A plethora of bonding systems has been developed for monolithic refractories either as proprietary systems or commercial products. This paper will provide a state of the art review of the different bond systems via an extensive literature review. A comparison of different bond systems for alumina-based monolithic castables will be given showing the different comparative characteristics for each system and their advantages as well as the trade off and constraints will be discussed.

1 The evolution of castable bond systems

The subject of refractory binders, which form refractory bonds, is vast, ranging across bonds derived from vitrification and ceramic reactions commonly found in fired refractories to specifically developed binders found in monolithic refractories and capable of forming in situ bonds. Literature references indicate that the first monolithic as “in situ” refractories can be traced back to at least the 19th century [1]. These often relied on clay or other minerals to provide a cohesive force after drying and firing. An early example was in 1856 when H.S.C. Deville prepared a refractory crucible using alumina aggregate and compound based on marble and lime [2]. It wasn’t until the 20th century that monolithics with specific and purpose-designed bonding systems started to emerge. Early examples include the first plastic monolithics [1] that were made by Schaefer in the early part of the 20th century prior to 1920. One of the early binders for refractory monolithics was calcium aluminate cement (CAC) and its industrial production was patented by Lafarge in 1908 [3]. The first references of a commercial refractory concrete can be found around 1922–1924 where work in both France and the USA led to the development of Bauxite/ Ciment Fondu® based compositions [4, 5]. During the early days of refractory concretes, the main aggregates available for use were calcined clays and crushed fired refractory bricks. By the outbreak of World War II refractory concrete using calcium aluminate binders was well established in many industries that used furnaces and ovens [6].

The first major sign of binders producing a bond via a chemical reaction appeared in the 1950s which saw the publication of a definitive review of phosphate bonding by Kingery [7]. By the 1950s–1960s, castables bonded with high-purity CAC or phosphates and using high-purity aggregates were common [8]. Developments of systems based around reduced calcium aluminate binders in deflocculated castables continued during the 1970s [9] along with a diversification of the types of different bonding systems. Lime-free castable binder systems originated with the arrival of the first phosphate-bonded systems. This was followed in the 1980s by the development of alumina-bonded systems using rho alumina [10] which was first reported in Japan. Other systems have been developed based upon resins, clay minerals and silicate bonding systems [11]. More recently, a form of silicate bonding has developed via the use of silica sols. Once again, the literature reveals that this was first reported in Japan [12–14] in the 1980s with a similar concept being

promoted in the USA in the 1990s [15] and more recently in Europe in the last decade [16]. This evolution is largely driven by the quest to eliminate lime as a route to enhance corrosion resistance. Better corrosion resistance is reported [17–19] for cement-free systems but physical and mechanical properties often remain superior with cementitious systems. A second value driver is based around the ability to dry out such systems at higher heating rates than classical castables. Current refractory technology now covers a huge range of castables that can be applied by a wide range of installation technologies and are suitable for an ever-increasing range of applications. The types of bonding systems in use can be segmented into a number of broad categories [1] and within each category there exists a plethora of different options:

- Ceramic bond – develops on firing of which clays are the most common type and may include glass and or ceramic bond. Other additives such as boron compounds can be added to enhance sintering [17].
- Hydraulic bonding – the most well-known and common hydraulic binder being calcium aluminate cement. Other bonding systems are based on hydratable alumina.

Keywords: monolithic castable refractories, bond systems
Hydraulic binders are characterised by their capacity to form hydrates when water is added which provide cohesive bonds up to the point that ceramic bonds form. Other hydraulic systems such as ordinary Portland cement and magnesium oxychloride (Sorel cements) have also been tried, largely unsuccessfully as precursors of refractory bonds.  

- Chemical/Mineral bonds – these include phosphates, silicates, sulphates and bonds derived from different acid-base reactions. The family of alkaline activated binders (also known as geopolymers) could be considered here though they are subject to a certain amount of hype they do have significant drawbacks in refractory applications.  
- Sol-gel bonds – given the emerging importance of colloidal systems they are cited as a specific category. The most common being alumina and silica sols.  
- Other – these include different organic binders derived from resins as well as mineral systems. A review of patent literature over the last 15 years reveals more than 30 different patents claiming specific bonding properties of a wide range of proprietary bond systems. Due to a lack of available literature they won’t be treated in any detail in this paper. In addition, the focus will be on essentially inorganic binders so for example, binders such as ethyl silicate or coagulation casting using enzymes will not be considered.

2 Review of different bond systems

It is impossible to cover all bond systems in such a review paper given the multitude of different bond systems that are currently in use. Therefore use was made of different literature searches [20] and via the American chemical society software known as SciFinder®-CAS [21] up to the end of 2012. This software provides a powerful search tool of published literature according to key words and concepts around the key words. The search across the different sources revealed more than 20 different bond systems including patented compositions for refractory/heat resistant applications. Specific proprietary bond systems have been excluded from this paper as have specific patented compositions. The results based on these filters are presented in Fig. 1 which shows the frequency of scores according to different key words. A total of 1178 articles referenced monolithic castables and of these 548 were based upon CAC containing systems. Thereafter an increasing interest is seen for colloidal systems with over 60 papers found with most of these occurring in the last 10 years. This literature review provides an orientation as to the most important bond systems within the family of alumina castables and hence the decision to focus on a limited number of castable bond systems, namely the most common CAC and other selected systems such as hydratable alumina, phosphate and colloidal systems.

2.1 Hydraulic bonding – calcium aluminate cements

The bonding capacity of calcium aluminates comes from their ability to form hydrates via a reaction with water [22, 23]. The different anhydrous phases present in a given calcium aluminate cement (CAC) depend upon a number of parameters of which the chemistry is the most important [22]. The dominant phase in all cases is CA, calcium mono-aluminate. The impact on castable properties due to these hydration reactions is as a function of the specific type of formulation logic employed:

- Conventional castables are placed with water, ranging from 8 to 20 %. The combination of the water and cement to form calcium aluminate hydrates results in the hardening of the castable. The majority of water is concentrated in the matrix between the fine particles and reacts with the cement to form the hydraulic bond. Upon heating, the hydraulic bond dehydrates, re-crystallizes above 900 °C and new mineral phases are formed via ceramic reactions. At service temperature (1200 °C) low melting phases CAS and C₂AS are formed when the castables contain natural aggregates and the presence of these liquid phases restrains the thermo mechanical performance.
- The 1970s saw the first era of the low cement castable (LCC) [24]. In the LCC system, the CAC cement is part of a complex binder system. This system can be viewed as an interdependent triangle of fine submicron fillers such as silica fume and additives which all play a role in the binding system. The reduced lime content and the fine oxide powders which favour the formation of a fired bonding matrix results in improved hot strength, higher thermal shock resistance, lower porosity, and increased corrosion and abrasion resistance in comparison to conventional castables.
- This evolution continued in the 1980s when concretes were produced with very low cement contents down to 2 % or less and are now known as ultra-low cement castables (ULCC) [25–26]. Ultimate performance with these products is enhanced via increased refractoriness (with lime contents as low as 0,2 %). Silica and alumina present in the matrix in ultra-low
cement formulations react with one another and precipitate mullite at temperatures above 1300 °C. This mullite formation increases hot strength considerably.

- Since then the number of different castables using CAC bonding within deflocculated castables has multiplied. The late 1980s and 1990s can be characterised by the development of new installation technologies where low cement castables could be placed by pumping and or self-flowing techniques [27]. This was followed by the era of wet gunning or shotcreting [28–29].

2.2 Hydraulic bonding – hydratable alumina

Hydratable alumina otherwise known as p (rho)-alumina is a transition alumina with a specific crystallinity. In the presence of water, rho alumina will form AH₂ gel and AH₃, gel. The features of these binders are summarised in Fig. 2 constructed from the literature [10, 30, 31–35].

2.3 Phosphate bonding

The use of phosphate bonding in refractories is well established and has been used for many years in shaped and non-shaped refractories, including castables [36, 37]. The subject of phosphate bonding is vast with many phosphates available which have differing active mechanisms and consequently differing refractory applications. Generally, two types of phosphate binders can be considered, one uses phosphoric acid [38–41] while the other is based on metal-phosphate compounds. This is summarised in Fig. 3.

2.4 Colloidal bond systems

A colloid or sol is a stable dispersion of particles in water [42]. Particles are small enough that gravity doesn’t cause them to settle. The most commonly used binders are based upon silica [56] sol but there is an increasing interest in alumina-based sols with extensive studies in the literature led by Prof. Pandolfelli and co-workers [43]. The pure silica sols are anionic and are typically sodium- or ammonium stabilised to a pH of 9–11. Through modification using sodium aluminate, however, the sols are stable down to a pH of 3–4. The bonding mechanism of silica sol occurs via a gelling phase.

When combined with other solid particles, the colloidal silica particles can be linked together in branched chains. The branched chains can be generated and the gelling action initiated by pH changes or additions of salts, oxides or solvents. The gelling can also be induced by water removal (e.g. drying). The hydroxyl groups (Si–OH) generate siloxane bonds Si–O–Si resulting in a three dimensional network via the expulsion of water. Removing H from the SiOH groups favours the gelation process. A cold setting mechanism can be induced via the destabilization of the sol through the addition of either CaO or MgO cations. The calcium can be present in the form of a minor addition of CAC. MgO favours the anionic reaction by the formation of Mg(OH)₂ and removal of Hydrogen from the Si–OH groups. This promotes Si–O–Si formation and enhances the gelation rate [44]. Patented compositions use this approach to ensure hardening in the cold state [45]. A main drawback is the green strength and the slow development of strength below 1000 °C.

There has been an increasing interest in such colloidal binders, partly driven by the expiry of some of the early patented compositions containing colloidal silica [45] and partly by the value derived from an ability to dry out colloidal silica castables at a high heating rate. This is derived from two specific properties namely the water removal as a function of temperature and the permeability of castables from using such binders. Literature [15] refers to the fact that colloidal silica bonded castables release water at lower temperatures than calcium aluminate bonded systems as the water is not chemically bonded. Castable binders based upon colloidal alumina have appeared over recent years. Difficulties with early systems
were related to the fact that sodium stabilization was used [46, 47] and that the solids content was not sufficiently high to ensure sufficient strengths. The latest generation products [48] attempt to improve this with the development of sodium-free stabilization sols. Ambient setting properties in the temperature range of 10 – 30 °C relative to other binders are difficult to assess as literature examples [49] are based upon curing at 50 °C.

2.5 Other silicate bonding
Castable binders based upon sodium silicate have been in evidence for many years [18]. They are generally represented by the formula Na₂O · nSiO₂mH₂O where n is commonly between <1 to 3. They are available in powder and liquid forms. The hardening of the binder suitable for castables is via air-drying and heat set but this is often a lengthy process. Gelation can be provoked by the addition of metal or other hardening agents. Sodium silico-fluoride is traditional-ly used as an alkaline metal silicate and causes gelation via the formation of silic acid. Problems associated with its handling and health risk have also led to other hardeners being employed such as aluminium phosphate [50]. Common uses for such bonding systems are in acid resistant applications with siliceous aggregates or for zinc metal applications.

2.6 Combination binders
The combined use of two or more individual binders is an option to overcome specific constraints mentioned above that may exist with a single bond system and to exploit any synergetic effects. This is not new or novel and a number of options exist today with examples of combinations of CAC, hydratable alumina, phosphates as well as colloidal silica demonstrated in already referenced literature [51–54]. The additional examples below demonstrate the potential to create synergetic effects in terms of castables properties.

2.6.1 Calcium aluminate and hydratable alumina
This system has been studied in the literature [50, 51] using a very low total binder content and sufficiently low calcium-containing system that it would be considered as a NCC (No Cement Castable according to ASTM C 401-91 definitions). The CAC role is to ensure a sufficient initial hydration to give strength to cast pieces and sections sufficient for de-moulding. Tests at Kerneos have been run with a hydratable alumina (~75 % Khi Alumina, BET ~190 m²/g) using different addition rates into a bauxite based LCC castable.

Results showed that, at additions up to 3 % in a LCC with 5 % of a 70 % CAC, the early (6–24 h as cast) strengths improved significantly (3×) with increasing hydratable alumina content. This came at the expense of decreasing permeability. Nonetheless, optimization opportunities do appear to exist through the combination of these two hydraulic bond systems.
2.6.2 Calcium aluminate and phosphate systems

Calcium aluminate cement and phosphate compositions have been widely studied in the literature [55–57] for a range of applications from civil engineering, technical concretes, dental applications as well as refractory systems.

Two basic types of phosphates have been investigated; monomer systems (formed from the reduction of phosphate minerals at high temperatures, e.g., orthophosphoric acids) and polymer structured condensed phosphates (for example the series of metaphosphates and polyphosphates, $M_n(P_2O_5)_m$) and $M_{n+2}(P_2O_{5+n})$. The addition levels relative to the (70 % CAC) cement are in the order of 20–40 %. The uses of these monomer type phosphates have proven to be difficult. Violent reactions are often seen which result in an extreme retardation of the hydraulic set associated with the calcium aluminate cement. They also result in poor mechanical strength development.

The reactions with condensed phosphates and calcium aluminate cements are difficult to generalize as widely differing effects are seen depending upon the phosphate used. For example, a strong pH dependence is seen with acid phosphates producing a marked setting time delay and basic phosphates driving an accelerated setting along with specific rheology profiles which need to be controlled with additives.

The reaction mechanism appears to be dissolution of CAC and phosphate to yield an exchange between the calcium ions and the sodium ions. Calcium phosphates are one of the end products as well as the classical CAC hydrate phases. After drying, calcium-aluminophosphates and sodium-alumino- phosphates result as does the cubic $C_3A_6H_6$ phase.

Tests at Kemeos have revealed that at 20 % cement addition rates and 2–6 % phosphate, a range of properties were found. Best results were obtained with pyro-phosphates, hexa-metaphosphates and tri-polyphosphates coupled with synthetic alumina aggregates. In this case, flexural strengths and abrasion resistance were up to three times the values obtained with CAC only based castable at temperatures up to 1100 °C and more specifically in the 800 °C range. Above 1100 °C properties decreased drastically. However, sensitivities to ageing were marked and all systems tested showed deterioration in properties with storage time. The combination of CAC and phosphate has also been explored as a cement clinker composition [58] with an alumino-phosphate cement being produced via a sol-gel process to give a hydraulic composition around CA and $\alpha(3CaO \cdot P_2O_5)$. It was found necessary to control the molar ratio of $SiO_2/Al_2O_3 + P_2O_5$ to ensure satisfactory hydraulic cementitious performance.

2.6.3 Calcium aluminate and colloidal silica

A number of literature references [59] as well as commercial products have explored the reactions with condensed phosphates and calcium aluminate cements. In this case, flexural strengths and abrasion resistance were up to three times the values obtained with CAC only based castable at temperatures up to 1100 °C and more specifically in the 800 °C range. Above 1100 °C properties decreased drastically. However, sensitivities to ageing were marked and all systems tested showed deterioration in properties with storage time. The combination of CAC and phosphate has also been explored as a cement clinker composition [58] with an alumino-phosphate cement being produced via a sol-gel process to give a hydraulic composition around CA and $\alpha(3CaO \cdot P_2O_5)$. It was found necessary to control the molar ratio of $SiO_2/Al_2O_3 + P_2O_5$ to ensure satisfactory hydraulic cementitious performance.
the domain of LCC as a means of trying to enhance mullite formation in the castable through the “homogenous” addition of silica via a colloidial silica. It was found that mullite formation was enhanced when 3% colloidal silica was added to a system that contained 7% of a calcined alumina and 7% of a 70% CAC in the matrix. Colloidal silica has also been found to be an efficient additive to reduce LCC working time and deliver a more rapid development of structure when added in amounts up to 1 mass-% of the total dry mix mass. Working time reduces from 300 min to around 10 min and is accompanied by a more rapid hardening profile as measured by ultrasonics.

The different combination binders discussed above are based on combinations of standard products but it can be easily imagined that bigger synergetic effects could be generated if each of the binders were co-optimised in a simultaneous development.

3 Discussion – the choice of bond system

The choice of bond is defined by a number of considerations. Perhaps the most obvious one is seen in the widespread terminology of “cement free” or “no cement” castables which infers that the removal of cement would be advantageous. Clearly, for certain applications the elimination of CaO coming from the CAC would be advantageous but the reality is more complex than these generic names suggest. The choice can be considered to be on a multi-criteria basis and would certainly need to integrate at least the following points considering refractory product type and installation characteristics along with required installed performance in a given refractory environment. In addition as resources become scarcer more sustainable solutions with lower environmental impacts will become more important and this will undoubtedly impact refractory choice in the future.

3.1 Refractory product type and installation

This discussion has been focussed on monolithic castables which presupposes a certain type of product which would need to offer sufficient working time to allow installation as a function of the installation method and then sufficient hardening and cohesive forces to withstand de-moulding and manipulation until in service. The latter depending on whether a large in situ casting, small or large precast, simple or intricate forms were being considered.

3.2 Installed performance

The usage environment can be classically described via chemical, thermal, and mechanical criteria and then each application positioned accordingly. From this, the target refractory properties are defined. This then reveals where there could be incompatibilities for a given bond system. For example:

• Calcium aluminates do not resist the high levels of chlorine or fluorine found in re-actors producing these chemicals. Nor would silica containing binders be expected to show a high resistance to strongly basic steelmaking slags.

• Incompatibilities generated due to the choice of aggregate system – for example, very small amount of alumina added to zircon aggregates severely reduces the refractoriness.

• Reducing or eliminating CaO would lead to more efficient mullite formation in alu-

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### Table 1 Comparison of relative attributes of refractory castable bond systems

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Constraints</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcium Aluminate Cement</td>
<td>• Presence of CaO which can be detrimental in certain applications and limits thermo-mechanical performance as a function of castable type</td>
</tr>
<tr>
<td>• Formulation flexibility and ability to be used in simple (CC) type products through to sophisticated ULCC castables</td>
<td>• Dry out rates of certain castable types — especially MCC and LCC with simple matrix systems</td>
</tr>
<tr>
<td>• Adaptability to all installation methods and ease of use.</td>
<td>• Ageing in low cement systems</td>
</tr>
<tr>
<td>• Robustness and reliability (as a function of castable type)</td>
<td></td>
</tr>
<tr>
<td>• Flexibility to be used in both A-S as well as essentially pure alumina systems</td>
<td></td>
</tr>
<tr>
<td>Hydratable Alumina</td>
<td>• Setting and hardening characteristics (when used alone) with low green strengths</td>
</tr>
<tr>
<td>• Thermo-mechanical properties above 1400 °C</td>
<td>• Sensitivity to shelf life – intrinsic ageing</td>
</tr>
<tr>
<td>• Creep properties and hot modulus for silica free systems</td>
<td>• Extreme sensitivity to dry out schedules, typically more than double CAC systems</td>
</tr>
<tr>
<td>• Thermal cycling/Thermal shock resistance</td>
<td>• Mechanical properties below 1000 °C</td>
</tr>
<tr>
<td>• Corrosion resistance to selected refractory environments</td>
<td></td>
</tr>
<tr>
<td>Colloidal Silica</td>
<td>• Need different on site logistics to handle liquid binder (low sustainability rating to ship liquids to point of use)</td>
</tr>
<tr>
<td>• High permeability and rapid water removal at low temperature = Rapid dry out schedules</td>
<td>• Setting time control and can be sensitive to low temperatures</td>
</tr>
<tr>
<td>• Precursor to form mullite to give high thermal shock resistance and hot properties</td>
<td>• Low green strengths and slow strength development</td>
</tr>
<tr>
<td>• No dependence upon water quality</td>
<td>• Concentrated supplier base for colloidal silica</td>
</tr>
<tr>
<td>• Hot adhesion</td>
<td>• Composition as a function of liquid binder addition</td>
</tr>
<tr>
<td>• Lubrication effect for pumping/wet shotcreting</td>
<td>• Presence of silica can exclude certain applications</td>
</tr>
<tr>
<td>• Resistance to acids</td>
<td></td>
</tr>
<tr>
<td>• Lower rebound for gunning</td>
<td></td>
</tr>
<tr>
<td>Phosphate</td>
<td>• Some are acid-based systems – toxicity</td>
</tr>
<tr>
<td>• Hot adhesion – hot installation via patch/gunning</td>
<td>• Workability and sensitivity to water dosage</td>
</tr>
<tr>
<td>• Mechanical properties between 250–1000 °C</td>
<td>• Setting time control</td>
</tr>
<tr>
<td>• Thermal cycling</td>
<td>• Ultimate refractoriness</td>
</tr>
<tr>
<td></td>
<td>• Mechanical properties above 1000 °C</td>
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</tbody>
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4 Conclusions

A vast array of different monolithic bond systems are available today with binders that create in situ bonds offering enhanced value either through the processing/place steps and/or the final installed performance. No single bond system delivers optimal performance in all applications under all types of installation conditions. Intrinsic characteristics have to be rather adapted to each situation and application in terms of placing technologies and installed performance. The selection of bonding system is a result of a multi-criteria approach with commonly antagonistic criteria. Each of the bond systems reviewed presented a series of characteristics and specific advantages along with a number of constraints. The degree of use and adoption across a range of castable applications depends to a degree on the number of constraints of each binder. Binders that yield bond systems with significant constraints only find use in niche or specific applications where their performance characteristics outweigh induced constraints. This can explain the widespread and overwhelming use of calcium aluminate bond systems in monolithic castables. These systems provide formulation flexibility and an ease of use that is unrivalled in terms of monolithic castables, not to mention being adapted for use over a wide range of applications.

However, the emerging class of colloidal systems demonstrates there are opportunities to deliver extra value in terms of reducing drying time, providing the end users accept their usage constraints. Thus, the motivation to continue the development of refractory bond systems remains as strong as ever. For further progress to be made, simultaneous development of the castable and the bond system is needed to deliver enhanced value without generating additional constraints during castable processing. These developments will surely fuel the growth of castables and their potential replacement for other types of refractories. In particular “combination” binders offer interesting perspectives relative to changing the performance-constraint relationships of existing bond systems.

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