



# Development of Water-Soluble Composite Salt Sand Cores Made by a Hot-Pressed Sintering Process

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## Abstract

A wide variety of water-soluble cores are widely used in hollow composite castings with internal cavities, curved channels, and undercuts. Among them, the cores made by adding binders of inorganic salts in the form of aqueous solutions have excellent solubility in water. However, excellent collapsibility is often accompanied by poor moisture absorption resistance. In this study, a water-soluble core with moderate strength and moisture absorption resistance was prepared by hot pressing and sintering the core sand mixture of sand, bentonite, and composite salts, and a tee tube specimen was cast. The experimental results showed that the cores with KCl-K<sub>2</sub>CO<sub>3</sub> as binder could obtain strength of more than 0.9 MPa and still maintain 0.3 MPa at 80±5% relative humidity for 6 hours; the subsequent sintering process can significantly improve the resistance to moisture absorption of the hot pressed cores (0.6 MPa after 24 hours of storage at 85±5% relative humidity); the water-soluble core prepared by the post-treatment can be used to cast tee pipe castings with a smooth inner surface and no porosity defects, and it is easy to remove the core.

**Keywords:** Water-soluble cores, Hollow composite castings, Salt cores, Collapsibility

## 1. Introduction

The water-soluble core is a component made of water-soluble salt as the primary material [1-4] or binder [5-7] for forming the inner surface of castings. It is widely used in the high - pressure die casting processes for manufacturing castings with complex cavities or curved channels due to its excellent water-soluble collapsibility and environmental friendliness, especially in the process of lightweight and integration of automobile, sanitary ware and other products [8-11].

Generally, water-soluble cores for die-casting require high strength, so this type of core is mainly made by the casting method. However, because of bearing a much smaller impact of molten metal liquid, cores with relatively low strength are suitable for gravity casting or low-pressure casting, and a wide range of materials and processes can be employed to form water-soluble cores such as pressure sintering and binder bonding process. Jaroslav [12] prepared a core with a mixture of slightly wet KCl and a small amount of addition under a pressure of 100 KN, which was used for the gravity process of magnesium alloy. Liu [13] used a MgSO<sub>4</sub> binder to prepare water-soluble cores by secondary microwave heating, which can be used to produce complex hollow



castings. Zhang [14] designed a water-soluble core with tensile strength up to 2.5 MPa by hot pressing using  $K_2CO_3$  aqueous solution as a binder, which also can meet the needs of gravity casting of non-ferrous metals. Although the water-soluble cores mentioned above have excellent water-soluble collapsibility, they cannot be used directly due to their moisture absorption after being placed in a humid environment, which restricts their wide application. Considering the hygroscopic problem, a precipitation method [15] was proposed for the  $K_2CO_3$  binder sand core. In this method, the core was dipped in an ethanol solution of  $CaCl_2$  and then heated. The hygroscopic resistance of the core was effectively improved because of the transformation of easy moisture absorption  $K_2CO_3$  into relatively anti-moisture absorption KCl. The direct use of the KCl as a binder seems feasible, but due to the relatively low solubility, the introduction of more KCl necessarily increases the water content, which leads to a low strength or shaping difficulty.

In this paper, a water-soluble sand core was prepared with KCl- $K_2CO_3$  composite salt by heating at a low temperature and then sintering at a relatively high temperature, which takes advantage of the high strength of  $K_2CO_3$  and the good humidity resistance of KCl. The tensile strength, water absorption rate, gas evolution and water-soluble rate of the sand core were investigated. In addition, the scanning electron microscope (SEM) was employed to observe the micro-morphology of the water-soluble cores.

## 2. Experimental part

### 2.1. Material and equipment

The raw sand was Dalin silica sand with 70/100 meshes. The binder consisted of analytically pure  $K_2CO_3$  and KCl (Sinopharm Chemical Reagent Co., Ltd.). The bentonite was 400 mesh from Shanghai Xincheng Fine Chemical Co., Ltd. The sand mixer was the SHY resin sand mixer. The sand samples were the standard “8” dog-bone sand specimen samples. The moisture-absorbability weight was tested by a type JA5003N electronic balance with an accuracy of 0.001 g. Other test equipment included the SWY universal hydraulic strength testing machine, and scanning electron microscopy (SEM, FEI Co., Ltd., Netherlands).

### 2.2 Preparation of sand samples and properties test

Fig. 1 shows the flowchart of the manufacturing process. Firstly, raw sand, bentonite and KCl with a certain proportion were mixed uniformly by stirring for 5 min. Secondly, the  $K_2CO_3$  solution was poured into the sand mixer and stirred again for 5 min to obtain the composite salt core sand. Next, the core sand was filled and pressed in the homemade mold with a heating device and then demolding after some time to prepare the standard “8” dog-bone sand specimen samples. Finally, the prepared samples were heated in an oven at a certain temperature for some time, and then the heated samples were placed in a resistance box and sintered at a higher temperature.

The strength and storage strength of the sand samples were tested. In this paper, the strength is the tensile strength of the water-soluble composite sand core cooled to room temperature after being heated in an oven or sintered in a resistance box. The storage strength is the tensile strength of the water-soluble core after being put into a stable humidity condition for several hours. The moisture absorption rate of the composite salt sand core is calculated by the equation of  $w = (M_t - M_o) / M_o \times 100\%$ , where  $w$  represents the moisture absorption rate of the sand sample,  $M_t$  is the mass of the sand sample exposed in the constant moisture surroundings for  $t$  hours,  $M_o$  is the initial mass of the sand sample. The water solubility of the core is calculated by testing the tensile strength of the sample immersed in water. All the testing results were the average value of five measurements.

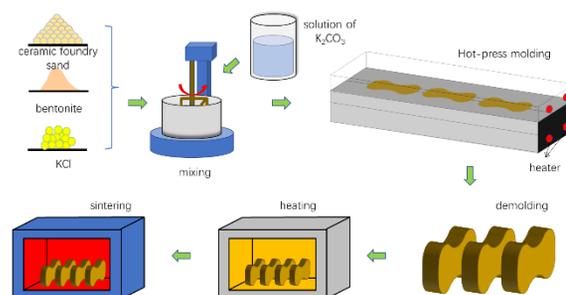


Fig. 1. The flowchart of the manufacturing process

## 3. Results and discussion

### 3.1. Initial tensile strength

#### 3.1.1. Effect of heating time and proportion of composite salts on water-soluble core

Core material consisted of 500 g raw sand, 40 g composite salts, 1 wt.% bentonite and 60 mL water were pre-molded in a die and heated at 140 °C for 30, 60 and 90 min. The effect of heating time and proportion of composite salts on the tensile strength of the water-soluble core are shown in Fig. 2. As shown in Fig. 2, the heating time has no significant effect on the tensile strength, especially when the proportion of composite salts is lower than 50 wt.%. However, the proportion of composite salts obviously affects the tensile strength, and the tensile strength shows a sharp increase when the proportion is between 30 and 50 wt.%. It is known that inorganic salts crystallize and precipitate from their aqueous solutions by nucleation and growth. When the water-soluble cores bonded with KCl- $K_2CO_3$  composite salt were heated at a certain temperature, the water in the compound salt solution evaporated fast, and the solution transformed into a saturated, even a supersaturated solution. Meanwhile, the  $K_2CO_3$  and KCl nucleated and grew up quickly, forming the bonding bridges between the sand grains and acting as a binder. When the value of  $K_2CO_3 / (K_2CO_3 + KCl)$  was small, i.e. the content of  $K_2CO_3$  was low but that of KCl was high, the solution concentration remained at a low level because of the low solubility of KCl in water. As a result, as the salt solution migrated into the voids between the salt particles or sand grains and then evaporated, not only the thickness

of the inorganic salt film crystallized on the surface of sand grains but also the number of sand grains to binder contact points increased with the increasing of  $K_2CO_3$ . It contributes to the low tensile strength of samples composed of KCl–(0–30%)  $K_2CO_3$  and the relatively high strength of specimens composed of KCl–(50–100%)  $K_2CO_3$ . In the following study, the heating time was chosen as 1 h and the proportion of composite salts was selected between 50% and 100%.

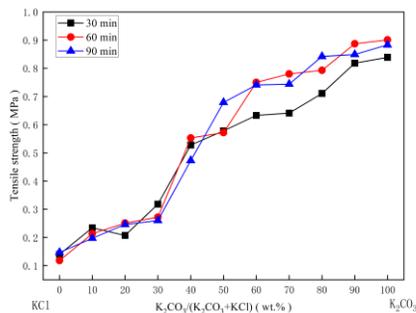


Fig. 2. Effect of heating time and proportion of composite salts on the tensile strength of water-soluble cores

### 3.1.2. Moisture absorption and storage strength

The moisture absorption was the amount of water absorbed by a water-soluble sand core within a certain time under a constant humidity condition. In this paper, the specimens were exposed to a relative humidity of  $80\pm 5\%$ . As shown in Fig. 3, the moisture absorption rate of water-soluble cores increases with the extension of the standing time and the proportion of  $K_2CO_3$ . As is known, the  $H_2O$  molecule is a polar molecule which has a weak interaction with ions and can be easily absorbed on the salt surface. The deliquescence of salt takes place when the ambient relative humidity (ARH) is equal to or exceeds the critical relative humidity (CRH) [16]. The CRH of  $K_2CO_3$  is about 40% [15], which is much lower than the ARH. Therefore, as the standing time prolongs, more and more deliquescence of  $K_2CO_3$  weights the cores. Due to the lower CRH of  $K_2CO_3$  than that of KCl (about 84%), the moisture absorption rate of the cores improves with the increase of  $K_2CO_3$  proportion.

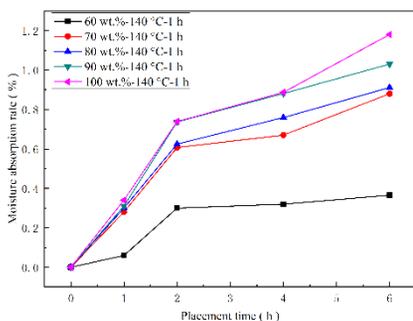


Fig. 3. Effect of placement time and  $K_2CO_3$  proportion on water absorption of water-soluble cores

The storage strength was the tensile strength of water-soluble cores heated at a certain temperature and then put in an ARH of  $80\pm 5\%$  for some time. Fig. 4 illustrates the storage strength of

water-soluble cores with different proportions of  $K_2CO_3$  at 0 and 6 h. As shown in Fig. 4, there is a steep decrease in storage strength when the water-soluble cores are stored for 6 h because the deliquescence of salts bonded between the sand grains weakens the bonding bridge. The storage strength also improves with the increase of  $K_2CO_3$  proportion because of more bonding bridges and thicker films of salts offered by the high concentration of  $K_2CO_3$ . However, a high proportion of  $K_2CO_3$  leads to bad surface quality. Therefore, the proportion of  $K_2CO_3$  between 60 and 80 wt.% is focused on in the following study.

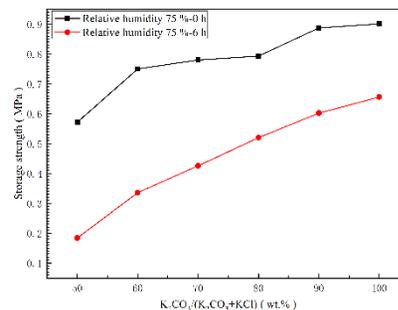


Fig. 4. Effect of placement time and  $K_2CO_3$  proportion on storage strength of water-soluble cores

### 3.1.3. Effect of heating temperature on tensile strength

The heating temperature affects the evaporation rate of water in the salt solution, which has an important influence on the tensile strength. Fig. 5 illustrates the trend of tensile strength changing along with the heating temperature. As shown in Fig. 5, there is a peak strength in the range of 140 °C to 180 °C for each concentration of  $K_2CO_3$  solution, which is controlled by the crystallization of inorganic salts and evaporation of water. Due to the demolding core heated in an oven, the evaporation of water in salt solution takes place on the outside first and extends to the inside gradually. Thus, the core gradually builds the bonding bridges from outside to inside. A higher temperature results in faster water evaporation, leading to smaller salt crystals and a higher bonding strength. However, the preformed bonding bridges crack under the impact of large amounts of water evaporation, which weakens the strength of the cores. These two opposite effects lead to the peak strength at 160 °C. Cores with a high proportion of  $K_2CO_3$  also demonstrate high tensile strength at different temperatures, as discussed in section 3.1.1. Therefore, the heating temperature of 160 °C was selected for the subsequent experiments.

### 3.1.4. Effect of sand ratio on tensile strength

The core material consisted of raw sand, 40 g composite salts, 1wt% bentonite and 60 mL water, and the samples were heated at 160 °C for 1 h. Fig. 6 shows the tensile strength varying with the content of composite salts. As shown in Fig. 6, the tensile strength of cores with all different proportions of  $K_2CO_3$  firstly increases with the increasing proportion of composite salts to sand and decreases after the strength reaches the peak strength. On the one hand, the higher proportion of composite salts to sand leads to much more bonding bridges and higher strength. On the other hand, the higher proportion of composite salts in core sand is also accompanied by a higher proportion of water which weakens the strength of the cores under the impact of water evaporation. As a

result, the peak strength appears at 10 wt.%. The effect of  $K_2CO_3$  content on tensile strength is similar to the previous study and is stated in section 3.1.1. In the following study, the 10wt.% proportion of composite salts to sand is selected.

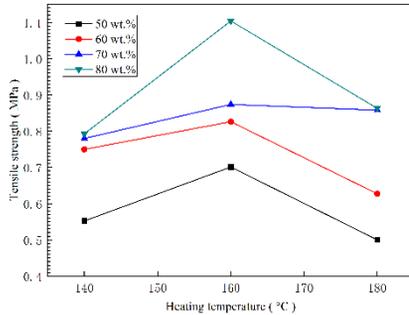


Fig. 5. Effect of heating temperature on the tensile strength of water-soluble cores

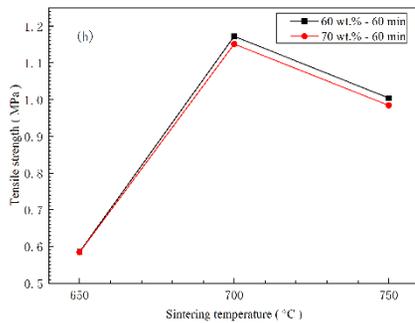


Fig. 6. Effect of composite salt content on the tensile strength of water-soluble cores

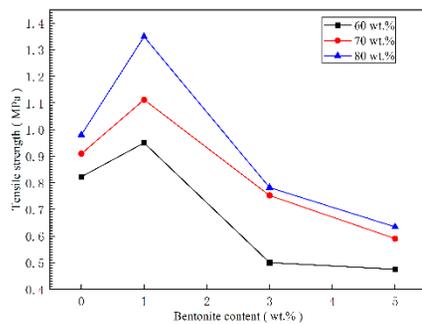


Fig. 7. Effect of bentonite content on the tensile strength of water-soluble cores

### 3.1.5. Effect of bentonite content on tensile strength

Fig. 7 shows the effect of bentonite content on tensile strength. As shown in Fig. 7, a rise in strength is observed when bentonite is added to the core sand, followed by a decrease with greater bentonite content. Bentonite provides a large number of crystalline nucleation cores for the crystallization of inorganic salt during the evaporation of the salt solution, which refines the inorganic salt grains, eliminates the weak interface between the crystals and improves the bonding strength between the sand grains. Thus, water-soluble cores exhibit low tensile strength when bentonite is

not present. However, as too much bentonite is added, the strength of the bonding bridge weakens as a result of the increased specific surface area and the decreased bonding bridge amount. Therefore, the bentonite addition amount of 1wt.% is selected in the following study.

## 3.2. Final tensile strength

The sand core prepared after heating molding was placed in a resistance box. The cores were sintered at 650, 700, 750 and 800 °C for 30, 60 and 90 min, respectively. Fig. 8 shows the effects of sintering temperature and sintering time on the final tensile strength of the water-soluble cores. As shown in Fig. 8, the final tensile strength firstly increases and then decreases with the increase of sintering time. It can also be found that the maximum tensile strength at 60 and 90 min of sintering is slightly higher than that at 30 min.

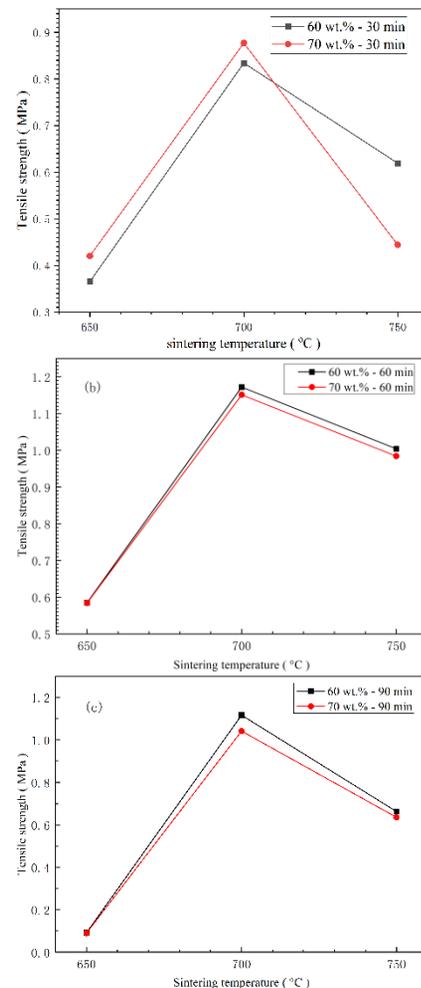


Fig. 8. Effect of sintering temperature and sintering time on tensile strength sintered for (a) 30 min, (b) 60 min, and (c) 90 min

SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> are the main composition of the sand grains and bentonite. Thus, the chemical reactions involved in the core sand mixture with K<sub>2</sub>CO<sub>3</sub> are mainly as follows:

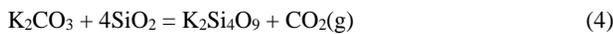
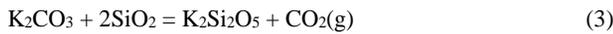
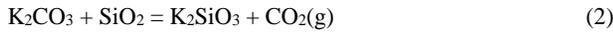
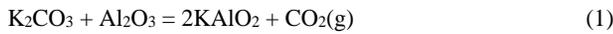


Fig. 9 shows the Gibbs free energies variation of reactions between K<sub>2</sub>CO<sub>3</sub> and oxides at 0–1000 °C. From the thermodynamic point of view, K<sub>2</sub>CO<sub>3</sub> tends to react with oxides (Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>) and produces K<sub>2</sub>O\*mSiO<sub>2</sub> and KAlSi<sub>m</sub>O<sub>n</sub>. However, the amount of bentonite is low, K<sub>2</sub>O\*mSiO<sub>2</sub> must be the main reaction product and performs a cohesive action. As the sintering temperature rises and the sintering time prolongs, K<sub>2</sub>O\*mSiO<sub>2</sub> phase with high bonding ability increases, which leads to improved tensile strength. However, the silica sand undergoes a phase change at 575 °C, which may cause significant stress and weakens its tensile strength when the cores are cooled from an excessive temperature.

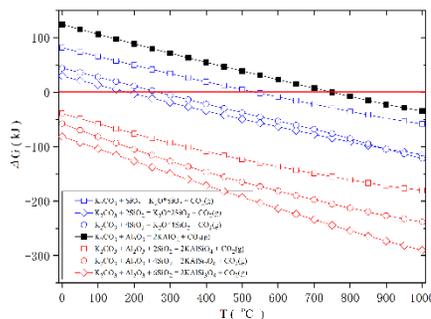


Fig. 9. Gibbs free energies variation of reactions

### 3.3. Moisture absorption and storage strength

Fig. 10 shows the moisture absorption rate of water-soluble cores sintered at 700 and 750 °C for 1 h varying with the placement time. As shown in Fig. 10, the moisture absorption rate improves with the extension of placement time, which results in the deliquescence of inorganic salt. The moisture absorption rate of sintered cores at 750 °C is much lower (0.15% for 6 h) compared to that without sintering (0.37% for 6 h, seen in Fig. 3), which indicates that the hygroscopic inorganic salt such as K<sub>2</sub>CO<sub>3</sub> and KCl transforms into other components with low hygroscopic ability. The moisture absorption rate of water-soluble cores sintered at 700 °C is higher than that at 750 °C, which means the reaction of former cores is inadequate and remains more K<sub>2</sub>CO<sub>3</sub>.

Fig.11 shows the storage strength of water-soluble cores sintered at 700 and 750 °C for 1 h varying with the placement time. As shown in Fig.11, the storage strength decreases with the extension of placement time. It is evident that these cores remain at a higher strength even placed in the same RH for 24 h (0.34 MPa and 0.58 MPa) compared to that without sintering just for 6 h (0.33 MPa, seen in Fig.4).

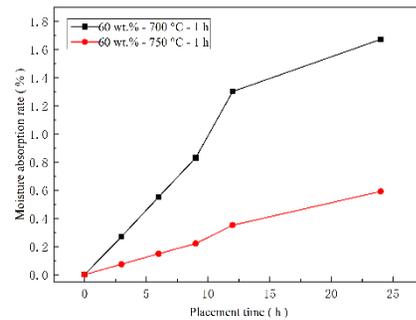


Fig. 10. Effect of placement time and sintering temperature on the moisture absorption rate of water-soluble cores

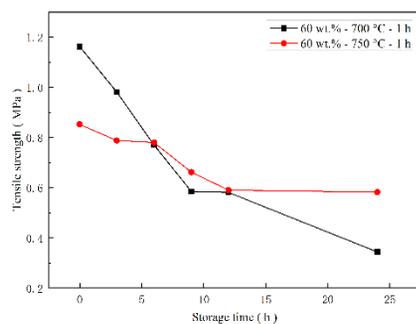


Fig. 11. Effect of storage time and sintering temperature on the storage strength of water-soluble cores

### 3.4. Gas evolution of water-soluble cores

The gas evolution of water-soluble cores is listed in Tab. 1. As shown in Tab. 1, the cores sintered at 750 °C have lower gas evolution than magnesium sulfate or resin - bonded sand cores, which indicates that composite salt sand core possesses low gas evolution and is suitable for the aluminum alloy with complex structures.

Table 1.

Performance	KCl-K <sub>2</sub> CO <sub>3</sub> bonded sand core		MgSO <sub>4</sub> bonded sand core [13]
	700 °C	750 °C	
Gas evolution (ml/g)	9.1	6.6	7.7

### 3.5. Tensile strength after soaking in water

Fig. 12 shows the tensile strength of water-soluble cores sintered at different temperatures after soaking in water for varying periods. As shown in Fig. 12, the tensile strength of water-soluble cores sintered at 700 and 750 °C decreases with the extension of soaking time. It is obvious that there is a stepdown of tensile strength at the first 30 s for both different sintering processes, and then the retained strength declines gradually until it almost loses its retained strength at 80 min. However, when the soaking time prolongs to 60 s, the retained strength of water-soluble cores is lower than 0.2 MPa, which grants an easy shake out of the cores from castings. Cores sintered at 750 °C, which shows a better capability of moisture absorption resistance, possess a slight ability of hydrolytic resistance compared to that sintered at 700 °C. It also indirectly proves that the reaction product during the sintering process possesses a better capability of hydrolytic resistance.

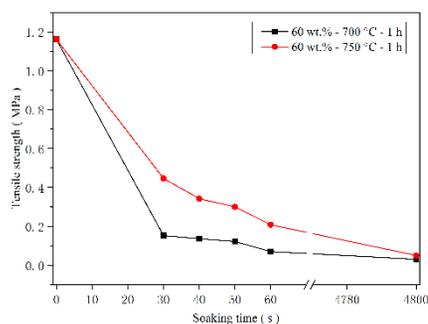


Fig. 12. Effect of soaking time on tensile strength

### 3.6. Morphology

The microscopic morphology of the KCl–K<sub>2</sub>CO<sub>3</sub> bonded sand core (before and after moisture absorption) is shown in Fig.13. As shown in Fig. 13(a), there are a lot of holes between the irregular sand grains, which provide channels for water during the washing out of the core. This explains the rapid loss of retained strength when it is placed in water. Fig. 13(b) shows a single sand grain coated with smooth inorganic salt film and a number of bonded bridge fractures. Elements of Si, O, K and trace amounts of Cl are detected in the smooth salt film at point b by EDX analysis, as shown in Fig. 14(b), while Al and a trace amount of Fe and Mg are detected at the area of fracture at point a except for Si, O and K as shown in Fig. 14 (a). Therefore, it can be concluded that the aggregation of bentonite in the liquid bridge between sand grains provides heterogeneous nucleation cores for inorganic salt crystallization during the evaporation process of salt solution. This is also confirmed by the EDX analysis located in the bonding bridge and the surface of sand grains, as shown in Fig. 14 (c) and (d). Fig. 13 (c) and (d) show the fracture morphology of water-soluble cores sintered at 700 and 750 °C for 1 h, respectively. They both have smooth and compact sand grain surfaces and good arc transition bonded bridges without any visible cracks. However, when they are placed in the RH of 85-90 % for 24 h, several cracks (as shown in Fig. 13 (e) and (f)) appear on the cross-section of the

core after tensile strength testing, which further confirms the ability of wet conditions to weaken bonding strength.

### 3.7. Hollow composite aluminium alloy casting test

The water-soluble cores with the formulation mentioned above were prepared by heating at 160 °C for 60 min in a resistance box and then sintering at 750 °C for 60 min. Commercial ZL101A aluminum alloy was used in this study and the pouring temperature was 700 °C. Fig. 15 (a) shows the simple hollow alloy casting fabricated with a water-soluble core. The casting result shows that the water-soluble cores still maintain the original shape during the casting process (as shown in Fig. 15b), which indicates that the composite salt - bonded sand core can sustain the impact of alloy melt liquid. When the simple hollow alloy casting with the core was put into water, the strength of the sand core intensity decreased rapidly. Then shake the casting in water, all of the core sand can leach out easily. The whole surface of the casting is smooth, and there is no obvious metal penetration, chemical scab and other defects, as shown in Fig. 15 (c). Therefore, KCl and K<sub>2</sub>CO<sub>3</sub> used as the binder for bonding core sand have good water-soluble collapsibility and can be used to form the interior shape of castings with good leachability.

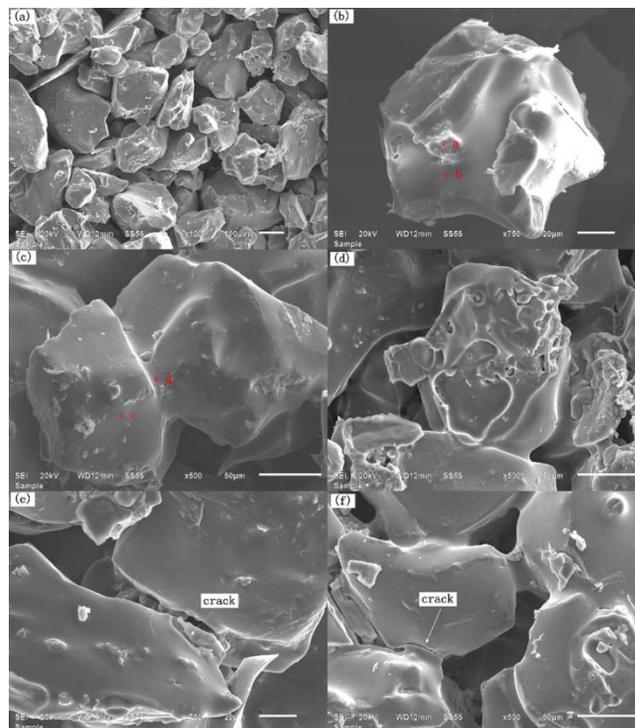


Fig. 13. Fracture morphology of the water-soluble cores. (a) water-soluble core without sintering; (b) a single sand particle coated with K<sub>2</sub>CO<sub>3</sub> and KCl; (c) water-soluble core sintered at 700 °C for 1 h; (d) water-soluble core sintered at 750 °C for 1 h; (e) water-soluble core of (c) placed in the RH of 85-90 % for 24 h; (f) water-soluble core of (e) placed in the RH of 85-90 % for 24 h.

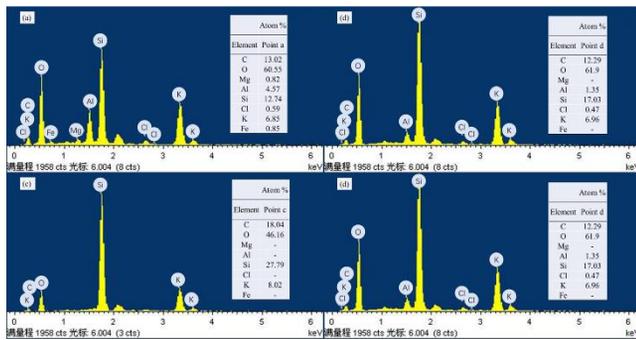


Fig. 14. EDX analysis of water-soluble core at the point of (a) and (b) in fig.14(b), (c) and (d) in fig.14(c)

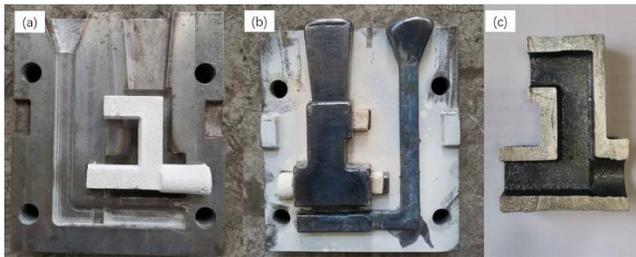


Fig. 15. The photo of aluminum alloy casting fabricated by water-soluble core and leached by water. (a) Casting molds with water-soluble cores fixed; (b) Aluminum alloy casting with the water-soluble core; (c) Dissected casting

## 4. Conclusions

In this study, the water-soluble KCl–K<sub>2</sub>CO<sub>3</sub> bonded sand core was developed and optimized. The microstructures of the cores were analyzed with the SEM-EDX. The result can be summarized as follows.

- 1) The un-sintered strength of the water-soluble composite salt core exceeded 0.9 MPa and reduced to 0.3 MPa after 6 hours of exposure at 80 ± 5% ambient relative humidity, which can meet the requirements for gravity casting of general aluminum alloy and also limit its application. The processing conditions were: 10 wt.% composite salts in which 60 wt.% K<sub>2</sub>CO<sub>3</sub>, 1 wt.% bentonite, curing at 140 °C for 1 h.
- 2) The process of sintering at 750 °C for 1 h can effectively improve the anti-hygroscopic of the un-sintered core and also guarantee the core a low gas evolution (6.6 mL/g) and collapse in 80 min.
- 3) Potassium chloride and the reaction product of K<sub>2</sub>O·mSiO<sub>2</sub> and KAISi<sub>m</sub>O<sub>n</sub> have better resistance to moisture absorption than potassium carbonate so that the sintered core maintains a high tensile strength at a relative humidity of 85±5% for 24 h. Potassium chloride is a readily soluble salt, thus ensuring that the sintered cores have good leachability after casting.

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