Thermal conductivity of Al-Salt composites

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1. Introduction

The present work was initiated with a view to examine the possibilities of determining the amount of entrapped aluminium metal in the salt – slag mixture, which is a mixture of chlorides of alkali metals mixed with Al_2O_3 , AlN and other compounds[1-2], a rest product after the secondary melting of aluminium. Basically, the way to judge the value of aluminum dross depends upon its amount of metallic aluminum and chlorides. “Skimming”, white dross, black dross and salt cake are the terms used depending on the amount of metallic Al [3]. The entrapped metal, which can be as high as 5 % of the salt cake is valuable from an economic view point. The present work has a wider objective of total recycling of the entire rest product from secondary aluminium smelting, which, apart from the economic advantage has a considerable positive effect on the environment.

A number of methods have been developed to ensure the accuracy of analytical measurements based on the relationship between porosity and density with the amount of metallic aluminum. [4–10]. However, such procedures require a higher level of sophistication and may not be suitable in practical situations [11].

In this paper, an attempt is made by measuring the thermal diffusivity of composites of the salts with metallic aluminium as a function of Al metal in the composite at room temperature in order to examine the applicability of this method to salt cake from aluminium remelting. The work was primarily directed to synthetic mixtures of the chlorides with aluminium powder, mixed in various proportions with a view to see if the results could be used as calibration values. One set of measurements were carried out as well with salt cake from the industry. Attempts were also made to interpret the results on

the basis of theoretical models for thermal diffusivity; viz. thermal resistance model (developed by Hsu et al.[12]) and physical model (initially developed by Maxwell [13]). It was expected that these models may enable a better understanding of the contribution of the interfacial effect between different components and the arrangement of different components in the salt cake to the thermal properties.

A successful application of this model will result in a simple elegant method that can be used by the aluminium industry in estimating the amount of metallic aluminium in the rest product.

2. Experimental

a) preparation of sample

Synthetic composites

In the present work, the composites were prepared considering the major ingredients of the salt cake. The precursor materials for the Al/NaCl/KCl compacts were commercially pure Al powder (45-630 mesh, ≥ 99.8 wt-%, Alfa Aesar), commercial NaCl powder (≥ 99.5 wt-%, Sigma-Aldrich) and commercial KCl powder (≥ 99 wt-%, Sigma-Aldrich), respectively. The NaCl and KCl powders were pre-dried in a muffle furnace at 473K for at least 120 min. The mass ratio of NaCl and KCl powders was fixed as 2:1 corresponding to the industrial salt cake composition and then mixed thoroughly with the Al powder at a specified ratio. Seven powder mixture samples, with different mass fractions of Al, viz. 0, 0.02, 0.04, 0.06 0.08, 0.10 0.12 were pressed into pellets using a hydraulic press at 5MPa. In Table 1, samples with No. 0, 2, 4, 6, 8, 10, 12, correspond to salt-aluminium composites with metallic aluminum contents: 0 wt-%, 2 wt-%, 4wt-%, 6 wt-%, 8 wt-%, 10 wt-% and 12 wt-%, respectively.

Table 1: The sample No. and the corresponding compositions

Sample No.	0	2	4	6	8	10	12
Al wt-%	0	2	4	6	8	10	12
KCl wt-%	33.33	32.67	32	31.33	30.67	30	29.33
NaCl wt-%	66.67	65.33	64	62.67	61.33	60	58.67

The compacts were placed in an electrical tube furnace pre-heated to 673K under purified argon gas. The resultant compacts had a diameter of 10mm and a thickness of 3

mm.

Salt cake sample

The industrial salt cake sample was subjected to chemical analysis. The assay is presented in Table 2.

Table 2: The chemical analysis of raw salt cake

Composition	Al ₂ O ₃	Al	AlN	MgO	Fe ₂ O ₃	KCl	NaCl	SiO ₂	CaO
Mass %	39.71-41.71	3-5	7	4.21	2.67	10.69	22.28	6.64	1.8

As shown in the table, the salt cake contained nearly 32 mass % of alkali chlorides, over 50 mass % non-metallic oxides, *i.e.* alumina, silicon oxide as major components. Stewart [14] suggested that the following compounds may be present in aluminum drosses: Al₂O₃, AlN, Al₄C₃, MgF₂, NaAlCl₄, KAlCl₄, MgAl₂O₄ besides that, there are also fluorides, carbides and organic materials, like ink, paint, etc. These are generally in extremely small amounts.

The pieces of metallic aluminium which have a dull grey were picked out from a certain amount of salt cake ranged in size from a maximum of about 2 cm down to about 1–2 mm. Some particles having metallic luster and some glassy coating were also picked out. Both of those were crushed and grounded into finer particles. The fines were separated using a 200 mesh sieve. The magnetic components in the large particle size fraction were then separated by U-magnet, the rest is collected. From the mass of salt cake and metallic aluminum, the content of metallic aluminium can be roughly estimated as:

$$Alwt\% = \frac{M_{\text{metallic-particle}}}{M_{\text{saltcake}}} \quad (1)$$

Where Al wt% is the content of metallic Al in the salt cake, $M_{\text{metallic-particle}}$ is the mass of metallic aluminum, M_{saltcake} is the mass of the salt cake.

The salt cake dross was crushed and ground into powders and sieved through 200 mesh sieve. The compacts of salt cake were made by following the same procedure as above.

b) Thermal diffusivity measurements

The thermal diffusivity of the samples was carried out at Company of 703, Aerospace Science and Technology Corporation, China. During the measuring, the top surface of the pellet sample is irradiated with laser beam, which provides an instantaneous energy pulse. The laser energy is absorbed on the top surface of a sample

and gets converted into the heat energy. The heat energy travels through the sample. Immediately after the laser pulse, the temperature of the rear surface of the sample is monitored by collecting the radiation using a photovoltaic infrared detector. The thermal diffusivity, α , is calculated from Eq. 2[15]:

$$\alpha = \frac{0.1388L^2}{t_{1/2}} \quad (2)$$

Where L is the thickness of the sample and $t_{1/2}$ is the time required for the temperature of the rear surface to reach half of its maximum temperature.

The morphology of the samples was studied by optical microscopy (Olympus Microscope PMG3). The porosity of compacts was determined by Micromeritics' AutoPore IV 9500, Tsinghua University, China, applying various levels of pressure to a sample immersed in mercury.

In both cases, the experiments were repeated in the case of selected samples.

3. Results

The results of the thermal diffusivities of the synthetic composites and the single sample of the dross are presented in Table 3. The corresponding porosity values are also presented in the same table.

Table 3: The thermal diffusivity and porosity results of different samples

Sample No.	0	2	4	6	8	10	12	Salt cake
Thermal Diffusivity/mm ² s ⁻¹	3.03	3.08	3.07	3.45	3.61	3.59	3.56	0.87
Porosity%	4.82	4.72	3.80	2.56		4.49	2.06	12.17

It is seen that the salt cake compact has a very high porosity and the thermal diffusivity value is correspondingly low. In the case of the synthetic compacts, the thermal diffusivity shows an increase with increasing content of metallic aluminium except in the cases of samples 4, 10 and 12, for which the diffusivity is nearly constant within the experimental scatter. There's no doubt that with the increase of metallic Al in the matrix, the porosity shows a tendency of decrease, even with facing the fact of the lack of No.8 sample result and large deviation of sample10. These values are presented in a graphical form in Fig. 1.

Fig.1: Results of thermal diffusivity and porosity measurements in the case of salt-aluminium composites with different contents of metallic Al in the matrix.

It is seen in Fig. 1 that, samples with zero and 2 wt-% Al do not show any variation in porosity. Beyond this, the porosity shows a tendency of decrease with the increase of metallic Al in the matrix, with the exception of sample No.10, which shows a large deviation from the general trend. It is also seen in this figure that the thermal diffusivity initially shows a constant value up to 4 wt % Al in the composite, beyond which there is a sharp increase with the increase of metallic aluminum content upto 8 wt % in the composites. In the case of the samples with 10 and 12 wt % Al in the composite, the values show a slight decreasing trend.

Typical micrographs taken from cross sections of synthetic salt-aluminum composites are shown in Figure 2. It can be seen that the aluminium particles are uniformly dispersed through the material. No appreciable pores or micro cracks in the composites could be detected. At higher aluminum contents, a tendency of metallic particles to agglomerate is evident.

Fig. 2: the surface optical microscope images of (a)No.6, (b) No.8, (c) No.10 and (d) No.12 samples

Table 4 shows the typical values of thermal conductivity, density, specific heat and thermal diffusivity values of pure Al, NaCl, KCl, Al_2O_3 and air, which are the constituents of the salt-Al composites.

Table 4: thermal properties of pure Al, NaCl, KCl, Al_2O_3 and air at room temperature [16]

	Al	NaCl	KCl	Al_2O_3	air
Density g/m^3	2.7×10^6	2.17×10^6	1.98×10^6	3.8×10^6	883
Thermal diffusivity m^2s^{-1}	105×10^{-6}	3.46×10^{-6}	4.9×10^{-6}	4.28×10^{-6}	38.5×10^{-6}
Thermal conductivity W/m K	256	6.5	6.7	13	0.034
Specific heat J/g K	0.9	0.86	0.69	0.8	1

The thermal conductivities of the specimens investigated in the present work could be calculated from experimental thermal diffusivities for the values of the specific heat and density according to the formula 3:

$$k = \alpha \times Cp \times \rho \quad (3)$$

Where k is the thermal conductivity, α is the thermal diffusivity, Cp is the heat capacity, and ρ is the density. The specific heat of the compact was calculated using the formula for the mechanical mixture:

$$Cp \times \rho = \phi_{KCl} Cp_{KCl} \rho_{KCl} + \phi_{NaCl} Cp_{NaCl} \rho_{NaCl} + \phi_{Al} Cp_{Al} \rho_{Al} + \phi_{air} Cp_{air} \rho_{air} \quad (4)$$

$$\phi_{KCl} = \frac{\frac{KCl - wt\%}{\rho_{KCl}}}{\frac{KCl - wt\%}{\rho_{KCl}} + \frac{NaCl - wt\%}{\rho_{NaCl}} + \frac{Al - wt\%}{\rho_{Al}}} \times (1 - \phi_{air}) \quad (5)$$

Where, ϕ is the volume ratio of each component, similar, in case of volume ratios of NaCl and Al were calculated as eqn. 5.

4. Discussions

It is important to derive theoretical relationships between aluminium contents in the salt-Al composites and the thermal conductivities so that these would serve as calibration curves for industrial samples taken out from secondary aluminium re-melting at a later stage. In order to extrapolate the curve, it is necessary to incorporate the experimental results in a suitable theoretical model. (In later calculation, there needs to be pointed out that the porosity of samples 8 and 10 are assumed as 2.3% and 2.15%, respectively)

4.1 Simple parallel and series approaches

The parallel arrangement (heat flux is parallel to the layers) represents a maximum in thermal conductivity. The serial arrangement (heat flux is perpendicular to the layers) represents the minimum conductivity.

In case of serial arrangement:

$$\frac{1}{k_{eff}} = \frac{\phi_{KCl}}{k_{KCl}} + \frac{\phi_{NaCl}}{k_{NaCl}} + \frac{\phi_{Al}}{k_{Al}} + \frac{\phi_{air}}{k_{air}} \quad (6)$$

In case of parallel arrangement:

$$k_{eff} = \phi_{KCl} k_{KCl} + \phi_{NaCl} k_{NaCl} + \phi_{Al} k_{Al} + \phi_{air} k_{air} \quad (7)$$

Fig.3: the comparison between experimental data and the prediction of parallel, serial models

Fig.3 allows a simple estimation of the effective thermal conductivity range that is to be expected. However, one can see that this range is rather large, thus, as we shall discuss later when considering more details about the sample structures.

4.2 Hsu et al. approach

Fig.4 :(a) a three dimensional cube and (b)its unit cell spatially periodic three-phase system, (c) equivalent electrical resistance.

Hsu et al. model combines the thermal resistance approach with the unit cell approach. By working with a structural unit cell, one can link the structure of the porous medium to the visual representation of the model. This cell is then split up into layers of solid and gas phases, so that the unit cell becomes a combination of serial and parallel thermal resistors.

Due to the similar properties between NaCl and KCl, these are treated as one single phase, *viz.* salts in the present work. In case of salts +air system:

$$k_{eff} = k_{salts} \phi_{salts} + k_{air} \phi_{air} = 4.82 \quad (8)$$

$$k_{salts} = 5.11W / mk \quad (9)$$

In the case of Al(salts)+air: protuberance has the blue cube, the white cube in the centre represented the metallic Al phase and chlorides phase, respectively. The rest, painted in red color, are the space for air being trapped in the pore.

$$\phi_{salts} = l_c^3 \quad (10)$$

$$\phi_{Al} = 6 \times l_A^2 \times \frac{(1-l_c)}{2} \quad (11)$$

Where, l_c and l_A are the length of cube cell for chlorides and metallic Al, respectively.

$$R_1 = \frac{\frac{1-l_c}{2}}{k_{Al}(\frac{l_{Al}}{2})^2} \quad R_2 = \frac{\frac{1-l_c}{2}}{k_{air}(\frac{1-l_{Al}^2}{4})} \quad (12)$$

$$R_3 = \frac{2(l_c - l_{Al})}{k_{salts}l_c^2} \quad R_4 = \frac{2(l_c - l_{Al})}{k_{air}(1-l_c^2)} \quad (13)$$

$$R_5 = \frac{2l_{Al}}{k_{air}(1-2l_{Al}(1-l_c)-l_c^2)} \quad R_6 = \frac{2l_{Al}}{k_{Al}(2l_{Al}(1-l_c))} \quad (14)$$

$$R_7 = \frac{2l_{Al}}{k_{salts}l_c^2}$$

$$k_{eff} = 2/R_{eff} = 2/(R_{eff1} + R_{eff2} + R_{eff3}) = \frac{2}{\frac{R_1 R_2}{R_1 + R_2} + \frac{R_3 R_4}{R_3 + R_4} + \frac{R_5 R_6 R_7}{R_6 R_7 + R_5 R_7 + R_5 R_6}} \quad (15)$$

Similarly, in case of other combinations, viz, Al(air)+salts, air(salts)+Al, air(Al)+salts, salts(air)+Al, salts (Al)+air are calculated in the same way. It can be seen from Fig.4, despite the trials with all possible combinations, no satisfactory fitting could be obtained.

Fig.4: the comparison between experimental data and all the combinations of Hsu et al. model

4.3 Modified approach

4.3.1 parallel and serial arrangement

Fig.5: modified three-dimensional cube (a) its unit cell spatially periodic chlorides + air, (b) its unit cell spatially periodic Al+ chlorides + air system.

It is important to estimate the sensitivity of metallic Al and air content to the resistance of the matrix, individually, thus understanding their geometry arrangements. In the real process, based on the observations that

- a) metallic Al has a larger particle size than that of the chlorides, and also
- b) the shapes may vary with different conditions due to the excellent ductility of metallic Al.

Aluminium can be considered to behave more like a fluid working as binder between the layers as shown in Fig.5b. It is normal to assume a serial air layer, as marked layer 1 in Fig.5a. However, the resistance of serial air layer is extreme sensitive to the thickness, *i.e.* (1-a) needs to be well-defined, otherwise, this would cause larger derivations. It is safer to avoid such arrangement, describe the system instead, using the arrangement as shown in Fig.5b.

In case of Al+ chlorides+ air system:

$$a = 1 - \frac{\phi_{Al}}{1}, \quad A = \frac{\phi_{salts}}{a} \quad (16)$$

Where a is the thickness of the serial metallic aluminum layer, A is the area of serial salts layer which faces to the heat flux.

$$R_{eff1} = \frac{1-a}{k_{Al}} \quad (17)$$

$$R_{eff2} = \frac{a}{A \times k_{salts} + (1-A)k_{air}} \quad (18)$$

$$k_{eff} = \frac{1}{R_{eff1} + R_{eff2}} \quad (19)$$

A comparison between the model calculation and experimental data is presented in Fig. 6.

4.3.2 Dunn et al. approach

Dunn et al.[17] have investigated the thermal conductivity of multiphase ellipsoidal inclusion composites using Eshelby's model[18]. The thermal conductivity of composites containing pores can be considered as a special case of multiphase composites, and therefore, can be obtained as follows:

$$\frac{1}{k_{cp}} = \frac{k_m}{k_c k_p} + \frac{(k_m - k_p)(k_c - k_m)}{3k_m k_c k_p} \quad (20)$$

$$k_p = \frac{1-Fp}{1+(\eta_2-1)Fp} k_m \quad (21)$$

$$Fp = \frac{\phi_{air}}{1-\phi_{Al}} \quad (22)$$

Where F_p , k_c , k_m , k_p , and k_{cp} are the volume fraction of pores, the thermal conductivities of the fully dense composites, the matrix, the matrix with some porosity, and the composites containing pores, respectively. k_c can be calculated by eqn. (23)[19-21], where the interfacial thermal condition is taken into account on the basis of Eshelby's model.

$$k_c = \frac{2\lambda(1-F) + \beta[3 + (\lambda-1)(1+2F)]}{\lambda(2+F) + \beta[3 + (\lambda-1)(1-F)]} k_m \quad (23)$$

$$F = \frac{\phi_{Al}}{1-\phi_{air}} \quad \lambda = \frac{k_{Al}}{k_{salts}} \quad (24)$$

Where F is the volume fraction of the metallic Al, the parameter β represents the interfacial thermal condition of the compositions. Value of β between 0 and ∞ represents the range of interfacial thermal conditions. In composites, an internal stress in the grains is generated by the mismatch and the scattering of the energy. Therefore, it is reasonable that a value, $\beta=8$ can be assigned in order to express generically the micro structural effect on the thermal conductivity of composites.

Fig.6: the comparison between experimental data and the prediction of parallel and serial arrangement, Dunn et al. model.

In Fig.6, the curve with red line shows a lower sensitivity to the increase content of Al in the compact compared with Dunn et al model, as a result of all of metallic Al phase was arranged in the serial position. The possibility of some part of metallic Al phase may be present on the parallel position was considered in these calculations, but this contribution to the final value of thermal properties is so difficult to estimate.

Despite the difference between the experimental data and Dunn et al model, it is seen that the trend regarding the increase of thermal conductivity with the increase of metallic Al in the salt composite is reasonably well-represented considering the experimental scatter and the uncertainties in the calculation of the thermal conductivities from the thermal diffusivities. Thus, in the present work, Dunn et al. model was used to predict the thermal conductivity in a narrow range of metallic Al in the salt cake corresponding to various porosity levels so that the content of metallic Al in the salt cake from secondary aluminium smelting can be estimated with reasonable accuracy. Such an exercise is shown in Fig.7

Fig. 7: prediction by Dunn et al model with relate to different content of metallic Al

In order to compare the thermal conductivities of the salt-Al composites with that of the industrial salt cake sample, some approximations have to be made with respect to the density and the specific heat of the industrial sample. For the composition of aluminum salt cake as presented in Table 2, it is assumed as the first approximation that the density is close to that of Al_2O_3 , *i.e.* 3.8g/cm^3 , the specific heat is close to 1J/gK . The thermal conductivity in this case would be only 3.11W/mk . Thus, the thermal conductivities of the salt cake from the aluminium industry would have a much lower thermal conductivity (after making assumptions regarding the density and specific heat of the sample) than the salt-sample composites used in the present work. Thiele [22] proposed that during the skimming the first oxide formed at $700\text{-}850^\circ\text{C}$ temperature in industrial practice, a tight protective layer of $\gamma\text{-Al}_2\text{O}_3$, a spinel-like cubic structure is formed around the metallic Al, which brings the oxidation of the enclosed metal quickly to a halt. This insulating layer of Al_2O_3 is compounded by the air layer surrounding each Al particle will affect the thermal diffusivity value and lead a discrepancy as observed in the present work.

In the practical application, the interfacial condition of metallic Al particle in the salt cake needs to be considered and which would vary with different types of drosses as mentioned in the introduction section. It can be treated like β in the Dunn et al. modeling

to adjust the validity region of the prediction. Dross with high metal content usually has a lower porosity due to the abundant liquid aluminum which could fill the existent pores. [23, 24] The connectivity condition between the granules would be changed under such circumstance compared with salt cake. Despite of so many variable factors, as long as the contribution of free aluminum in the dross to the thermal conductivity falls into an analyzable region, it could be a new standard method for evaluating of the aluminum content.

5. Conclusions

Thermal diffusivity measurements as a function of temperature on salts-Al composites having various compositions (0, 2, 4, 6, 8, 10, 12wt pct metallic Al) were carried out in the present work. The thermal diffusivity and the thermal conductivity were found to increase with the metallic Al content. The experimental results are in reasonable agreement with the Dunn et al model.

The complexity of the salt cake leads a discrepancy compared with the prediction by Dunn et al model based on the composition of reference salts-Al composites. Further work will be focused on including the interfacial condition of metallic Al particle in the salt cake as a new parameter in the thermal conductivity model for accurate prediction, along with an estimation of the resistance offered by the oxide layer around aluminium particles.

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