SELF-HEALING CASTABLE REPAIRS ITS OWN CRACKS (‖)

--Effect of Expansive Materials on Self-healing Behaviors of Mullite Based Castables

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ABSTRACT

Effect of oxidation-induced crack healing agents such as SiC, Si and B_4C on self-healing behaviors of an andalusite based castable has been reported in ICR2019. Expansion and phase transformations crack healing agents are presented this year. Kyanite and andalusite as additives are investigated into pertinent castables to clarify the relations between phase transformation and self-healing performance. The attained results demonstrate that kyanite and andalusite cann't drop self-healing starting temperature, but improve self-healing efficeincy including self-healed strength and strength recovery rate. There is no direct relationship between phase transformation with expansion and self-healing behavor. A special mechanism of crack self-healing is discussed. Liquid phase sintering phenomena and densification (coalescence) are two parameters that affect the self-healing procedure. Dynamic elastic modulus (MOE) are tested and calculated to predict thermal shock resistance while phase transformation processing through ultrasonic pulse velocity method.

INTRODUCTION

Oxidization is saturated in sillimanite group materials. Those materials can not reveal oxidation-induced crack healing ability.
However, expansive materials, such as andalusite, kyanite, quartz and *in-situ* Al_2O_3-MgO spinel formation, could at least party fulfil self-healing function when refractories service in high temperature[1-10].
Due to the thermal cycling of thermomechanical stresses in refractory lining can lead to joint opening in both, bricked and monolithic linings. Such joints may cause a prevailing wear unless they are closed by expansion of the material at the hot face. The expansive materials containing refractories lead to a macroscopic expansion at the hot face, capable of closing such joints.

Sillimanite group minerals includes kyanite, andalusite, and sillimanite – all of which are naturally occurring polymorphs of $Al_2O_3{\cdot}SiO_2$. These minerals are also commonly referred to as the 1:1 "alumino-silicates". Despite their similarities, each of these minerals has a unique crystalline structure. The Sillimanite Minerals' decomposition temperature and volume expansion are as follows.

Table1 Sillimanite group minerals' decomposition and expansion

Minerals	Decomposition Temperature, °C	Approximate volume expansion%
Sillimanite	1530-1625	5,6
Andalusite	1350-1500	4,9
Kyanite	1325-1410	18,8

Andalusite has intrinsic properties, once sintered (mullite precursor, composite microstructure, and thermal shock resistance) that yields castable and brick that exhibit very high thermo-mechanical stability.

Kyanite, which occurs in nature is an elongated, "lath-like" crystals, has many important (and several unique) properties that make it a valuable raw material in refractory and ceramic products. It also decomposes into a high hot strength phase mullite at a relatively low temperature (1325°C), thereby inducing faster sintering. But most importantly, it undergoes a large and irreversible expansion at this temperature – and it is this expansion that is used by many refractory and ceramic manufacturers to reduce shrinkage during initial firing.
In addition, the "lath-like" habit of the kyanite crystal lends cold "green" strength and abrasion resistance to products that are made from it.

The propose of this investigation is to clarify a mechanism of crack self-healing through exploiting expansion of materials and/or phase transformations.
Kyanite and andalusite dopants in castable refractories were chosen to discuss self-healing properties with high temperature in laboratory.

CASTABLES PREPARATION AND TESTING METHOD

1. Castables Preparation

Mullite based low cement self-flow castables were selected to be tested. The castables' compositions are shown Table 2.
In Table 2, recipe S is used as a reference, S7 is a kyanite dopant castable and S8 is an andalusite dopant castable.

Table 2 Tested Mullite Based Castables Composition

	S	S7	S8
Mullite M60	75	69	65
Kyanite	0	6	0
Andalusite	0	0	10
Calcium Aluminate Cement	6	6	6
Fume Silica	5	5	5
Alumina Fines	14	14	14
Total	100	100	100

For each specimen, a 3 kg batch mixture using the formulation in Table 1 was prepared, by dry mixing for 2 minutes at slow speed, using a mixer with 5 liter capacity mixing bowl. Then wet mixing was prepared by addition of 6,5% water after 5 minutes mixing at slow speed. Test specimens were prepared by self-flow casting the mixture into the stainless steel molds, having the dimensions of 130×50×50 mm. The castables were cured at room temperature (20°C) for 24 hours. After 24 hours curing time, the specimens were allowed to be dried at the temperature of 110°C for 24 hours, and then fired at 1200°C, 1300°C, 1400°C, 1500°C and 1600°C respectively for 5 hours. The cold crashing strength (CCS) was measured according to the ASTM C133. Permanent linear changes (PLC) of the specimens fired at different temperature were measured. Bulk density and apparent porosity were all determined by standard methods.

2. Tests and Methods of Evaluating the Self-Healing Efficiency of Castables

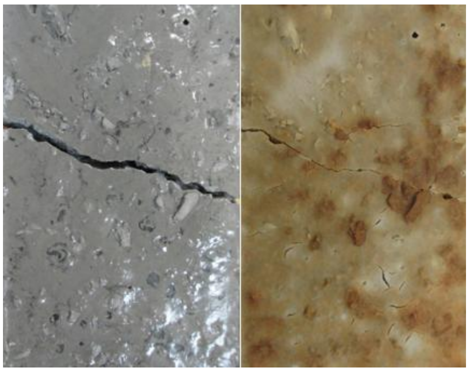

Figure 1 Damaged specimen S8 at 110°C and healed after firing at 1600°C

Specimens were damaged by a hammer at room temperature and fired at several temperatures for 5 hours. Figure 1 shows a specimen at room temperature and after fired at elevated temperature. It is shown crack was healed after firing, even though inconspicuous crack still appeared on the surface.

3. Strength Recovery Behavior

The lower bound of the available temperature region was experimentally determined from the strength recovery behavior. The strength recovery rate was evaluated from the strength of the specimen healed at several temperatures for 5 hours, abbreviated as crack-healed specimen in this paper.
The self-healing temperature was determined to be the lowest temperature at which the strength of crack-healed specimen was recovered up to the strength of non-cracked specimen.

Cold crushing strength (CCS) test was conducted. There is a unapparent healed crack before crushing. By way of comparison, the strengths of non-cracked and healed specimens were similarly measured.

The strength recovery rate % is expressed by the ratio of healed strength to non-cracked original strength.

4. MOE Testing

The modulus of elasticity (**MOE**) analyses were developed by means of the ultrasonic pulse velocity (UPV) of the castable samples using direct transmission measurements performed by the contact method. Ultrasonic pulse velocity (UPV) measurements were carried out by using a commercial portable ultrasonic non-destructive testing instrument. The apparatus is a digital tester with the following main parts; an electrical pulse generator, an amplifier, and a timing circuit, which was connected to a pair of transducers to measure the time interval elapsing between the onset of a pulse and its arrival at the receiving transducer.
To achieve the UPV measurements the following steps were taken.

- Transmitting and receiving transducers were positioned at opposite ends of each specimen (Figure 2). Then, this instrument generates low-frequency ultrasonic pulses.
- Measurements of the travelling time of the signal through the specimen of pulses between both transducers were done.
- The dynamic elastic modulus (MOE) of samples, E_d were determined using Equation (1).

The relationship between elastic constants and the velocity of an ultrasonic compressional wave pulse in an isotropic elastic medium of infinite dimension is given by:

$$E_d = pV^2 \frac{(1+\mu)(1-2\mu)}{(1-\mu)} \quad (1)$$

where E_d = the dynamic elastic modulus (GPa)
μ = the dynamic Poisson's ratio, for refractory 0.2-0.3
p = the density (g/mm^3)
V = the compressional pulse velocity (m/sec)
V (m/s) = Path length (mm)/Transit time (μSec)

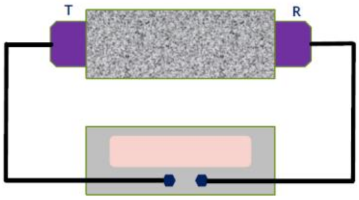

Figure 2 Transducer arrangement: Transmitting (T) and receiving (R) transducers positioned at opposite ends of each standard specimen.

TESTING RESULTS AND CONSIDERATION

1. Self-healing Starting Temperature

Refractory Castables are usually operarted at high temperature. It is important to know the starting temperature for self-healing.

The three specimens have been fired for 5 hours at different temperatures. Cracks self-healing has happened when there was an enough temperature. The self-healing starting temperatures are acquired as Table 3.

110°C dry-out specimens	S	S-7	S-8
Self-healing starting temperature	1500°C	1550°C	1550°C

Table 3. Self-healing starting temperature

It is evident that the starting temperatures of S7 and S8 are beyond expansion range of kyanite and andalusite which described in Table 1. This implies that the expansions of kyanite and andalusite do not lead to self-healing. Further discussion will be conducted later on.

2. Kyanite and Andalusite's Thermal Expansion and Phase Transformations.

Kyanite is an anhydrous aluminosilicate, with crystalline structure triclinic, it belongs to the Al_2O_3-SiO_2 system, and its chemical formula is $Al_2O_3{\cdot}SiO_2$; its decomposing at high temperatures produces mullite ($3Al_2O_3{\cdot}2SiO_2$) and free silica (SiO_2). The mullitization process accompanies 16–18 vol % expansion, making it be widely used as expanding agent in the refractory field.

Decomposition occurs according to the following reaction:

$$\text{Kyanite} \rightarrow \text{Mullite} + \text{Crystoballite} \quad (2)$$
$$3(Al_2O_3{\cdot}SiO_2) \rightarrow 3Al_2O_3{\cdot}2SiO_2 + SiO_2$$

Thermal Expansion & Conversion of Kyanite (Virginia) [5] shown in Figure 3.

The mullitization of kyanite begins at about 1250°C, and is completed virtually instantaneously at 1450°C which is much faster & at a much lower temperature than either Andalusite or Sillimanite.

At the high temperature (1350°C), kyanite particles are decomposed to mullite still without glassy phase and the metastable alumina presents in the mix reacts with amorphous silica to produce additional mullite, this is called secondary mullitization[5-6].

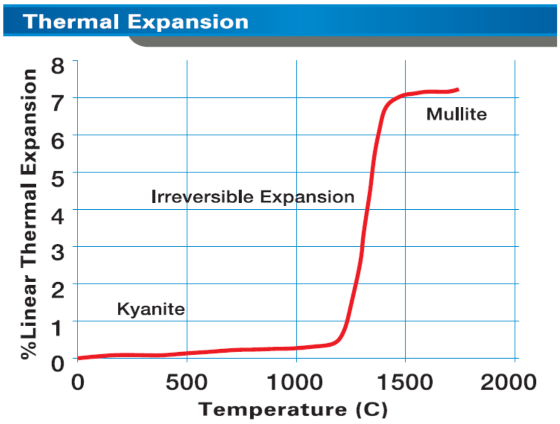

Figure 3 Kyanite demonstrates unique expansion characteristics when heated to (and through) its decomposition temperature

The silica formed during this thermal transformation partially react with impurities mainly alkalis and iron oxide to form a liquid phase and finally a vitreous silicate phase which impairs the quality of product by deteriorating the high temperature properties like hot MOR, thermal shock resistance of the products.

Andalusite, a natural aluminosilicate mineral provides high thermal shock resistance to refractory products. At high temperature (> 1200 °C), andalusite single crystal grains are transformed into a composite made of a 3:2 mullite single crystal with a capillary network filled with silica rich liquid. Because of the specific microstructure and the presence of the liquid phase, during thermal shocks, the mullitized andalusite crystals exhibit a behaviour typical of composite materials: microcracks are deflected by mullite/glass interfaces and stopped in glass zones. During a further heating, cracks generated by a thermal shock are healed. The composite microstructure of mullitized andalusite crystals seems to be a strong advantage in order to increase thermal shock resistance. It can explain the good behaviour of fired andalusite bricks regarding thermal cycling[3-4].

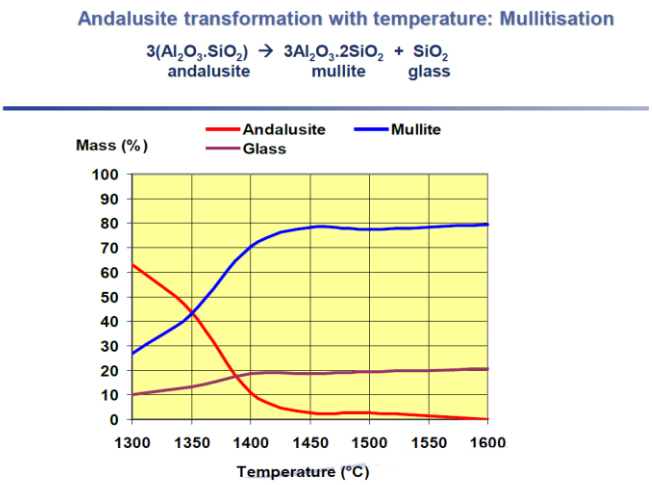

Fig. 4 Phase weight content, according to sintering temperature

The thermal-transformation of andalusite gives mullite (87 %) and silica glass (13 %) at 1000–1500°C[3-4]. The following reaction shows this thermal transformation process:

$$\text{Andalusite} \rightarrow \text{Mullite} + \text{Glass} \quad (3)$$
$$3(Al_2O_3 \cdot SiO_2) \rightarrow 3Al_2O_3 \cdot 2SiO_2 + SiO_2$$

This mullitisation is accompanied with a volume expansion of 5–7 %. Figure 4 illustrates the evolution of this transformation with temperature.

This silica glass can coalescence each other and can react with alumina to form secondary mullite (1400–1550°C).
These two phenomena of silica glass create two ceramic matrices.

The decomposition of kyanite can't directly produce glass in Equation (2). However, the decomposition of andalusite could produce glass in Equation (3). This difference will effect their high temperature properties.

3. PLC, Bulk Density and Apparent Porosity Analyses as well as Liquid Phase Sintering

In this investigation, kyanite dopant specimen S7 demonstrates an expansion range between 1200 and 1500°C. There is a much higher PLC in Figure 5. Moreover, it illustrates a dropping MOE (Figure 20) that will lead to a better thermal shock resistance. However, it doesn't show improved CCS in Figure 17.

Similarly but differently, andalusite dopant specimen S8 shows an expansion range between 1350 and 1500°C in Figure 5. After that shrinkage is accelerating and PLC is becoming much lower after 1550°C. Sintering continues till 1600°C and a much higher CCS is gotten in Figure 17.

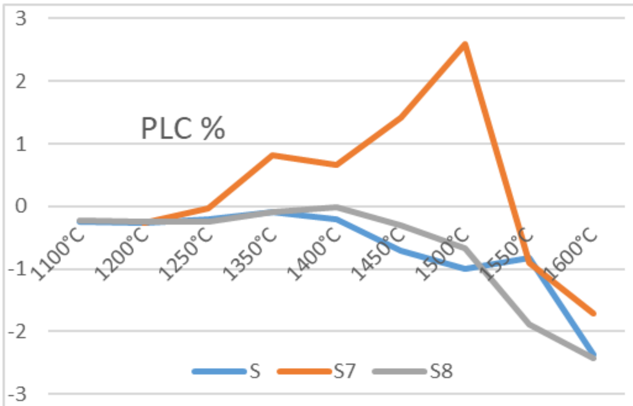

Figure 5 Permanent linear changes (PLC) with elevated temperature

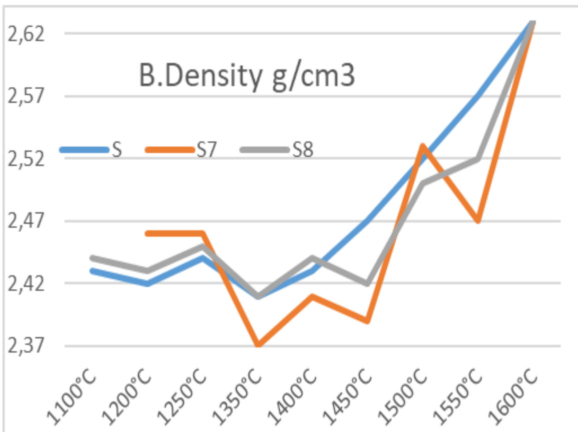

Figure 6 Bulk Density with elevated temperature

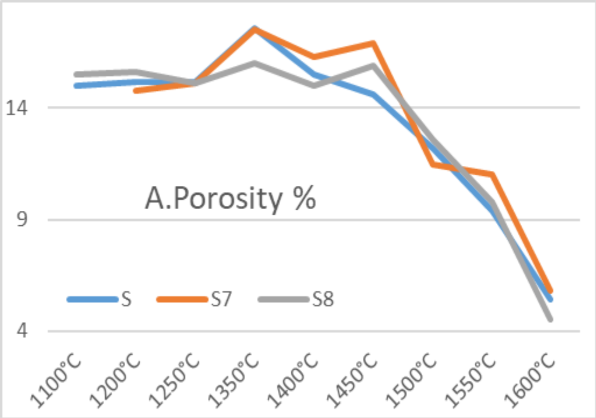

Figure 7 Apparent porosity with elevated temperature

- Low Cement Castables (LCC) Sintering

Mullite formation in refractory castables has been found to depend on several factors. Mullite grows from a liquid. The liquid softens the castable at some intermediate temperature(1200-1300°C) [2]. The softening temperature depends on impurities present in the matrix.

In terms of reference specimen S in this investigation, the lowerest density, highest porosity and highest PLC are found at 1350°C which shown on Fig 5, 6 and 7.

It is supposed that mullization reaches a highest rate at 1350°C for specimen S. Thus initial stage of sintering begins and mullitization process continues to 1500-1550°C (Fig.5), it subsequently proceeds to final stage of sintering untill 1600°C.

- Liquid Phase Sintering

A schematic of the microstructure changes during liquid phase sintering in Figure 8, starting with mixed powders and pores between the particles. During heating the particles sinter, but when a melt forms and spreads the solid grains rearrange. Subsequent densification is accompanied by coarsening. For many products there is pore annihilation as diffusion in the liquid accelerates grain shape changes that facilitates pore removal.

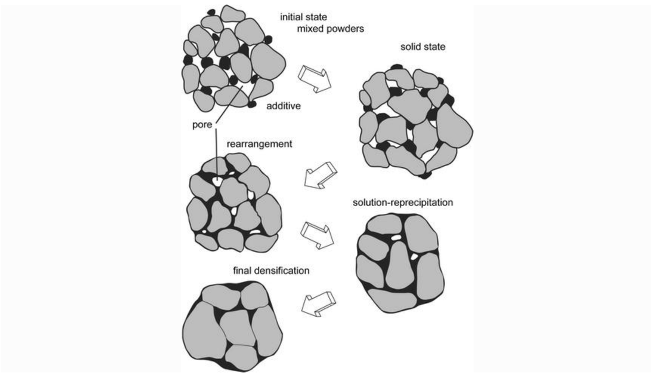

Figure 8 A schematic of the microstructure changes during liquid phase sintering

Kyanite's (S7 in Fig. 5, 6 and 7) expansion and phases transformation occur between 1200 and 1500°C. After completely mullization, specimen S7 proceeds to sinter untill 1600°C.

Moreover, betwee 1350 and 1500°C, anadlusite's (S8 in Fig. 5, 6 and 7) expansion and phases transformation happen. Specimen S8 proceeds to sinter untill 1600°C after mullization is completed.

Compared with Specimen S, not only does kyanite dopant specimen S7 defer the densification procedure with its decomposition between 1200 and 1500°C; but also S8 andalusite defers that between 1350 and 1500°C. Those deferments lead to a stage of porosity enlargement (Fig. 7) that hinder the densification procedure which shown in Figure 6.

Again, compared with Specimen S, both S7 and S8 defer the final sintering temperature from 1500°C to 1550°C which shown in Figure 5. It leads to a last stage with density increasing (Figure 6) and porosity dropping (Figure 7) that enforce the densification.

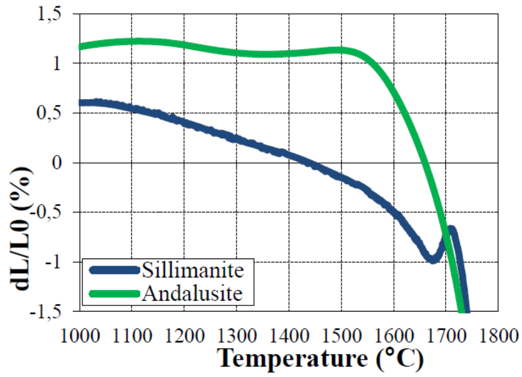

Figure 9 Thermal expansion of Andalusite & Sillimanite fines.

Furthermore, Figure 9 details a thermal expansion comparison of andalusite and sillimanite products[3]. It is found that andalusite starts shrinking and sintering around 1550°C. This temperature corresponds with the self-healing starting temperature of S8.

4. Microstructure Analyses of Crack Self-healing Behavors

Microstructure Analyses were done through optical microscopic observations of the three 1600°C fired specimens.

Small amounts of liquid phase are present during sintering, it is liquid phase sintering for low cement castables.

Liquid glassy phases are filled into the cracks when sintering. It is evident in Figure 10, and confirmed by Figures 11-14. Continuing sintering leads to densification and coalescence. Bridgings between crack walls and coalescence phenomena are shown in Figures 11, 12 and 14.

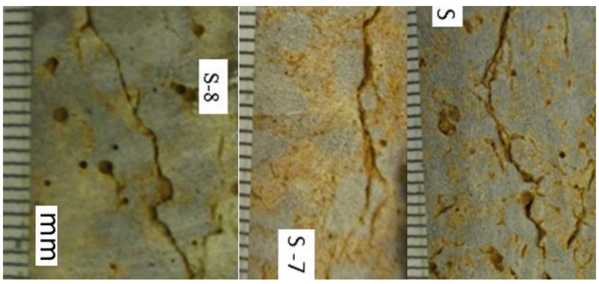

Figure 10 Self-healed cracks filled with glassy phase after 1600°Cx5hrs

On the observation of cross-sections through naked eye, there are more irregular closed pores shown on reference specimen S. It also indicates small size closed pores on specimen S7 and larger size of closed pores on specimen S8 (Figure 10). Those should correspond to expansions of kyanite and andalusite. Thus kyanite and andalusite dopants make castable matrixs more homogenizing.

In Figure 11, liquid glassy phase is filled between both sides of crack. There is a bridge linking between the walls. The bridge is attributable to densify coalescence.

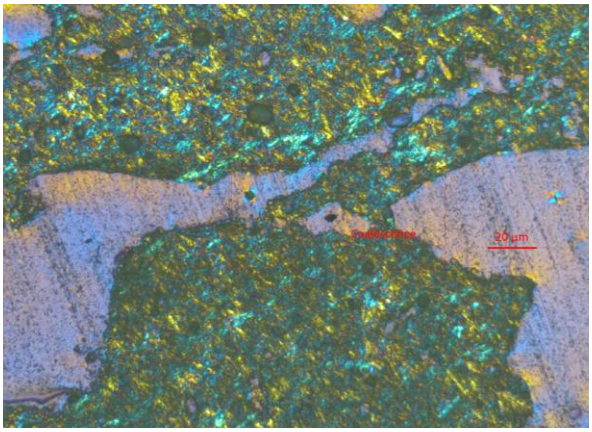

Figure 11 Reference Specimen S 1600°Cx5hrs, densifying coalescence and bridging between the crack walls

In Figure 12, the crack is going along with boundary between a large grain and matrix. Liquid glassy phase is filled between both sides of the crack. There is a bridge linking the walls. The bridge is attributable to densify coalescence.

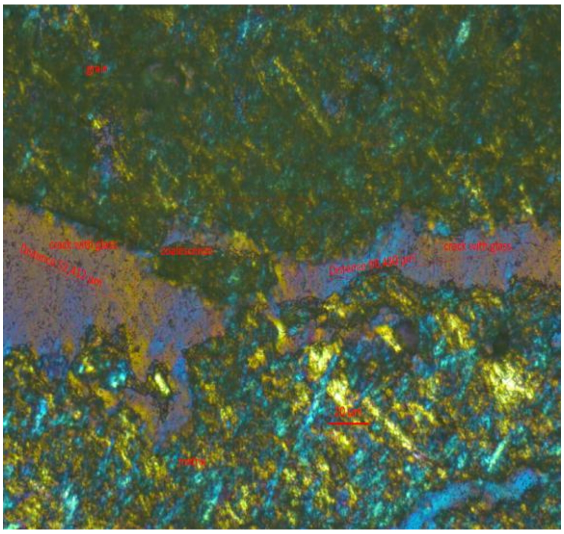

Figure 12 Specimen S7 1600°Cx5hrs, densifying coalescence and bridging between the crack walls

In Figure 13, a glassy phase filled crack is approaching to be closure. Bridging and coalescencing are going to be happened.

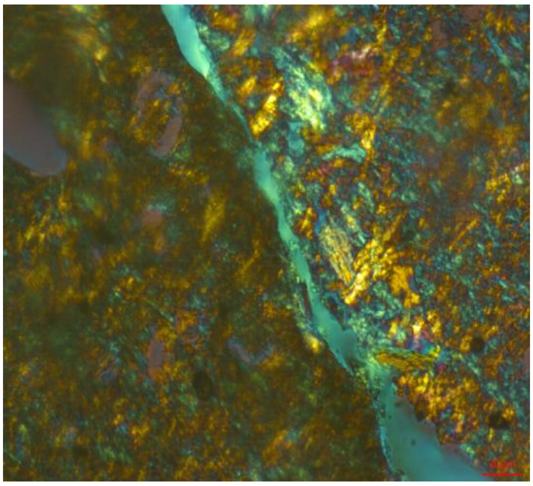

Figure 13 Specimen S8 1600°Cx5hrs, crack filled with glassy phase, densification is leading to crack closure.

In Figure 14, a glassy phase filled crack is approaching to be closure. More bridging and coalescencing are ongoing.

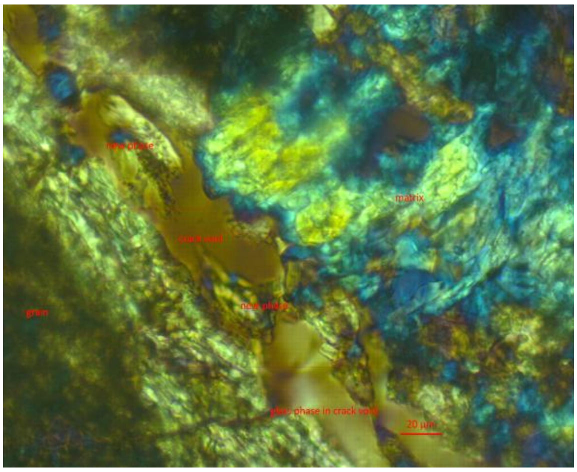

Figure 14 Specimen S-8 1600°Cx5hrs, crack filled with glassy phase, densifying coalescence and bridging between the crack walls

It is perspicacious to find that much more bridgings and coalescences between crack walls in kyanite dopant specimen S7 than other specimens S and andalusite dopant S8. It reflects the greatest self-healing efficiency upon kyanite dopant in Figure 18 and Figure 19 at 1600°C firing condition.

5. Mechanism of Crack Self-healing through exploiting expansion of materials and/or phase transformations

The self-healing starting temperatures are beyond the extent of expansion of kyanite and andalusite which indicated in Table 2.

It is demonstrated that there is no direct relationship between phase transformation (with expansion) and self-healing behavor.

One of categories for ceramic's self-healing is attributed to the re-sintering of cracks by secondary sintering[10]. Therefore, similar mechanisms should be dominant on the crack-healing in refractory castables.

The local healing of the cracks in the refractory structure can be assigned to liquid phase sintering. Presented liquid glassy phases fill into crack void; moreover the compact shrinkage occurred from each side of the crack wall toward each other. When there are compact shrinkage and densification, growing grains come in contact with each other, the local sintering phenomenon happens at high temperature which can finally result in crack closure. At high enough temperature, compact shrinkage and densification allow all the grains growing from one side of the crack wall and come in contact with the grains on the opposite side. Then the sintering phenomena can close the whole crack and complete healing can be achieved.

Thereby liquid phase sintering phenomena and compact shrinkage or densification (coalescence) are two parameters that affect the self-healing procedure of LCC castable.

Low Cement Castable(LLC) S

Regarding regular low cement castable S, a schematic diagram of the crack healing mechanism is proposed in Figure 15.

The self-healing process is described as follows:

Heating→ liquid phase sintering → liquid glassy phase filled into the crack void → compact shrinkage or densification (coalescence)→ crack-healed

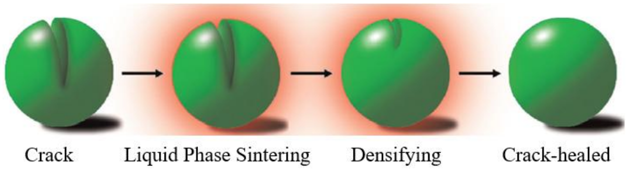

Figure 15 Schematic of Crack-healing in LCC Castables

The following three conditions must be met for a crack-healed area to recover completely.

- The volume between the crack walls must be completely filled glassy phases formed by liquid phase sintering; then the bridging of cracks due to densifying coalescence.
- The strength of the crack-healing substance must be equivalent to or higher than that of the matrix.
- The crack healing substance must be strongly bonded to a matrix.

Kyanite/andalusite dopant LLC S7 and S8

With kyanite and/or andalusite dopants, the low cement castables S7 and S8, a schematic diagram of the crack healing mechanism is demonstrated on Figure 16.

Expansion of kyanite or andalusite can only close cracks and touch crack walls, but do not bond and link together two sides of crack walls. Furthermore, addition of kyanite or andalusite extends liquid phase sintering and densification procedure to a higher temperture. After completion of expansion, sintering is continuing and densification commencing, thus the cracks walls are bonding. Finally, virtually self-healing is realized.

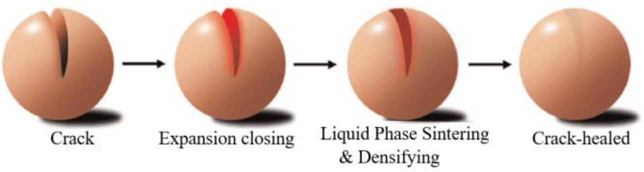

Figure 16 Schematic of Crack-healing in kyanite-andalusit dopant LCC Castables

It is elucidated in Figure 16 that there is an extra expansion closing cracks stage before liquid phase sintering and densification. Therefore, liquid phase sintering and compact shrinkage or densification phenomena are still two crucial parameters that affect the self-healing procedure.

The self-healing process is described as follows:

Heating → crack walls touch and close as of expansion → liquid phase sintering → liquid glassy phase filled into the crack void → compact shrinkage or densification (coalescence)→ crack-healed

There is a glass phase and a crystalline phase. If the crack-healing substance was crystalline, then the crack-healed specimen would exhibit high bending strength even at an elevated temperature. However, if crack-healing substance was glassy phase, then the crack-healed specimen would exhibit low bending strength at an elevated temperature. A key point of crack-healing technology is how many crystalline phases are deposited in a crack-healing substance.

Based on above schematics Figure 15 & 16, it is easily understood that materials' expansion and phase transformations don't reveal a direct crack healing ability.

6. Strength, Self-healed Strength and Strength Recovery

Both kyanite and andalusite are decomposed to mullite in high temperature. The mullitization of andalusite has glassy phase, and kyanite without glassy phase accordingly.

The silica formed during kyanite thermal transformation partially react with impurities mainly alkalis and iron oxide to form a liquid phase and finally a vitreous silicate phase which impairs the quality of speciemn S7 by deteriorating the strength which shown in Figure 17.

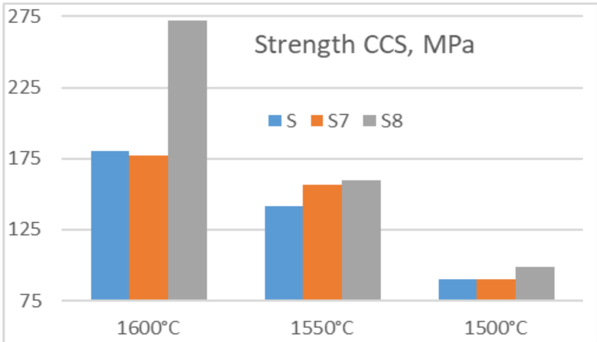

Figure17 Cold crushing strength(CCS) with elevated temperature

It is found that specimen S8 gains a highest strength (Figure 17) in high temperatures of 1550°C and 1600°C.

A possible explanation could be that andalusite help castables develop stronger bondings by creating a complementary bondings by a secondary mullite formation between the glass phase on the surface of mullite and calcined alumina; and the high viscosity of glass phase linked to a low amount of impurities present.

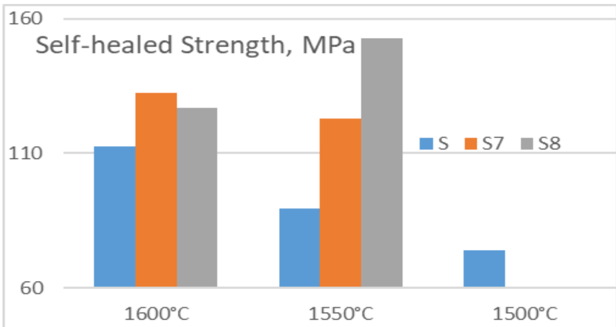

Figure 18 Self-healed strength of specimens with temperature

Self-healed Strength:

It is evident that kyanite and andalusite specimens S7 & S8 have better self-healed strength in Figure 18. It must be related to the quality and quantity of glassy phase and the amount of mullite while liquid phase sintering happening.

The amounts of glassy phase and mullite are increasing in specimen S7 and S8 duo to mullization of kyanite and andalusite. Sintering is the key parameter that affect the self-healing procedure. Sintering in practice is the control of both densification and grain growth.

Higher amounts of glassy phase and mullite result in higher self-healed strength in Figure 18.

Self-healing efficiency can also be descrived as strength recovery rate % which is the ratio of self-healed CCS to non-crack CCS.

Compared with andalusite, kyanite dopant S7 has a better self-healing efficiency at 1600°C. Andalusite dopant S8 has lower efficiency at 1600°C duo to it's highest unimpaired no-crack CCS in Figure 17.

Both andalusite and kyanite have much higher self-healing efficiency at 1550°C.

Self-healing efficiencies are illustrated in Figure 18 &19. Those imply that expansion/phase transformation dopants such as kyanite and andalusite are functioning effectively.

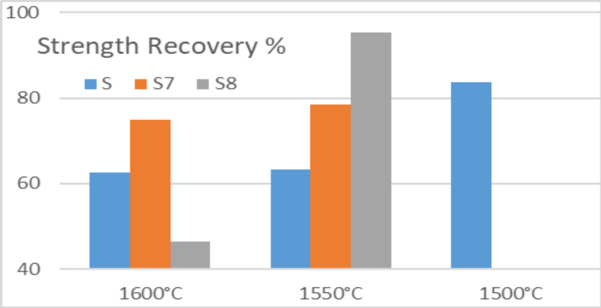

Figure 19 Self-healing Strength Recovery of Mullite based Castables when specimens were pre-dried at 110°C

Without kyanite and andalusite, the castable can self-healing at earlier temperature at 1500°C. However, expansive materials andalusite and kyanite prevail at higher temperature and more efficient for self-healing (Figure 18 &19).

7. MOE Results Analysis

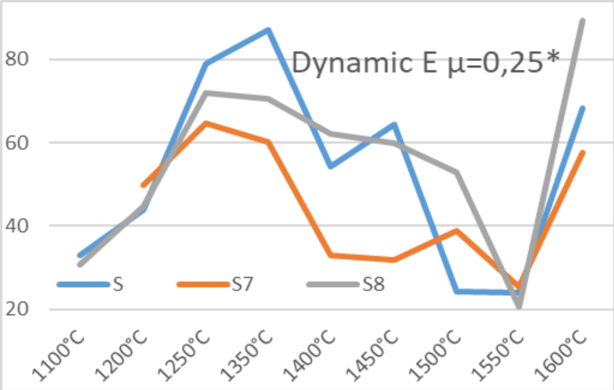

Figure 20 Modulus of Elasticity with treated temperature

Reference specimen S: when temperature is less than 1350°C, sintering and densification are controlled by CaO-Al_2O_3-SiO_2 system. Thus MOE is going up. After 1350°C, mullization process begins and it is an expansion process, thereby MOE is going down untill 1500°C. Mullization is completed before 1500°C, then sintering re-starts after 1550°C. On Figure 5 and Figure 20, there is a flat stage between 1500 and 1550°C which corresponding with rearrangement of liquid phase sintering shown in Figure 8.

Kyanite and andalusite dopant Specimens S7 and S8: when temperature is less than 1250°C, sintering and densification are controlled by CaO-Al_2O_3-SiO_2 system, MOEs are going up. After 1250°C, kyanite and/or andalusite begin to decomposite and mullizate. It is an expansion process respectively, therefor MOEs are going down untill 1550°C. After 1550°C, decomposition and mullization are completed, further sintering and densification are ongoing, thereby MOEs are going up quickly.

MOE curve of S7 is always lower than S8, it notices that kyanite dopant is more effective than andalusite as of thermal shock resistance imporvement.

CONCLUSIONS

The cracks self-healing behaviors of mullite based low cement castables were investigated up to 1600 °C for 5 hours. Dopants such as kyanite and andalusite couldn't change the self-healing mechanism. Nevertheless, they can elevate the self-healing starting temperature from 1500°C to 1550°C due to kyanite and andalusite defer sintering, meanwhile, final sintering temperature is rising.

The expansions of kyanite and andalusite could close and seal the cracks, but could not lead to cracks bonding or self-healing. Moreover, liquid phase sintering phenomena and compact shrinkage or densification (coalescence) are two parameters that affect the self-healing procedure of LCC castable.

In high temperatures of 1550°C and 1600°C, kyanite and andalusite help castables develop stronger self-healing bondings by mullitization and secondary mullite formation. Furthermore, self-healing efficiencies including strength recovery rates and self-healed strengths are ameliorated.

In a word, although kyanite and andalusite can't reduce self-healing starting temperature, it is important to note that self-healing efficeincy is improved.

Modulus of Elasticities (MOE) with treated temperatures indicates that kyanite dopant is more effective than andalusite for thermal shock resistance improvement.

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