Metal-Ceramic Shell Interactions during Investment Casting

C. Mahimkar, V. L. Richards, and S. N. Lekakh
Missouri University of Science and Technology, Rolla, Missouri

Copyright 2011 American Foundry Society

ABSTRACT

Interactions of liquid steel with preheated ceramic shell molds can adversely affect the surface quality of investment castings and increase casting cleaning and finishing expenses. This phenomenon was studied using a special cube-shaped specimen with a deep pocket. The temperature field in the specimen and shell during the casting process was simulated with MAGMASoft. A foam pattern was used to form investment casting shells prepared in the Missouri S&T Laboratory with three different prime coats: silica, zircon and alumina. For comparison, shells prepared around the same specimen were obtained from three industrial foundries. After foam pattern removal and firing, the shells were preheated to 800°C (1472°F) and poured using HY130 steel. Shell samples in contact with steel were taken from the pocket region of the castings, polished and SEM/EDS analyzed. Multiple interaction products included complex Mn-Si-O, Al-Si-Mn-O and Fe-Si-Mn-Al-O oxides were identified. Formation of interaction products was also forecast using thermodynamic phase equilibrium in the system: liquid steel-ceramic-slag-gas. The experimental results are discussed with respect to thermodynamic predictions. The results can be used for shell material selection.

Key words: Ceramic shell, liquid steel, interactions

INTRODUCTION

Investment casting process is now used widely for production of small and medium sizes of precision steel castings having complicated geometry for different applications. The process starts with dipping foam or wax pattern in the slurry made of binder and flour (fused silica, zircon, alumina etc.). Refractory powders (crushed stucco) are applied to the slurry-coated pattern. Each shell coat is allowed to dry for three to four hours depending on the humidity of the surrounding atmosphere. The shell building process consists of one or two prime coats, four to five back up coats and a seal coat. Then pattern is removed from the shell by melting wax or thermal decomposition of foam and shell is cleaned from pattern residue and strengthened by firing. At the same time, sintering of ceramic takes place to give a structure to the investment casting shell that has enough strength to hold the pressure of liquid metal. Over the years, the process has evolved in order to have better surface finish and thereby reduce the cost of finishing and machining. When steel is poured into preheated ceramic shells, the prime coat of the shells comes in contact with the melt and its oxides. Thus, there is the possibility of melting and/or chemical reactions at the mold-metal interface. Metal-mold interactions have been studied for decades in the casting industry but most of the work was dedicated to study the specific burn-in/burn-on surface defect formation when using green sand and no-bake sand molds. Gilliland found the mold material at or near mold-metal surface often becomes a burnt sand layer, which has experienced temperatures equal to or very close to solidus point of metal being cast. He conducted experiments by pouring gray iron, ductile iron and steel into sand molds and observed interface reactions in hot and cold regions. Examination using energy dispersive microprobe analyzer showed Fe, Mn, Si, P, Ni as major components of surface defects. Brooks, Beckermann and Richards developed a method to predict burn on and penetration defect locations using casting simulation. The suggested method for burn-on prediction is based on the simulation of the locations where mold is above certain critical temperature, generally above steel solidus temperature while adjacent casting regions have not yet solidified. Colligan, Van Vlack, and Flinn studied factors affecting metal-mold interactions by casting AISI 1080 steel in green sand molds and resin bonded sand molds with different Mn contents and explained the phenomenon based on melt penetration, interface reactions and a specific Mn addition problem. Zhang and Morin studied effect of inhibitor gas on mold-magnesium reactions in investment casting. Auger electron spectroscopy revealed a reaction layer occurring on the cast part surface in which elements Mg, Al, Si, S, O were present.

Most of the work done so far is based on green and resin bonded sand. This paper deals with study of steel-ceramic shell interaction products.

PROCEDURES

Industrial trials of large complex castings showed that ceramic shell material in the deep pocket regions of the castings adjacent to heavy sections is most difficult to remove during break out of the shell molds. Hence, it is necessary to study the interaction products in this type of region. A special 4"x4"x6"cube shape specimen with ½" side wall thickness and 1½” bottom thickness was designed as shown in Figure 1. The vertical downspue was attached to the bottom of the cube. Vent was attached to prevent gas entrapment and fill the shells completely during the pour.
MAGMAsoft software was used for simulation. To determine the temperature history in the prime and the back up coats as well as in casting, three control points were placed in the shell and one was placed in the casting near the corner of internal pocket as shown in Figure 2a. The one in the casting denotes the temperature of the solidifying steel; the other three monitored temperature in the prime and back up coats at points placed 0.5 mm, 3 mm, and 6.5 mm from casting surface respectively.

User defined thermal property databases were created for steel (HY130) and fused silica based investment casting shell. A fine mesh was selected for the shell to predict the temperature at the control points accurately. The simulation was started at 1650°C (3002°F) tap temperature and shells preheat temperature at 800°C (1472°F) and finished when casting completely solidified (Figure 2b).

Simulation results predicted that the temperature of the prime coat of the shell goes above the liquidus temperature of the steel while the adjacent casting region still has some amount of liquid phase. The combination of these conditions increases the probability of steel-shell interactions in the internal pocket region being studied (Figure 3).

MODELING SOFTWARE

Liquid HY130 steel in equilibrium with prime layer (solid phase), and limited amount of oxygen (gas phase) was used to study the possible formation of slag layer (liquid phase) on the steel. Thermodynamic modeling was done using FactSage software to study the amount and compositions of the possible liquid products of the reaction. Fact-Felq, Fact-Stel and Fact 53 databases were selected. Preliminary calculations showed that pure steel do not react with pure oxides in the shell at pouring temperatures in inert gas atmosphere. The possible liquid products on metal – ceramic mold interface can be formed when atmosphere contained oxygen. Calculations for this case were done in two steps. In step one, the equilibrium composition of slag phase formed on liquid steel surface in contact with 1.5% O₂ was calculated. The second model was run considering reaction of liquid slag, formed in step one, with solid prime coat material (silica, zircon, or alumina) to check predicted liquid/solid products formation.
EXPERIMENTAL PROCEDURE

Three types of shell with different prime coats were prepared in Missouri S&T Foundry. Dipping slurries for the prime coat were made of colloidal silica binder in all cases in mixtures with three different types of 200-mesh flour: silica, zircon and alumina. These are respectively referred to as silica, zircon, and alumina prime coats. Fused silica stucco (50-100 mesh) was applied directly on the prime coat in all cases. All of the other five coats used fused silica based slurry with the same colloidal silica as a binder and applying 30-50 mesh silica stucco. Finally, one seal coat was given to all shells. In addition, shells prepared around the same pattern were obtained from three different commercial foundries A, B, C. All shells were fired to 900°C (1652°F) for 3 hours at Missouri S&T Foundry. All these foundries used some amount of zircon in mixture with silica in prime coat. Figure 4 shows the shells prepared at Missouri S&T Foundry as well as industrial foundries.

Low carbon, alloy steel (HY130) was melted in 100 lb induction furnace. A cover of argon gas was maintained on top of the melt to prevent reaction of liquid steel with atmosphere. Melt was poured into ladle and deoxidized using 0.06% aluminum followed by 0.06% Ca-Si wire. Shells were preheated to 800°C (1472°F) before pouring. Two heats were conducted. Shells prepared at Missouri S&T Foundry were poured in first one and industrial shells in second one. Chemistries of the HY130 steel in two heats are shown in Table 1.

![Fig. 4. Shells before pouring (a) and castings after pouring (b).](image)

| Table 1: Chemistry of HY130 Steel in Two Heats. |
|-----------|-----------|-----------|---|---|---|---|---|---|
|           | C         | Si        | Mn | Cr | P  | S  | Ni | Al |
| Heat 1    | .17       | .52       | .63| <.001 | .005 | 4.1 | .06 |
| Heat 2    | .17       | .50       | .64| <.001 | .006 | 4.1 | .06 |

After the castings had solidified, the shells were knocked off except from the pocket region. Figure 4e shows the castings with ceramics intact in the pockets of region cube castings. Mixture of epoxy resin and hardener was poured in these pockets and kept for 10 hours to get it hardened. Epoxy helped to keep the ceramic shell to be in contact with steel during sample preparation. After being stabilized by the epoxy the samples of the steel/ceramic shell interface were cut from the pocket region and mounted again in epoxy in order to have the specific sample size for ESM/EDS analysis. Samples were then polished from 180- to 800-grit SiC paper; afterwards they were polished with diamond paste 3, 1 and ½ micron using low speed cutting fluid. Samples were then carbon coated to decrease electrical charging under Scanning Electron Microscope (SEM). SEM/EDS ASPEX PICA-1020 system (Particle Identification and Characterization Analyzer) was used to observe interaction products.

RESULTS

Figure 5 shows interaction products observed for silica prime coat shells. Al-Si-Mn-O phases were observed at the exact corner region (in the right-angled portion) Pictures clearly show their penetration in the shell. Fe-Si-Mn-O phases were observed near corner region but slightly away from it. Figure 5 also shows that reaction product had no penetration in steel and sited on the shell side, some time penetrated deep inside ceramic coat. Al-Si-Mn-O phases had more tendencies for penetration when compared to Fe-Si-Mn-O phases.

Figure 6 shows reaction products observed for alumina prime coat shells. Al-Si-Mn-O phases were at the exact corners similar to silica prime coat shells. It can be seen in Figure 6 that they have penetrated more into the shell. Fe-S-Mn-O phases were slightly away from the corner but even they can be seen at the higher depths.
Fig. 5. Silica prime coat shells interaction products: (a) Al-Si-Mn-O, (b) Fe-O-Si-Mn phases.
Figure 7 denotes the interaction products observed for the shells with zircon prime coat. Fe-O-Zr-Si products were observed which showed there has been some melting of the prime coat at the interface. These phases were observed only near the contact surface between casting and shell. Another type of product observed distinctly was Mn-Si-Al-O phase, which was in the corner region. This phase was found near surface and inside the prime coat (Figure 7b).
Figures 8-10 show the interaction products observed for Industrial foundry shells Foundry A, B and C respectively. Similar to results observed for the Missouri S&T shells Al-Si-Mn-O products were observed to be present at corners for foundry A and B with depth of penetration of it being more in case of foundry B. Fe-Si-O-Mn products were observed in case of all three foundries with maximum penetration in case of foundry B and least in case of Foundry C.
Fig. 8. Interaction products (a) Al-Si-Mn-O and (b) Fe-Si-Mn-O phases in silica prime coat shells (Foundry A).
Fig. 9. Interaction products (a) Al-Si-Mn-O and (b) Fe-Si-Mn-O phases in silica prime coat shells (Foundry B).
DISCUSSIONS

THERMODYNAMIC PREDICTION

As was mentioned above, liquid low and medium carbon steel does not directly react with pure oxides (silica, alumina, and zirconia) at pouring temperatures in inert atmosphere. Situation changed significantly when surrounding atmosphere during pouring contains oxygen. In this case, steel will spontaneously react with formation of different reoxidation products. Reoxidation products were denoted in this paper as “slag” for the purpose of thermodynamic modeling. A first model was run in modeling software for prediction of the possible slag composition, which could be formed when liquid steel comes in contact with the oxygen from the atmosphere during mold pouring. This thin oxidized layer on liquid steel surface will contain a mixture of oxides of the metallic components in the ratio of 40-50% Si, 10-20% Fe, 20-30% Mn with small amount of Al and Cr oxides. The reoxidation slag thus formed has a long solidification interval and definitely could be liquid above the solidus temperature of steel (Figure 11). The reoxidation product (slag) also has a high solubility for the shell component oxides. The main solid phases in solidified slag (steel reoxidation product) are solid solution \((Fe,Mn)SiO_3\) and solid solution mullite type \((Al_2O_3-SiO_2)\). This type of liquid slag can readily react with prime coat during melt cooling and casting solidification in the shell.

The second equilibrium model was run to predict the complex reactions, which could take place between liquid steel reoxidation slag, and solid prime coats consisted of silica, alumina or zircon. The main goal of these calculations was prediction of possible liquid product formation at temperatures in the prime coat predicted from thermal modeling. If a liquid reaction product predicted to be formed has larger potential solubility for the solid ceramic it will be an indication of the severity of the possible metal/mold interaction. The predicted possible reaction products are shown in Table 2. Silica ceramic can readily react with the slag containing \(Mn\) and \(Fe\) oxides at pouring and steel solidification temperatures with formation of liquid phase. This liquid phase has possibility of dissolving of a large amount of solid silica from the shell without solid precipitates. Reaction of reoxidation slag with alumina also creates liquid phase region stable at pouring and steel solidification temperatures. Only reaction of reoxidation slag with zircon produces a lower quantity of stable liquid phase at 1550°C (2822°F). For comparison of possible behavior of different prime coats in contact with liquid reoxidation slag, the calculated fraction of solid product formed was plotted versus ceramic shell/slag ratio (Figure 12). These thermodynamic calculations were done at maximum predicted temperature in the shell (1550°C). Different shell/slag weight ratios were used as inputs. To the right of equilibrium curve in Figure 12 contained some amount of solid precipitates and left field is liquid slag. This represents saturation of the liquid slag. Increasing fraction of solid product and lower shell/slag ratio decreased the possibility of interface reaction. Steel reoxidation slag can assimilate the significantly larger amount of silica and alumina prime coats when compared to zircon coat. When solid product is predicted to form as a large
fraction of the final reaction product (more than 0.2-0.5), the dissolution reaction of prime coat by reoxidation slag will be significantly decelerated.

The experimentally observed chemistries of phases in interaction regions were compared with the thermodynamically predicted reaction products and similarity was conformed.

Pouring temperature has an important effect on intensity of formation of interaction products. It is important to note, that reactions take place when shell temperature in particular location rises above steel solidification temperature. More superheat will result in greater amount of liquid slag formed resulting in reactions at the interface as well as higher shell temperatures during pouring and solidification. Hence, care must be taken to minimize superheat as far as possible before the shells are poured. Casting geometry, for example deep pockets connected to hot spots in casting body, can presumably have more surface reaction products. This will largely depend on the porosity in the prime coat. In Investment casting the value of the optimal porosity of the prime coat is important because some level of porosity is necessary for overall shell permeability. From other side, high porosity could intensify penetration of reaction product deep into prime coat.

It was observed that interaction products and their depth in the shell was more for materials silica and alumina in the prime coat. For the shells with zircon prime coat, both interaction products and depth were less. At the same time, industrial practice and shelling procedure significantly affected on depth of reaction product formation in prime coats comprised zircon flour. Finally, Figure 13 shows the maximum depth of location of reaction products in studied shells. The different results for industrial shells were a combination of prime coat composition and shell porosity.
Table 2: Equilibrium Model Prediction for Interaction Products by Modeling Software.

<table>
<thead>
<tr>
<th>Reactions</th>
<th>Interaction product</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid steel reoxidation slag + SiO\textsubscript{2} prime coat</td>
<td>Liquid slag + SiO\textsubscript{2} + (Fe\textsubscript{x}Mn\textsubscript{1-x})SiO\textsubscript{3} + mullite (Al\textsubscript{2}O\textsubscript{3}-SiO\textsubscript{2}).</td>
</tr>
<tr>
<td>Liquid steel reoxidation slag + Al\textsubscript{2}O\textsubscript{3} prime coat</td>
<td>Liquid slag + Al\textsubscript{2}O\textsubscript{3} + (Fe\textsubscript{x}Mn\textsubscript{1-x})SiO\textsubscript{3} + mullite (Al\textsubscript{2}O\textsubscript{3}-SiO\textsubscript{2}).</td>
</tr>
<tr>
<td>Liquid steel reoxidation slag + ZrSiO\textsubscript{4} prime coat</td>
<td>Liquid slag + ZrSiO\textsubscript{4} + (Fe\textsubscript{x}Mn\textsubscript{1-x})SiO\textsubscript{3} + mullite (Al\textsubscript{2}O\textsubscript{3}-SiO\textsubscript{2}).</td>
</tr>
</tbody>
</table>

CONCLUSIONS

Metal-ceramic shell interaction products were studied in investment casting of low carbon alloy steel. Al-Si-Mn-O and Fe-Si-Mn-O were observed as the most common interaction products at the interface between shell and steel. Depth of penetration depended on composition of prime coat and shell porosity at constant pouring temperature and casting geometry. Shell with zircon in the prime coat had least amount of interaction products and less depth of reaction. Described mechanisms of formation of interaction products and factors affected of severity of reaction can be considered for prevention of surface defect formation in investment casting.

ACKNOWLEDGMENTS

The authors would like to thank U. S. Army Benet Labs for funding this research through contract number W15QKN-07-2-0004. The authors wish to acknowledge participating foundries for sending the shells for this study. The authors would also like to thank undergraduate helpers Jamie Fitzgerald and Tom Towey for their help in sample preparations.

REFERENCES