

MINIMUM SOLID AREA MODELS FOR THE EFFECTIVE PROPERTIES OF POROUS MATERIALS – A REFUTATION

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Minimum solid area (MSA) models are popular models for the calculation of the effective properties of porous materials and are frequently used to justify the use of a simple exponential relation for fitting purposes. In this contribution it is shown that MSA models, and the simple exponentials they support, are misleading and should be avoided. In particular, taking Young's modulus and conductivity (thermal or electrical) as examples, it is shown that MSA models are based on the unjustified (and unjustifiable) hypothesis that the relative Young's modulus and relative conductivity are identical, and moreover equal to the MSA fraction itself. This claim is generally false for isotropic materials, both random or periodic. Although indeed a very specific case exists in which this claim is true for the properties in one specific direction (viz., extremely anisotropic materials with translational invariance), in this specific case MSA models are redundant, because the relative properties are given exactly by the volume- or area-weighted arithmetic mean. It is shown that the mere existence of non-trivial cross-property relations is incompatible with the existence of MSA models. Finally, it is shown by numerical (finite-element) modeling that MSA models provide incorrect results even in the simplest of the cases for which they were originally designed, i.e. for simple cubic packings of partially sintered isometric (initially spherical) grains. Therefore, paraphrasing Box, MSA models are not only wrong, but also useless, and should be abandoned.

INTRODUCTION

Minimum solid area (MSA) models, also called minimum contact area models, have a long tradition in ceramic science and technology. To the best of our knowledge, the idea itself has its roots in powder metallurgy and is mentioned in a 1949 paper by Balshin [1], but in the ceramics literature it has been explicitly mentioned (albeit not under this name) for the first time in Duckworth's discussion [2] of Ryshkewitch's 1953 paper on the "Compression strength of porous sintered alumina and zirconia" [3]. Duckworth gently criticizes the compressive tests, but confirms Ryshkewitch's results for flexural and torsional tests. Then, in an attempt to explain the observed exponential decrease of strength values for ceramics in a relatively wide porosity range (and the failure of the exponential description beyond a certain porosity value), Duckworth suggests further research of this problem, and among the factors to be investigated he suggests the "reduction in the effective cross-sectional area", but at the same time adds the warning that "if this were the only factor, the strength-density ratio would not change with porosity" (which was evidently the case in Ryshkewitch's and Duckworth's data and is just an alternative formulation of the fact that strength-porosity curves are usually not linear). A few years later, in 1959, with the Coble-Kingery data on the flexural strength of

porous alumina already available [4], Knudsen took up the problem and focused attention on tensile strength [5], for which measured data were available at that time for partially sintered iron and steel compacts. It was he who made the first calculations of "intergranular contact area", i.e. the MSA for three simple packings of monosized spherical particles: "cubic" (actually simple cubic), "orthorhombic" (actually simple hexagonal) and "rhombohedral" (actually face-centered cubic or hexagonal closest packed). The results of these basic but tedious calculations (together with the inadequate terminology used by Knudsen) have been reproduced by innumerable (both journal and textbook) authors, too many to be cited here. Knudsen, albeit aware of Balshin's idea [1] to use a power-law relation for fitting strength-porosity dependences, came to the conclusion that the simple exponential relation is more adequate [5], which is not surprising when considering partially sintered metal compacts, although the Coble-Kingery values [4] could have convinced him of the opposite. Knudsen himself was well aware of the fact that for a real material with random microstructure the MSA cannot be easily determined (in fact, without 3D tomography this is impossible). Obviously for this reason he has confined himself to the calculation of the aforementioned three simple models (packings of monosized spheres, including those with the extreme packing fractions 52 % and 74 %),

just in order to show that for sufficiently small porosities the semi-logarithmic plot of the MSA versus porosity exhibits an approximately linear decrease, similar to the tensile strength.

Inspired by Duckworth's and Knudsen's idea to use the simple exponential for fitting the porosity dependence of strength, Spriggs in 1961 applied for the first time a simple exponential relation of the same type for fitting the porosity dependence of the tensile modulus (Young's modulus) [6], but without connecting this use of the exponential to MSA models. The idea to use the simple exponential for the Young's modulus has been immediately welcomed by Knudsen [7] and then extended to the shear modulus by Spriggs and coworkers in 1962 [8, 9].

In the years to come, the simple exponential relation became one of the most popular and most widely used relations for fitting property-porosity dependences, but as far as we know the MSA model fell into oblivion (except for a brief note by Rice in 1976 [10], which seems not to have had any significant impact, however), until Rice revived the MSA concept in the 1990s [11-15], culminating in his 1998 monograph [16], and – being evidently convinced of its usefulness – managed to popularize MSA models among the younger generation of materials scientists [17-25]. A major step beyond earlier researchers was Rice's suggestion to use MSA models also for non-mechanical properties, e.g. conductivity [14, 16]. With this extension by Rice, MSA models can be briefly summarized by the statement that the MSA fraction is not only equal to the relative Young's modulus but also to the relative conductivity.

However, irrespective of the MSA fraction, it was well known (for those who knew it well) already in the 1950s that the relative Young's moduli and relative conductivities cannot be equal in general (the only exception is the very special case of translationally invariant microstructures, for which the properties in one special direction can be calculated exactly via the area-weighted arithmetic mean), since it had been shown by Dewey [26] and Mackenzie [27] that even in the linear approximation (exact solution of the single-inclusion problem for very low porosities) the numerical coefficients (which are a function of the Poisson ratio [28]) for the porosity dependence of elastic moduli of materials with spherical voids are different from those for the thermal or electrical conductivity (for which the value 3/2 had been found by Maxwell [29]). Of course, this difference remains relevant in all corresponding nonlinear relations [30], which must necessarily approach the linear approximations in the low-porosity limit, and also in the case of non-spherical, e.g. spheroidal, pore shape [31, 32].

Moreover, for more than one decade now, there is increasing evidence for the existence of so-called cross-property relations, i.e. interrelations between different relative properties, e.g. the relative Young's modulus and

electrical or thermal conductivity [33-36]. In this paper we show that the mere existence of non-trivial cross-property relations is incompatible with MSA models. Further we show that MSA models are not only wrong, but also useless and highly misleading.

RESULTS AND DISCUSSION

Effective properties, relative properties, micromechanical bounds and elementary cross-property relations

Effective properties are the overall, macroscopic properties of heterogeneous materials, i.e. materials with internal phase boundaries. In the case of multiphase materials, including porous materials as a special case, they depend on the volume fractions (e.g. the porosity ϕ) and all other details of the microstructure. Relative properties of porous materials P_r are ratios of effective properties and the corresponding properties of the dense materials, i.e.

$$P_r = \frac{P}{P_0} \quad (1)$$

where P is the effective property of the porous material and P_0 the property of the dense solid phase (or phase mixture). For certain properties, e.g. elastic moduli and thermal or electrical conductivity, micromechanical bounds are available that delimit the domain of the possible values these properties may attain [37].

Although the homogenization models proposed by Voigt [38] and Reuss [39] were recognized in 1952 by Hill [40] to be universally valid upper and lower bounds, respectively, for the effective elastic moduli of dense polycrystalline materials, it was not until 1960 that Paul [41] stated that the volume-weighted arithmetic mean and the volume-weighted harmonic mean were upper and lower bounds, respectively, also for the effective elastic moduli of multiphase materials. The corresponding bounds for conductivity had been discovered much earlier (in 1912) by Wiener [42]. Now for porous materials, in which the pore phase has zero or negligible property values, the lower bounds of this type degenerate to zero. On the other hand, the upper bounds for the Young modulus and conductivity are identical in this case and can be written as

$$E_r \leq 1 - \phi \quad (2)$$

and

$$k_r \leq 1 - \phi \quad (3)$$

These are universal bounds (Wiener-Paul bounds, in the sequel "WP bounds"), valid for both isotropic and anisotropic microstructures of any type. However, in certain directions of materials with anisotropic microstructures these inequalities degenerate to equalities. This is the case if and only if the microstructures exhibit translational invariance (e.g. materials with oriented pore channels of arbitrary but constant cross section) and only

in the direction parallel to the translation (i.e. parallel to the pore channels). In this case we have the trivial cross-property relation

$$E_r = k_r \quad (4)$$

Moreover, for a translationally invariant microstructure the solid volume fraction (i.e. the relative density of the material or the packing fraction of the particles) is equal to the solid area fraction in any cross section perpendicular to the direction of translation. This solid area fraction is constant along the translation direction and therefore (in this specific direction) at the same time equal to the MSA fraction, i.e.

$$1 - \phi = \phi_s = \frac{A_s}{A} \Bigg|_{\text{any section}} = \min \left(\frac{A_s}{A} \right), \quad (5)$$

where ϕ_s is the solid volume fraction, A the total section area and A_s the area of solid sections, so that the ratio of the latter two is the solid area fraction (in the above case a constant value for any section in this specific direction). The MSA hypothesis can be written in the simple form

$$\min \left(\frac{A_s}{A} \right) = E_r = k_r. \quad (6)$$

Unfortunately, this equation, although valid in the aforementioned very specific case, is wrong in general. That means, except for the aforementioned very special anisotropic microstructure, for which the relative elastic modulus and conductivity in one special direction can be exactly calculated via the volume- or area-weighted arithmetic mean (linear mixture rule), there is no other microstructure for which any of the equality signs would be valid. In particular, this implies that MSA models cannot be correct for any isotropic microstructure, irrespective of whether it is random or not.

Shortly after Paul's finding that the volume-weighted arithmetic and harmonic means are upper and lower bounds, respectively, of the effective elastic moduli, Hashin and Shtrikman, using variational principles, found more restrictive bounds for the effective conductivity and elastic moduli of isotropic materials [43, 44]. Again, for porous materials the lower bounds degenerate to zero identically. The upper Hashin-Shtrikman bounds ("HS bounds") are

$$E_r \leq \frac{1 - \phi}{1 + \phi} \quad (7)$$

for the Young's modulus (to an excellent approximation, but principally dependent on the Poisson ratio [28]) and

$$k_r \leq \frac{1 - \phi}{1 + \phi/2} \quad (8)$$

for the (electrical or thermal) conductivity (exactly [30]). As in the case of the WP bounds, also the HS bounds can be realized ϕ exactly by certain microstructures, at least in principle [37], and materials with microstructures

that obey the upper HS bound for the Young's modulus (or other elastic moduli) will obey it also for the conductivity. Therefore, just by solving one of the two equations with respect to the porosity and inserting into the other we obtain the cross-property relation [35]

$$E_r = \frac{3k_r}{4 - k_r}. \quad (9)$$

This HS-based cross-property relation is an excellent (and for practical purposes completely sufficient) approximation to the cross-property relation

$$E_r = \left[\left(\frac{(1 - \nu_0)(9 + 5\nu_0)}{7 - 5\nu_0} \right) (k_r^{-1} - 1) + 1 \right]^{-1} \quad (10)$$

found by Sevostianov et al. [33, 34], which takes into account the fact that Equation 7 above is not exact but changes very slightly with a change in the solid Poisson ratio. It is obvious that already the mere existence of such a cross-property relation is in conflict with the MSA hypothesis.

It is clear that for isotropic two-phase microstructures with a uniform random arrangement of grains or pores the area fraction is the same, on average, in arbitrary positioned and oriented sections. Moreover, according to the Delesse-Rosiwal law [45, 46] this area fraction corresponds to the volume fraction. That means, also in this case, similar to the very specific case mentioned above, the solid area fraction in any section is at the same time the MSA fraction. In other words, in this case the MSA fraction does not add any new information, because it is identical to the solid volume fraction (i.e. simply the complement to porosity). On the other hand, for periodic microstructures, e.g. cubic packings of partially sintered isometric particles, a minimum of the solid area fraction does indeed exist (for any direction). However, in this case MSA models treat the microstructures as if they were translationally invariant (which is not the case), erroneously assuming that the MSA fraction is the equal to the solid volume fraction. As a conclusion it can be said that MSA models are wrong in all cases except in cases in which they are redundant.

Cross-property relations based on predictive model relations

Isotropic porous materials with microstructures whose properties realize the HS upper bounds are very rare, and real materials of this type are virtually non-existent. Actually we are not aware of any data in the literature that would indicate Hashin-Shtrikman-type behavior. However, there are many examples of real materials that follow approximately the power-law relations (Gibson-Ashby relations for open cell-foams [47])

$$E_r = (1 - \phi)^2, \quad (11)$$

$$K_r = (1 - \phi)^{3/2}, \quad (12)$$

or our exponential relations [28, 30, 48-52]

$$E_r = \exp\left(\frac{-2\phi}{1 - \phi}\right), \quad (13)$$

$$k_r = \exp\left(\frac{-\frac{3}{2}\phi}{1 - \phi}\right), \quad (14)$$

It should be recalled, that this type of exponential, proposed as a functional-equation-based predictive model by the authors, does not violate the upper HS bound, in contrast to the simple exponential that has been proposed by Spriggs for fitting elastic moduli data [6, 8, 9] and extensively used in the context of MSA models [11-25]. Of course, if desired, also our types of exponential relations could be used for fitting purposes (by letting the numerical coefficients 2 and 3/2 vary), being as flexible as the simple exponential, but avoiding its basic shortcoming. Interestingly, both types of relations, power-law and exponential, yield the same cross-property relation [36]

$$E_r = k_r^{4/3}. \quad (15)$$

According to our experience this is the most realistic of all cross-property relations currently available for materials with isometric pores. It can be expected that, irrespective of whether the porosity dependence of relative properties is closer to the power-law (Equations 11 and 12) or our exponential (Equations 13 and 14), this cross-property relation will provide a good prediction of the relative Young's modulus when the relative conductivity is known and *vice versa*. Actually we have found this cross-property relation to be useful far beyond the range delimited by these two relations. What is more important, however, is the fact that also this non-trivial cross-property relation is obviously in contradiction to the MSA model hypothesis, Equation 6.

Finally, for those who would tend to argue that for small porosities MSA models should be acceptable, we would like to add a cross-property relation for infinitesimally low porosities. For spherical pores this cross-property relation is [36],

$$E_r = \frac{4k_r - 1}{3}, \quad (16)$$

as a simple consequence of the fact that the single-inclusions of the Young's modulus and the conductivity, respectively, are [26, 27, 37]

$$E_r = 1 - 2\phi, \quad (17)$$

$$k_r = 1 - \frac{3}{2}\phi. \quad (18)$$

Of course, this cross-property relation (Equation 16) is not very practical, because infinitesimally low porosities are usually not very interesting, but it clearly shows that even for the smallest porosities the relative Young's

modulus is different from the relative conductivity, and thus even in this case MSA models principally fail. It is true that the numerical coefficients (Maxwell and Eshelby-Wu coefficients) in these relations are functions of pore shape (for a more detailed treatment the reader may refer to our previous papers [31, 32]), but there is no pore shape for which these coefficients are equal for these two properties.

Refutation of MSA models via finite-element modeling

It has been shown that the mere existence of non-trivial cross-property relations contradicts the basic MSA model hypothesis, Equation 6, i.e. seriously questions the admissibility of MSA models. In an attempt to rescue at least a little bit of the MSA concept, some might now want to argue that MSA models may be acceptable for concave pores, i.e. interstitial voids between convex particles, for which they have originally been designed.

This is indeed a question that cannot be answered by invoking analytical models. Therefore, last but not least, we show by numerical (finite-element) modeling that MSA models are wrong even for the simplest thinkable case for which they have been originally designed, i.e. for materials with concave pores (interstitial voids) in a simple cubic packing of isometric grains (initially spherical particles), partially sintered to different density. For this purpose an idealized microstructure of this type has been constructed via the GridGeo module of the commercial GeoDict[®] software package (MATH2 MARKET, Kaiserslautern, Germany). The unit cell consisted of $200 \times 200 \times 200$ voxels, the sphere diameter has been set to $100 \mu\text{m}$, and idealized partial sintering has been simulated by gradually reducing the lattice constant and thus allowing the spheres to overlap. This idealized partial sintering (without neck formation) corresponds exactly to one of the paradigmatic key examples cited in the context of MSA models. Examples of these microstructures are shown in Figure 1. Figure 2 shows the decrease of the porosity from its initial maximum value of 47.6 % for particles with point contact (where the

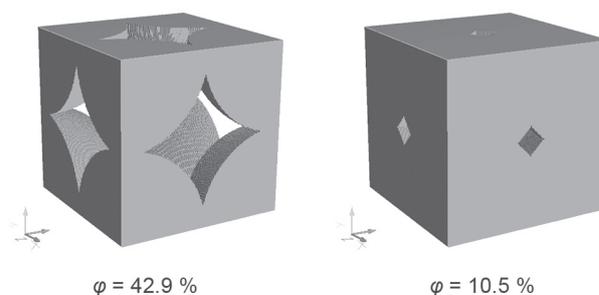


Figure 1. Examples of digital microstructures with different porosity created via idealized partial sintering, i.e. by increasing the particle overlap (without neck formation).

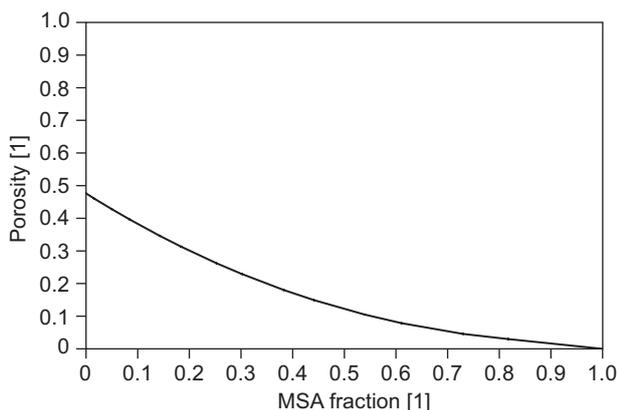


Figure 2. Porosity of partially sintered arrays of isometric (initially spherical) particles in simple cubic packing as a function of the MSA fraction.

MSA fraction is zero) to zero for an MSA fraction of unity, i.e. a dense material.

Based on this digitally created microstructure, the Young's modulus and conductivity have been calculated numerically using the ElastoDict and ConductoDict modules, respectively, of the GeoDict[®] software for material properties with a phase contrast of 1:1000 in the case of conductivity and infinity in the case of the Young's modulus.

Figure 3 shows the MSA fraction dependence of the relative Young's modulus and relative conductivity of partially sintered isometric (initially spherical) particles in simple cubic packing. It is evident that the results for the relative Young's modulus and conductivity are completely different and that both are different from the MSA prediction.

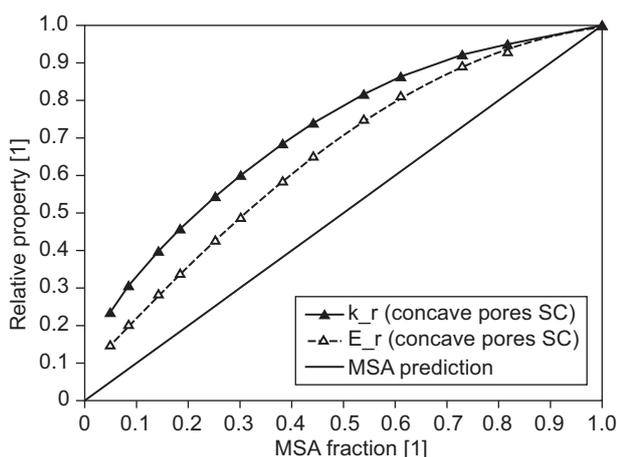


Figure 3. Dependence of the relative Young's modulus and relative conductivity of partially sintered simple cubic packings of isometric (initially spherical) particles on the MSA fraction.

This closes our argumentation. It is clear that when the MSA model fails to predict the relative properties even for the simplest microstructure for which it has been originally designed and for which success of the MSA model has been claimed for decades, the MSA model is

likely to fail in all cases where translational invariance is absent. That means all claims on the apparent success of MSA models were premature and are now known to be wrong.

Thus, irrespective of the fact that the simple exponential commonly used in MSA models has serious disadvantages in itself (even as a fit relation), and irrespective of the answer to the question, which of the cross-property relations is the most realistic (to be answered in a forthcoming paper), it can be said with certainty that

$$\min\left(\frac{A_s}{A}\right) \neq E_r \neq k_r, \quad (19)$$

That means the basic hypothesis of MSA models, Equation 6, and all subsequent claims based on this hypothesis, are wrong.

It is remarkable that Spriggs, who was the earliest proponent of the simple exponential relation (which he used extensively), never propagated MSA models, and it seems that also other "grand old men" of ceramic science never took them too seriously. Probably they intuitively felt that something was wrong with these models, although at that time numerical (finite-element) modeling, which is indispensable to complete the proof, was not yet available. We dare surmise that also Knudsen had an uncomfortable feeling with the (unjustified and unjustifiable) key assumption of his 1959 paper, and this might have been the reason why he himself never returned to MSA models. So it remains to be hoped that the next generation of materials scientists will not be misled by this unlucky heritage in the years to come.

CONCLUSION

The fact that Knudsen drew attention to tensile strength made the MSA concept at first sight highly plausible and has certainly contributed to its further dissemination. It seemed almost intuitively clear that the tensile strength is determined by the weakest cross section. And indeed, up to this point, everything is correct. But Knudsen went one essential step further to conclude that the relative strength is equal to the relative (i.e. normalized) MSA, i.e. the MSA fraction. We are convinced that already this conclusion is wrong, but for strength it is not easy to provide a proof of this claim. Therefore we have made no attempt to do so. Rice, however, drew the logical conclusion that, if tensile strength, then also the tensile modulus (Young's modulus) and the conductivity should be equal to the MSA fraction. This is a statement that is – at least for the simple model structures proposed by Knudsen and Rice – amenable to verification or falsification. What we have shown in this paper is, that the MSA model is wrong in the simplest case that can be checked by numerical calculation, viz. the effective Young's modulus and conductivity of a simple cubic packing of isometric (initially spherical) grains, partially sintered

to different density. Moreover, the mere existence of non-trivial cross-property relations between the Young's modulus and conductivity shows that equality of relative properties and the MSA fraction, as postulated MSA models, is elusive for all microstructures, except for the trivial case of translationally invariant (i.e. extremely anisotropic) microstructures in one specific direction, for which the effective elastic moduli and conductivities are exactly given by the area-weighted arithmetic mean. That means, *cum grano salis* it could be said that MSA models are wrong in all cases, except for those in which they are redundant. It should have become clear, however, that the failure of MSA models does not consist in the fact that they are too idealized to describe real materials. This is a deficiency of most other models as well. The reader should have noticed that our critique does not even mention the fact that in practice there is no feasible way to determine the MSA for real materials. Although this feature is not very nice indeed, because it hinders the use of these models for predictive purposes, many authors seem to accept models that are useful only for fitting purposes and have no predictive value in practice. The crucial point is that even for the simplest case of a partially sintered simple cubic packing of isometric (initially spherical) grains the MSA result is wrong, both for the Young's modulus and the conductivity. With respect to this situation, recalling Box's statement [53, 54] that "all models are wrong, but some are useful", it may be said that MSA models are not only wrong, but also useless.

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