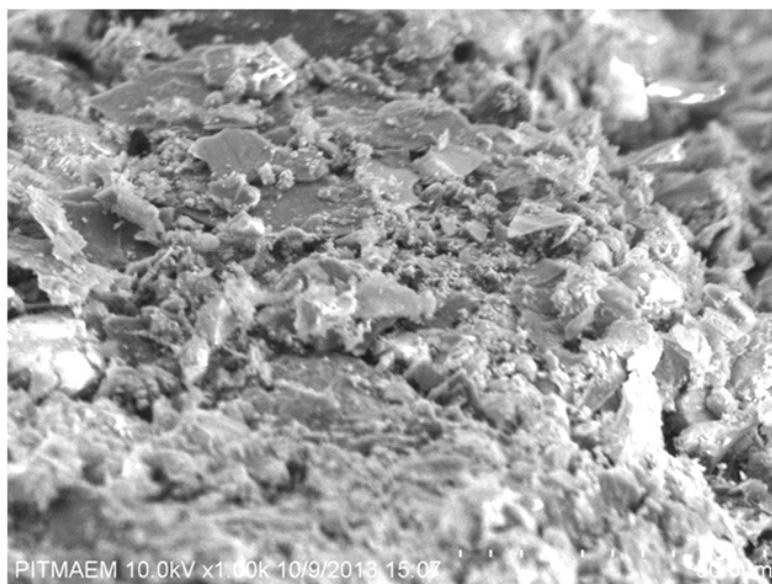
Figure 3: EDS analysis of dry refractory coating (a) and (b).

A series of commercial coatings samples were tested but only two coating samples are analyzed by EDS to estimate the approximate chemical composition. The XRD results of the FRC illustrated the existence of almost similar phases. The EDS analysis of A1 and A2 FRC samples are presented in Fig. 3 (a) and (b) respectively. The analysis showed the presence of oxygen and carbon elements in the heat treated FRC samples. The strong signals of Fe and Si are possibly associated with the hematite

(Fe_2O_3) and quartz (SiO_2), respectively. The largest signal of carbon in the EDS spectra were related to the graphite that was added to the FRC. The addition of graphite could effectively resist the metal penetration and could provide good surface finish by forming a stable colloidal suspension in alcohol. Similarly, Hematite was added in the FRC to reduce the carbon defects in the grey iron casting.



(a)



(b)

Figure 4. Morphology of the dried refractory coating, (a) A1 and (b) A2 samples.

The Fig. 4(a) and (b) exhibited the surface morphology of samples A1 and A2 respectively. The grain size of the FRC was non-uniform and particle shape was irregular. However, the fine filler particles could settle in the pores of sand grains and

may enhance the surface finish of the mold. It has been suggested in the literature that the shape and size of the filler particles in the FRC are always considered critical for the production of high quality casting of improved surface finish [15].

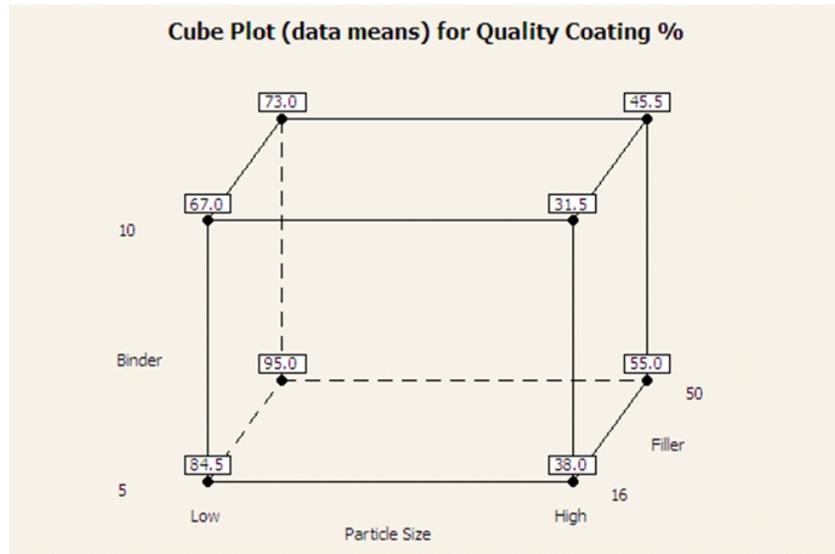


Figure 5: Cube Plot for Quality Coating.

The cube plot for the quality coating rating (%) was shown in the (Fig.5) contained eight corner points with different combinations of binder, fillers and particle size. Maximum quality (%) was observed at 95 that are considering best quality and low cost and the minimum quality (%) had been observed at

31.5 that were of very poor quality and high cost. The poor quality was attributed to improper and higher percentage of binder as sand particles may adhere to the casting surface. Similarly, all other corner points had different percentages of binder, filler and particle size.

Table 1: Factorial Experimental Design.

Std Order	Run Order	Center Pt	Blocks	Particle Size	Binder (%)	Fillers (%)	Quality Coating (%)
4	1	1	1	Coarse	10	16	30
3	2	1	1	Fine	10	16	65
8	3	1	1	Coarse	10	50	44
11	4	1	1	Fine	10	16	69
15	5	1	1	Fine	10	50	71
6	6	1	1	Coarse	5	50	52
2	7	1	1	Coarse	5	16	36
7	8	1	1	Fine	10	50	75
14	9	1	1	Coarse	5	50	58
16	10	1	1	Coarse	10	50	47
10	11	1	1	Coarse	5	16	40
12	12	1	1	Coarse	10	16	33
9	13	1	1	Fine	5	16	82
1	14	1	1	Fine	5	16	87
5	15	1	1	Fine	5	50	94
13	16	1	1	Fine	5	50	96

Table 2: Estimated Effects and Coefficients for Quality Coating % (coded units).

Term	Effect	Coef	SE Coef	T	P
Constant		61.19	0.7153	85.54	0
Particle Size	-37.37	-18.69	0.7153	-26.12	0
Binder	-13.88	-6.94	0.7153	-9.7	0
Filler	11.88	5.94	0.7153	8.3	0
Particle Size*Binder	5.88	2.94	0.7153	4.11	0.003
Particle Size*Filler	3.63	1.81	0.7153	2.53	0.035
Binder*Filler	-1.87	-0.94	0.7153	-1.31	0.226
Particle Size*Binder*Filler	0.37	0.19	0.7153	0.26	0.8
S=2.86138	Press=262				
R-Sq=99.09%	R-Sq (Pred)=96.36% R-Sq (Adj)=98.29%				

The complete Regression model for Quality Coating (%) is:

$$\text{Quality Coating (\%)} = 61.19 - 18.69(\text{Particle Size}) - 6.94(\text{Binder}) + 5.94(\text{Filler}) + 2.94(\text{Particle Size} \times \text{Binder}) + 1.81(\text{Particle Size} \times \text{Filler}) - 0.94(\text{Binder} \times \text{Filler}) + 0.19(\text{Particle Size} \times \text{Binder} \times \text{Filler})$$

Table 2 indicated that there was no significance of two-way interaction between Binder and Filler as well as three interactions between all factors (i.e., Particle size, Binder and Filler) effect on quality coating (%). But the two-way interaction of particle size with Binder and Filler was significantly affecting the quality coating (%) because p-values of

those interactions were less than 0.05. The p-values for all three main effects were less than 0.05 (Particle Size = 0.000, Binder = 0.000, and Filler = 0.000). The relative effect values (Particle Size = 37.37, Binder = 13.88 and Filler = 11.88) indicated the strength of effects (e.g. Particle size setting moved from fine to course than quality coating (%) decreased 37.37% etc.).

The $R^2=99.09\%$ and adjusted $R^2=98.29\%$ represented the percentage of variation in the quality coating (%) data explained by the particle size, Binder and Filler in the model while Predicted R^2 value indicated that the model could predict 96.36% future data.

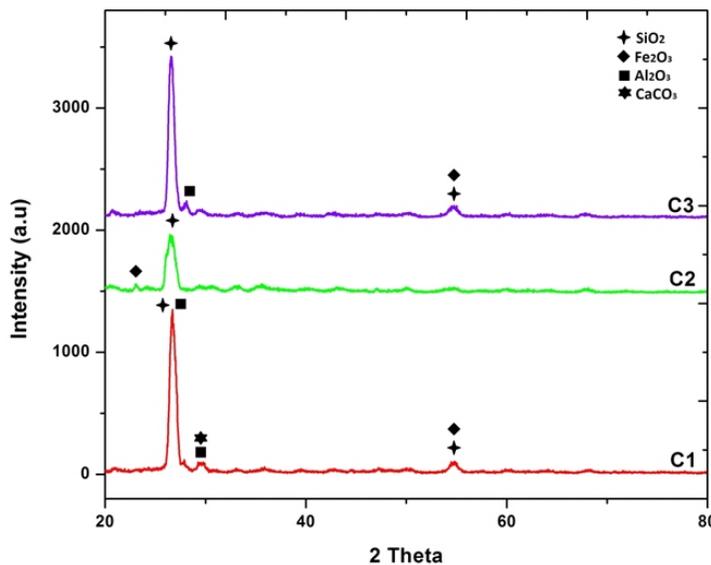


Figure 6: XRD pattern of developed foundry refractory coating.

The developed refractory coatings were shown in (Fig. 6). It was observed that SiO_2 was present in all coating samples and in unknown samples (Figs. 1 & 2). All three samples C1, C2 and C3 showed the presence of CaCO_3 , Fe_2O_3 and Al_2O_3 respectively.



(a) Surface Finish of C1



(b) Surface Finish of C2



(c) Surface Finish of C3

Figure 7: Surface Finish comparison of applied Foundry Coating (a), (b) and (c).

Practically prediction of casting defects in casting process is important factor in resource management that could be helpful to save energy, money and improvement in the production capacity of the foundries. Comparison of the surface finish of the three samples after application of refractory coating can be estimated visually as shown in Fig. 7(a), (b) and (c). The sand particles were adhered to the casting surface both in C1 and C2. The presence of sand particles on the surface of castings highlighted the ineffectiveness of the binder used in coating formulation. This may be due to the washed out of FRC during interaction with molten metal at a temperature approximately 1200°C . Additionally, rough casting could be produced by using the coarse particle size of the coating constituents. It can be seen that FRC (C3) presented a defect free casting having smooth and bright surface finish compared to other formulations.

3.1 Particle size Analysis of refractory coating

The average particle size of the FRC samples was determined by sieve analysis. Nwaogu et al.[6] reported that the average particle size of the coating constituents should be less than 100 microns for better surface finish. The coating constituent had an average particle size of 140 mesh number that was equal to approx. $106\ \mu\text{m}$ and in the bulk volume 200 mesh number which was around $75\ \mu\text{m}$ was

Both silica and alumina are highly desired constituents in the FRC due to its high coefficient of thermal expansion and high fusion point. Calcium carbonate is used as a filler material in FRC and is beneficial to produce smooth and bright finished casting.

determined. Defect free casting also has been produced by applying Mullite based refractory coating having particle size $40\text{-}45\ \mu\text{m}$ as reported by Presetic et al. [15].

3.2 Baume Test:

Hydrometer is used to measure the viscosity of the FRC samples before applying on mold and core surfaces. The spirit-based coating was mixed thoroughly before measuring the viscosity of the FRC slurry. The viscosity of the FRC was measured to be 13.

3.3 Effect of Coating on Microstructure, Mechanical Properties and Chemical Analysis:

The FRC was applied uniformly sprayed on the mold and core surfaces before pouring of molten metal. After complete solidification, fettling and short blasting, the universal component of tractor transmission case was sectioned, 200 mm UTS bar was drawn from 18 mm section thickness for UTS testing, mechanical properties, microstructure and chemical analysis.

Table 3: Chemical and Mechanical properties of Grey Cast Iron.

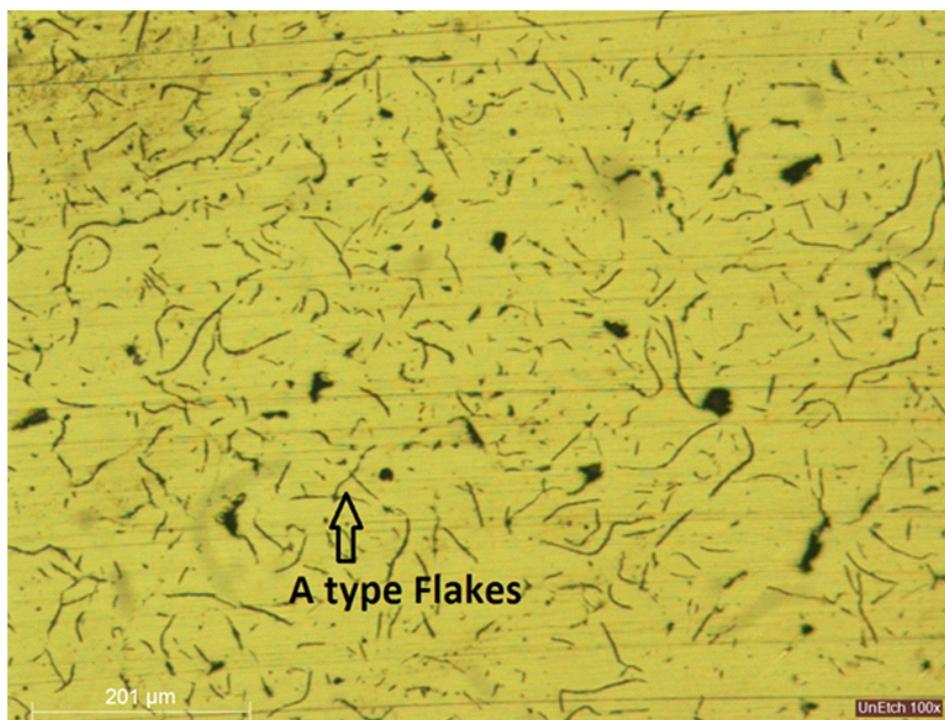
	Elements (Weight %)				
	C	Mn	Si	P	S
BS 1452 Grade 220	3.2-3.4	0.6-0.8	2.0-2.5	0.1-0.5	0.15 max
Observation	3.26	0.62	2.38	0.12	0.002
(a)					
Properties	Hardness (HB)	Ultimate Tensile Strength (UTS) N/mm ²			
BS EN 1561	179-229	220			
Observation	229	245			
(b)					

Table 3 illustrates the chemical composition and mechanical properties of the grey iron casting with desired specifications having good surface finish. In cast iron C, Si and P were the major elements based on Carbon Equivalent (C.E) and played important role during the formation of casting. Casting shows brittleness and chilling due to excess percentage of %P and %S respectively. The alloying composition was controlled during casting to produce BS 1452 Grade 220 castings. It is clear in Table 3 that FRC did not affect the chemical composition and

mechanical properties of the casting that is the matter of primary concern [20].

3.3.1 Microstructure:

The microstructure of Grade 220 (Grade 14) showed uniform distribution of A type flakes of random orientation as shown in Fig. 8. Presence of A-flakes is the requirement of grey iron casting. The microstructure of the cast iron was composed of 90% pearlite and 10% ferrite. Nwaogu et al. [5] reported that refractory coating had not produced any effect on microstructure of casting.

**Figure 8:** Microstructure Analysis of the Grade 14 at 100x (Unetch).

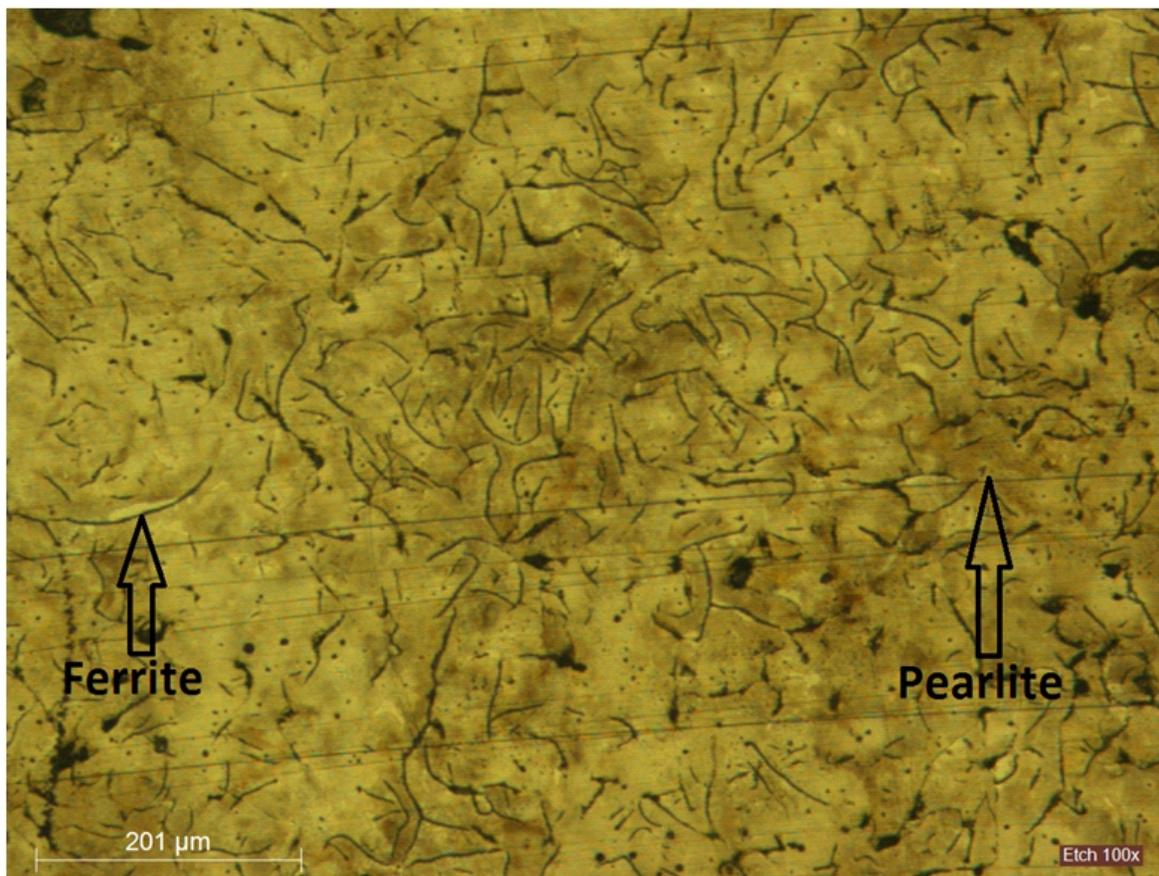


Figure 9: Microstructure Analysis of the Grade 14 at 100x (Etched in 2% Nital).

4. Conclusions:

X-ray Diffraction (XRD) results showed that all the coatings contain Fe_2O_3 and SiO_2 as the main components. By the use of the Design of Experiment strategy, the best combination of coating quality and low cost was selected. Particle size analysis of FRC illustrated the average particle size of $75 \mu\text{m}$ and found to be most suitable for improving the casting surface finish. This particle size is also helpful produce coating in suspension form in alcohol based liquid carriers. The applied FRC did not show any deleterious effect on the chemical composition, mechanical properties and microstructural features of the final cast metal which is the most promising quality of this FRC. The formulated FRC clearly showed the superior surface finish, which produced a defect free casting. The improved surface finish of the casting eliminated the expensive post-casting operations i.e. cleaning and shot blasting, which is the

inspiring feature of the FRC.

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References:

1. H. Jafari, M. H. Idris and A. Shayganpour, "Evaluation of significant manufacturing parameters in lost foam casting of thin-wall Al-Si-Cu alloy using full factorial design of experiment", *Trans. Nonferrous Met. Soc.*, 23, 2843-285,1 2013.
2. J. Thiel, M. Ziegler, P. Dzilekanski and S. Joyce, "Investigation into technical limitations of silica sand due to thermal expansion", *AFS Transactions paper*, AFS, Schaumburg, IL, USA, Vol. 07-145, 2007.
3. R.J. Brown, "Coatings for moulds and cores", *Foseco Ferrous Foundryman's Handbook*, 226-

- 230, Reed Educational and Professional Publishing Ltd, 2000.
4. U.C. Nwaogu, T. Poulsen, C. Bischoff and N.S. Tiedje, "Influence of New Sol-Gel Refractory Coating on the Casting Properties of Cold Box and Furan Cores for Grey Cast Iron", Proc. 69th World Foundry Cong., Hangzhou, China, October, Foundry Institute of Chinese Mechanical Engineering society, 651-652, 2010.
 5. U.C. Nwaogu, T. Poulsen, R.K. Stage, C. Bischoff, and N.S. Tiedje, "New solgel refractory coatings on chemically-bonded sand cores for foundry applications to improve casting surface quality", Surf. Coat. Technol., 205, 40354044, 2011.
 6. U. Nwaogu and N.S. Tiedje, "Mechanical Properties of chemically bonded sand core materials dipped in sol-gel coating impregnated with filter dust: novel approach to improve casting quality", Int. j. Metalcast., 25 (5), 307-317, 2012.
 7. L.R. Horvath and S.S. Bates, "Color change refractory coating technology as a quality control tool", AFS Transactions paper, AFS, Schaumburg, IL, USA, Vol. 06-070, 2006.
 8. R. Gottipati and S. Mishra, "Process optimization of adsorption of Cr(VI) on activated carbons prepared from plant precursors by a two-level full factorial design", Chem. Eng. J., 160, 99-107, 2010.
 9. V. Ponnusami, V. Krithika, R. Madhuran and S.N. Srivastava, "Biosorption of reactive dye using acid-treated rice husk: Factorial design analysis", J. Hazard. Mater., 142, 397-403, 2007.
 10. G.R. Henderson, "Process experimentation with two or more factors", Six Sigma Quality Improvement with Minitab, 2th edn, Chp. 8, 326-354, John Wiley and Sons, Ltd, 304-362, 2011.
 11. R.L.P. CarnIn, M.V. Folgueras, R.R. LuvIzao, S.L. CorreIa, C.J. da Cunha and R.S. Dungan, "Use of an integrated approach to characterize the physicochemical properties of foundry green sands", Thermochemica Acta., 543, 150-155, 2012.
 12. Z. AæImovI, æ-PavlovIæ, L. AndrIæ, V. MIlosevIæ and S. MIllcevIæ, "Refractory coating based on cordierite for application in new evaporatepattern castingprocess", Ceram Int., 37, 99-104, 2011.
 13. Y. Akbarzadeh, M. Rezaei, A. A. Babaluo, A. Charchi, H.R. AzImi and Y. Bahluli, "Microstructure, permeability and rheological behavior of lost foam refractory coatings", Surf. Coat. Technol., 202, 46364643, 2008.
 14. S. Kumar, P. Kumar and H. S. Shan, "Characterization of the refractory coating material used in vacuum assisted evaporative pattern casting process", J. Mater. Process. Technol., 209, 26992706, 2009.
 15. A. PrstIæ, Z. AæImovIæ-PavlovIæ, A. TerzIæ and L. PavlovIæ, "Synthesis and characterization of new refractory coatings based on talc, cordierite, zircon and mullite fillers for lost foam casting process", Archives Metall. Mater., 59, 89-95, 2014.
 16. B.A. Wills and T.J. NapIer-Munn, "Sieve Analysis", Minerals Processing Technology, 7th edn, Chp. 4, 92, Elsevier Science & Technology Books, 2006.
 17. M. K. Joyce, M. Rebros, S. M. Ramratan, "Comparing alternate foundry refractory coating measurement systems", AFS Transactions paper, AFS, Schaumburg, IL, USA, Vol. 08-126, 2008.
 18. "Specifications of grey iron castings" BCIRA, BS 1452:1977, 1981.
 19. "Founding-Grey Cast Irons", BS EN 1561:1997.
 20. S. H. Avner, "Introduction to Physical Metallurgy", 2nd edn, Chp.11, Cast iron, 423-446, Tokyo, McGraw-Hill, 1974,
 21. N. M. Rafael and Z. Michael, "ASM Hand Book", 'Casting', 9th edn, Volume 15, 1372-1375, 1988.