# Stochastic Modelling of Removability of NaCl in Sintering and Dissolution Process to Produce Al Foams

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**Abstract.** The sintering and dissolution process (SDP) is a novel method for manufacturing Al foams. One concern of the process is the presence of residual NaCl in the as manufactured Al foams under certain circumstances, which may have undesirable effects on the properties of the foams. This paper develops a stochastic model to predict the fraction of entrapped NaCl based on the assumptions that the particles of the Al and NaCl powders used in SDP are spherical and monosized and that they are distributed randomly in the preform. The model predicts that the fraction of entrapped NaCl in the foam decreases with increasing volume fraction of NaCl in the preform and with decreasing NaCl-to-Al particle size ratio. The model predictions are in general agreements with the preliminary experimental measurements. The model provides a basis for the selection of Al and NaCl powders in order to minimise the entrapped NaCl in the foam.

Keywords: sintering and dissolution process, aluminium foam, residual NaCl, stochastic modelling

#### Introduction

Metal foams, especially Al foams, have recently attracted many interests in both academia and industry because of their good combinations of strength, stiffness, ductility, temperature capability and durability [1–6]. With the development of commercially viable manufacturing technologies, metal foams have found increasing applications in a wide range of structural and functional products, for example, non-flammable ceiling and wall panels for thermal and sound insulation, roadside fences for noise reduction, heat exchangers and filters. The most important attribute of metal foams, however, is their capability of absorbing large amount of impact energy. They can be used as lightweight panels for buildings against buckling and impact, crashboxes and passenger-door inserts in cars to improve the crashworthiness and passenger safety, and protective skins of military vehicles against explosives and projectiles.

The sintering and dissolution process (SDP) is a novel method developed for manufacturing Al foams [7–9]. In SDP, an Al or Al alloy powder is first mixed with a NaCl powder at a pre-specified volume ratio and compressed into a preform under an appropriate pressure. In most cases, a small amount of Mg powder is also added in the powder mixture to improve the subsequent sintering process. The preform is then sintered at a temperature either slightly above or slightly below the melting point of the Al matrix for 2.5-50 hours, depending on the composition and size of the preform, so that the Al matrix forms a well-bonded networked structure. The sintered preform is finally placed in a hot water bath to dissolve the NaCl particles, leaving behind an Al foam. SDP is one of the most cost-effective routes for producing Al foams. The main advantages of SDP are: (a) the pore size, pore morphology and porosity of the foam can be controlled accurately by selecting an appropriate NaCl powder and its volume fraction in the Al-NaCl powder mixture; (b) either homogeneous

or gradient distribution of pores can be achieved in the foam by mixing the Al and NaCl powders thoroughly or by varying the Al-NaCl ratio gradually in the mixture; (c) a wide range of pore sizes from 0.1 to 3 mm can be achieved; and (d) net-shape foam components or skinned panels can be fabricated in a single step.

One of the issues concerned with the applications of SDP, however, is the presence of residual NaCl in the resultant foam under certain circumstances. Excessive residual NaCl can not only result in post manufacture corrosion of the foam but also increase the relative foam density, leading to deteriorated structural, mechanical and chemical properties of the foam. It is therefore desirable that the residual NaCl in the foam is reduced to a minimum level, if not completely eliminated. There is a need to study the effect of process conditions on the removability of NaCl in SDP.

The preliminary experimental studies [7, 8] have shown that the amount of entrapped NaCl in the as manufactured foam is mainly affected by the composition of the initial Al/NaCl powder mixture, i.e. the volume fraction of NaCl in the preform, and the particle sizes of the Al and NaCl powders. With a high volume fraction of NaCl in the preform, most NaCl particles are in contact with each other and form a continuous threedimensional network. Because all the NaCl particles in the network can be dissolved away by water, there is only small amount of residual NaCl in the resultant foam. In contrast, with a low volume fraction of NaCl in the preform some NaCl particles, either individual or clustered, are enclosed completely by the Al matrix. These isolated NaCl particles cannot be dissolved away and remain in the foam. The attainable porosity of the foams is usually limited to the range 50-85%. At a certain volume fraction of NaCl in the preform, an inappropriate NaCl-to-Al particle size ratio can also lead to significant residual NaCl in the resultant foam. The higher the particle size ratio, the lower the particle number ratio between NaCl and Al in the preform. In other words, an NaCl particle has a higher probability to be surrounded by Al particles. As a consequence, more NaCl particles are likely to be enclosed by the Al matrix after compaction and sintering and retained in the foam. Despite the validity of these phenomenological explanations, quantitative and mechanistic understanding of the relationship between the residual NaCl and the process conditions is still lacking.

This paper is to develop a stochastic model for predicting the amount of residual NaCl entrapped in an Al foam for a given process condition and to discuss its applicability in real situations, with an aim to provide a scientific basis for the selection of Al and NaCl powders for use in SDP.

## Model

In SDP, the Al and NaCl powders are first thoroughly mixed at a pre-specified volume or mass ratio. The powder mixture is then compacted under a high pressure, where the Al particles undergo plastic deformation whereas the NaCl particles remain intact. After full compaction, the preform becomes a composite of a continuous Al matrix embedded with the NaCl particles. In most cases, the NaCl particles that are in contact with each other in the pre-compaction mixture will remain so in the preform after compaction and form a continuous network. Those NaCl particles that are surrounded by Al particles and separated from any other NaCl particles in the pre-compaction mixture will be enclosed completely by the Al matrix after compaction. The subsequent sintering of the preform results in the bonding between the deformed Al particles in the matrix and a small amount of shrinkage, associated with a reduction in the porosity, but has virtually no influence on the spatial distribution of the NaCl particles in the Al matrix. The removability of NaCl in the subsequent dissolution is therefore largely determined by the connectivity of the NaCl particles in the pre-compaction powder mixture. The networked NaCl particles will be dissolved in water whereas the isolated NaCl particles, whether single or in clusters, will be entrapped in the Al matrix. The problem of removability of NaCl in SDP therefore becomes a study of the connectivity of the NaCl particles in the pre-compaction powder mixture.

In the development of the mathematical model for predicting the volume fraction of the isolated NaCl particles in an Al/NaCl preform, the following assumptions are made: (1) the particles of both the Al and NaCl powders are spherical and monosized; (2) the Al and NaCl particles are distributed completely randomly in the Al/NaCl powder mixture, as shown schematically in Fig. 1(a); and (3) the connectivity of the NaCl particles, i.e. the number and locations of the contacts between the NaCl particles, in the Al/NaCl powder mixture remains unchanged after compaction and sintering, as shown schematically in Fig. 1(b).

Let us consider an Al/NaCl powder mixture of a unit net volume, i.e. excluding porosity, with NaCl having a volume fraction of f and Al (1 - f). The numbers per



Figure 1. Schematic diagrams of (a) an Al/NaCl powder mixture and (b) a subsequently compacted and sintered Al/NaCl preform.

unit volume of the NaCl and Al particles in the powder mixture,  $n_{\text{NaCl}}$  and  $n_{\text{Al}}$  respectively, are

$$n_{\rm NaCl} = \frac{f}{\frac{4}{3}\pi R^3}; \quad n_{\rm Al} = \frac{1-f}{\frac{4}{3}\pi r^3}$$
 (1)

where *R* and *r* are the radii of the NaCl and Al particles, respectively. The total surface area per unit volume of the particles of the two powders in the mixture is

$$S = n_{\text{NaCl}} 4\pi R^2 + n_{\text{Al}} 4\pi r^2 = \frac{3f}{R} + \frac{3(1-f)}{r} \quad (2)$$

The connectivity of NaCl particles in an Al/NaCl powder mixture can be examined by considering the number of direct contacts between the NaCl particles. A contact between two NaCl particles can be seen as associated with the surface area of a sphere crown,  $A_c$ , for each of the particles, as shown in Fig. 2. The size of



*Figure 2.* Schematic diagram showing the dependence of the surface area of the sphere crown associated with a NaCl/NaCl contact on the relative sizes of the Al and NaCl particles.

the sphere crown depends on the sizes of the NaCl and Al particles. For a close packed Al/NaCl powder mixture, the Al particles fit into the interstices between the neighbouring NaCl particles. The circumference of the sphere crown can be determined by drawing a straight line between the centre of a NaCl particle and the contact point between the neighbouring Al and NaCl particles, as shown in Fig. 2. The surface area of the sphere crown can be calculated by

$$A_c = 2\pi R H = 2\pi R^2 \left( 1 - \frac{\phi + 2}{\sqrt{\phi^2 + 6\phi + 5}} \right) \quad (3)$$

where *H* is the height of the sphere crown and  $\phi$  is the NaCl-to-Al particle size ratio (*R*/*r*). The detailed derivation of the expression of *H* in terms of *R* and *r* is given in the Appendix.

As a sphere crown with an area  $A_c$  on an NaCl particle has a potential to form a contact with another NaCl particle, each sphere crown can be treated as a point. The number of such points in the powder mixture of a unit solid volume is therefore

$$N = \frac{n_{\text{NaCl}} \cdot A}{A_c} \tag{4}$$

where  $A = 4\pi R^2$  is the surface area of a single NaCl particle.

The number of NaCl/NaCl contacts in the mixture is largely determined by the total surface area of the NaCl particles relative to that of the Al particles. The random distribution of the NaCl particles in the powder mixture can be treated as a situation where the points are distributed randomly on the collective surface of all the particles in the mixture. The problem then becomes to examine whether a point falls on the surface of a NaCl particle or not. If the point falls on the surface of a NaCl particle, it is an NaCl/NaCl contact; otherwise, it is a NaCl/Al contact. The density of points on the total surface, i.e. the number of points per unit surface area, is

$$\lambda = \frac{N}{S} \tag{5}$$

On average, the number of points on the surface of a single NaCl particle is therefore

$$\mu = \lambda A = \frac{2}{\left(1 - \frac{\phi + 2}{\sqrt{\phi^2 + 6\phi + 5}}\right) \left(1 - \phi + \frac{\phi}{f}\right)}$$
(6)

Because these points are NaCl/NaCl contacts,  $\mu$  is actually the average number of NaCl particles in contact with a NaCl particle in the powder mixture.  $\mu$  is named as coordination number for the NaCl particles but is not necessarily an integer.

The random distribution of points over a surface can be described by the Poisson distribution [10]. For an overall point density of  $\lambda$ , the probability that there are *n* points falling on the surface of an NaCl particle is

$$P_n = \frac{(\lambda A)^n}{n!} e^{-\lambda A} = \frac{\mu^n}{n!} e^{-\mu}$$
(7)

where n is either 0 or a positive integer.

Let us first consider the single NaCl particles that have no contact with any other NaCl particles in the powder mixture. The fraction of these particles with respect to all the NaCl particles in the Al/NaCl powder mixture is equal to the probability that no points fall on the surface of a single NaCl particle and can therefore be expressed by

$$p_1 = P_0 = e^{-\mu} \tag{8}$$

Between a pair of NaCl particles that are in contact with each other but not in contact with any other NaCl particles, each particle has one contact point. The fraction of these particles with respect to all the NaCl particles in the Al/NaCl powder mixture is equal to the probability that there is one point falling on the surface of each of the NaCl particles and can be expressed by

$$p_2 = P_1 \cdot P_1 = \mu^2 e^{-2\mu} \tag{9}$$

A cluster of 3 NaCl particles that are in contact with each other but not in contact with any other NaCl particles in the powder mixture can appear in two forms. The numbers of NaCl/NaCl contacts that the particles can have are either (2, 2, 2) or (2, 1, 1). The fraction of these particles with respect to all the NaCl particles in the Al/NaCl powder mixture is equal to the sum of the probabilities that there are two points falling on the surface of each of the NaCl particles and that there are two points falling on the surface of one particle and one point on the surface of each of the other two particles. It can be expressed by

$$p_3 = P_2^3 + P_2 \cdot P_1^2 = \left(\frac{1}{8}\mu^6 + \frac{1}{2}\mu^4\right)e^{-3\mu}$$
(10)

A cluster of 4 NaCl particles that are in contact with each other but not in contact with any other NaCl particles can appear in 6 forms: (3, 3, 3, 3), (3, 3, 2, 2), (3, 2, 2, 1), (3, 1, 1, 1), (2, 2, 2, 2) and (2, 2, 1, 1). The fraction of these particles with respect to all the NaCl particles in the Al/NaCl powder mixture can therefore be expressed by:

$$p_{4} = P_{3}^{4} + P_{3}^{2} \cdot P_{2}^{2} + P_{3} \cdot P_{2}^{2} \cdot P_{1} + P_{3} \cdot P_{1}^{3} + P_{2}^{4} + P_{2}^{2} \cdot P_{1}^{2} = \left(\frac{1}{1296}\mu^{12} + \frac{1}{144}\mu^{10} + \frac{5}{48}\mu^{8} + \frac{5}{12}\mu^{6}\right)e^{-4\mu}$$
(11)

Similarly, the fraction of a cluster of any number of particles can be predicted by adding up the probabilities of all the forms the cluster can appear. The fraction of all the NaCl particles entrapped in the foam, with respect to all the NaCl particles in the initial Al/NaCl powder mixture or perform, is therefore the sum of all the isolated NaCl particles in single, pair or cluster forms. In practice, however, the probabilities of the isolated clusters of more than 4 particles are tedious to calculate and often unnecessary because they are often very small. It can be demonstrated that  $p_n$  decreases rapidly with increasing n, i.e. the more NaCl particles a cluster has the less likely it is completely isolated from the other NaCl particles. The probability that a cluster of four or more NaCl particles is not in contact with any other NaCl particles in the Al/NaCl preform is negligibly small compared with those of the single, double or triple particles. The fraction of all the entrapped NaCl

particles can be approximated by

$$p \approx p_1 + p_2 + p_3$$
  
=  $e^{-\mu} + \mu^2 e^{-2\mu} + \left(\frac{1}{8}\mu^6 + \frac{1}{2}\mu^4\right)e^{-3\mu}$  (12)

#### **Illustrations and Discussion**

Figure 3 shows the fraction of entrapped NaCl as a function of the coordination number, as calculated from Eq. (12), as well as the contributions from the single, double, triple and four particle clusters, as calculated from Eqs. (8)-(11). The fraction of entrapped NaCl decreases rapidly with increasing coordination number. It is more than half at  $\mu = 1$  but is reduced to less than 1/3 at  $\mu = 2$ , 1/10 at  $\mu = 3$  and 1/20 at  $\mu = 4$ . When the coordination number is greater than 5, the entrapped NaCl is negligibly small. It is also shown that the majority of the entrapped NaCl particles are single particles. The fraction of the isolated pairs of NaCl particles is also considerable, reaching a maximum of 0.13 at  $\mu = 1$ . The contributions of the isolated clusters of 3 or 4 particles are relatively small. The isolated clusters of 5 or more particles are very rare. It should be noted that the fraction of the entrapped NaCl is expressed with respect to the volume of the NaCl in the initial Al/NaCl powder mixture or preform. The volume fraction of the entrapped NaCl with respect to the foam is equal to the product of p and f and is therefore much smaller. From a practical point of view, a coordination number greater than 4 is sufficient to achieve a clean foam with insignificant residual NaCl.



*Figure 3.* Relationship between the fraction of entrapped NaCl and the coordination number.



*Figure 4.* Constant-coordination-number curves showing the variations of coordination number with the volume fraction of NaCl in the initial Al/NaCl preform and the NaCl-to-Al particle size ratio.

The coordination number depends not only on the volume fraction of NaCl in the initial Al/NaCl powder mixture or perform but also on the NaCl-to-Al particle size ratio. Figure 4 shows a selected number of constant-coordination-number curves against the volume fraction of NaCl in the preform, f, and particle size ratio,  $\phi$ , as obtained from Eq. (6). It is shown that the coordination number increases with increasing volume fraction of NaCl in the preform and with decreasing NaCl-to-Al particle size ratio. The effect of particle size ratio, however, is only marked when it is below 6. In theory a lower NaCl-to-Al particle size ratio is favourable because for a fixed mixture composition a lower particle size ratio leads to a higher coordination number and consequently a lower fraction of entrapped NaCl in the foam. In practice, however, the particle size of the Al powder normally needs to be smaller than that of the NaCl powder. In order to ensure that the pore shapes replicate the geometries of the NaCl particles, the Al particles needs to be small enough to squeeze into the interstices between the NaCl particles. Furthermore, the size of the Al particles also determines the minimum thickness of the cell walls of the foam. There is an upper limit to the Al particle sizes that can be used in SDP. Therefore, the NaCl-to-Al particle size ratio is normally greater than 1.

Figure 4 can be used to graphically determine the coordination number for a given set of f and  $\phi$  values and subsequently to estimate the residual NaCl in the resultant foam. It can also be used for identifying the optimum particle size ratio required to obtain a certain coordination number for a given volume fraction of NaCl in the preform. For example, to achieve a coordination number of 4 for a volume fraction of NaCl



*Figure 5.* Variations of the fraction of entrapped NaCl with the volume fraction of NaCl in the initial Al/NaCl preform at different NaCl-to-Al particle size ratios, compared with the experimentally determined values [8].

of 0.5 in the preform, the NaCl-to-Al particle size ratio cannot exceed 5.

Figure 5 shows the variations of the fraction of entrapped NaCl, p, with the volume fraction of NaCl in the initial preform, f, at a series of particle size ratios,  $\phi$ , compared with the experimentally determined values reported in [8]. In the experiments [8], commercially pure Al and NaCl powders composed of nearly spherical particles with particle sizes ranging from 100 to 400  $\mu$ m and from 100 to 1000  $\mu$ m, respectively, were used. Both the Al and NaCl powders were divided into two different size groups using an analytical sieve and were then mixed into different NaCl/Al size combinations:  $(106-250 \ \mu m)/(250-$ 400  $\mu$ m), (250–1000  $\mu$ m)/(250–400  $\mu$ m), (160–250  $\mu$ m)/(53–106  $\mu$ m) and (250–1000  $\mu$ m)/(53–106  $\mu$ m), corresponding to mean particle size ratios of 0.6, 1.7, 2.4 and 7.5, respectively. The powder mixtures were first put into  $\phi 21 \times 50$  mm steel tubes and cold compacted under a pressure of 200 MPa. The specimens were then sintered in an electrical radiation furnace at a temperature of 650°C for 2.5-30 hours and cooled down to the room temperature. They were finally placed in flowing hot water at  $\sim 95^{\circ}$ C for several hours until all the free NaCl particles were completely dissolved. The weights of the specimens at each processing stage were measured and thus the fractions of the residual NaCl in the final foam specimens were obtained. Figure 5 shows that the model predictions show the same trends as those of the experimental values. They agree very well quantitatively, except for relatively high particle size ratio conditions ( $\phi = 2.4$  and  $\phi = 7.5$ ) where the

measured values are higher than the predictions. It is shown that the fraction of entrapped NaCl decreases with increasing volume fraction of NaCl in the preform for a fixed particle size ratio and increases with increasing NaCl-to-Al particle size ratio for a fixed volume fraction of NaCl in the preform. When the volume fraction of NaCl in the preform is above 0.7, there is virtually no NaCl entrapped in the resultant foams. When the particle size ratio is kept below 2 the fraction of entrapped NaCl is small for any volume fractions of NaCl in the preform above 0.3.

It should be noted that the experimentally determined fractions of entrapped NaCl in the Al foams made by SDP were based on nominal NaCl-to-Al particle size ratios [8]. The nominal particle size ratios of 0.6, 1.7, 2.4 and 7.5 corresponded to wide ranges of 0.3–1.0, 1.0–5.0, 0.6–4.0 and 2.3–20, respectively [8]. They deviated significantly from the assumptions under which the present model is developed.

The assumptions made in the development of the present model are often difficult to be satisfied completely in practical SDP. The particles of the NaCl powders used in SDP are normally rounded cubic rather than spherical. Both the Al and NaCl powders usually have varied particle sizes within controlled ranges. The powder mixtures are always somewhat looser than the close packing conditions. It is also unavoidable that the particles in the powder mixture undergo rearrangement during compaction. Despite these errors discussed above, the model can provide simple and often reliable estimations.

Under extreme conditions the model itself can give intermediate values that contradict the reality. It does not take into account the fact that the maximum coordination number for monosized NaCl particles is 12, which is reached when the particles are closely packed and occupy 74% of the sample volume. Instead, the coordination number as calculated from Eq. (6) can exceed 12 when the volume fraction of NaCl in the initial Al/NaCl powder mixture is above a certain value. This critical value, at which the coordination number is 12, is higher than 0.8 for  $\phi > 1$  and lower than 0.8 for  $\phi < 1$ . This inconsistency, however, does not affect the predictions on the fractions of entrapped NaCl in the Al foams. Firstly, any coordination number greater than 6 would lead to virtually no entrapped NaCl and makes no difference. Secondly, the volume fraction of NaCl in the Al/NaCl preform concerned in SDP is normally in the range 0.5–0.8 [7, 8], because more NaCl would result in a discontinuous Al network and less NaCl would result in too much residual NaCl in the foam.

### Conclusion

The fraction of entrapped NaCl in the Al foam manufactured by SDP is a function of the volume fraction of NaCl in the initial Al/NaCl powder mixture and the NaCl-to-Al particle size ratio. Assuming that the particles of both the Al and NaCl powders are spherical and monosized and that they are distributed randomly in the powder mixture and subsequent preform, the fraction of entrapped NaCl in the foam with respect to the volume of NaCl in the preform can be expressed by

$$p \approx e^{-\mu} + \mu^2 e^{-2\mu} + \left(\frac{1}{8}\mu^6 + \frac{1}{2}\mu^4\right)e^{-3\mu}$$

where  $\mu$  is an average coordination number for the NaCl particles in the Al/NaCl powder mixture and is related to the volume fraction of NaCl in the preform, f, and the NaCl-to-Al particle size ratio,  $\phi$ , by

$$\mu = \frac{2}{\left(1 - \frac{\phi + 2}{\sqrt{\phi^2 + 6\phi + 5}}\right)\left(1 - \phi + \frac{\phi}{f}\right)}$$

The predictions of this stochastic model are in general agreements with experimental measurements.

#### **Appendix: Derivation of H**

The relationship between the height of the crown sphere, *H*, and the NaCl and Al particle radii, *R* and *r*, can be derived by geometrical analysis. Let us first find out the lengths of *gk* and *gh* in Fig. 2. Because the right triangles  $\Delta kgh$  and  $\Delta kef$  are similar, there exists a relationship gk:ek = hk:fk, where ek = hk = R and fk = (R + r). *gk* can therefore be expressed in terms of *R* and *r* as

$$gk = \frac{R^2}{R+r} \tag{13}$$

*gh* is related to *gk* by the Pythagorean theorem,  $gh^2 + gk^2 = hk^2 = R^2$ , and can also be expressed in terms of *R* and *r*:

$$gh = \frac{R}{R+r}\sqrt{r(2R+r)}$$
(14)

The height of the sphere crown is simply: H = ae - ac = R - ac. Let us now find out the length of ac. Because the right triangles  $\triangle acd$  and  $\triangle agh$  are similar, there exists a relationship ac:ag = cd:gh, where ag = ah - gk = 2R - gk and cd is related to ac by the Pythagorean theorem,  $cd^2 + ac^2 = ad^2 = R^2$ . Given these relationships, H can be derived as:

$$H = R - \frac{R(R+2r)}{\sqrt{R^2 + 6Rr + 5r^2}} = R\left(1 - \frac{\phi + 2}{\sqrt{\phi^2 + 6\phi + 5}}\right)$$
(15)

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