

Simple and precise measurements of fibre volume and void fractions in metal matrix composite materials

JACQUES E. SCHOUTENS

MMCIAC, Kaman Tempo, 816 State Street, Santa Barbara, California 93102, USA

A method for determining the fibre volume fraction, V_f , and the void fraction, V_g , in a metal matrix composite (MMC) material is described. These quantities are determined from specimen weight measurements in air and in a liquid using a laboratory balance. For a material without voids, V_f can be determined with an uncertainty less than 0.5% with a balance precision of 0.01%. By making the same measurements before and after etching away the matrix, using the same balance precision, V_f and V_g can be determined to an uncertainty of about 3 and 6%, respectively. It is also shown theoretically that by indenting a specimen containing no fibres and only a uniform distribution of small voids, the void fraction can also be determined from weight measurements before and after indentation.

1. Introduction

Two of the most important parameters that influence the mechanical properties of metal matrix composite (MMC) materials are the fibre volume fraction and the void fraction. The fibre volume fraction is the most important parameter because it determines the strength and stiffness properties of an MMC composite. The void fraction is a consequence of material processing. It is a parameter often needed for understanding material processing and the development of improvements.

The effects of voids upon the longitudinal strength properties of unidirectional continuous fibre reinforced MMC are often negligible [1] if the voids are uniformly distributed throughout the materials, are small in individual volume, and represent less than about 2 to 3% of the total volume. This follows from the fact that, for a sufficiently large value of fibre fraction, the continuous fibres assume the bulk of the loading on the MMC. Transverse properties may be affected to a greater extent. However, the presence of voids in discontinuous or whisker reinforced MMC materials, even in small fractions, can significantly reduce the material strength. Typically, a 2% void fraction in a 30 volume per cent (vol%) SiC/Al

will reduce the tensile yield strength by about 50%. The same void fraction in a unidirectionally continuous fibre reinforced MMC will result in a reduction in longitudinal tensile yield strength of less than 1% [1].

Consequently, from the above discussion and the need to ascertain ever more precisely the strength properties of MMC materials, there is a continuing requirement for measuring these parameters with precision. The methods for measuring fibre volume fraction can involve exceedingly complex equipment such as an image analysing television scanner and computer [2] with a precision no better than 1 to 2%. The measurement of void fraction is generally accomplished from metallurgical sections using statistical analysis. Both methods require the destruction of the sample and are time consuming and often expensive.

Whittenberger *et al.* [3] have investigated the determination of fibre fraction in resin reinforced composites. They show that the volume fraction is directly relatable to the lamina thickness even for fibre orientation as large as 45 degrees from the normal to a cut section. By a simple calculation, it can be shown that the method yields the rule of mixtures.

This paper discusses the simple non-destructive method of measuring the fibre volume fraction using a precision balance. The method is ideal for continuous fibre reinforced MMC where the void fraction is unimportant from the standpoint of strength. The analysis is extended to include the measurement of void fraction. However, in this case it is necessary to etch away part of the matrix material. This paper also discusses the expected precision attainable and the requirement for such precision, and exemplifies the analyses with numerical results for boron/aluminum and graphite/aluminum.

2. Comments on precision balance measurements

Recently, Pratten [4] reviewed the measurement of the density of small samples and showed that such measurements can be carried out to a high degree of precision. However, he does not make specific reference to MMC. Density measurements are of three types [4]: ultra-precise density measurements of large objects carried out by standards laboratories; approximate density measurements, to within $\pm 1\%$, of very small objects; and the precise measurement of the density of small samples (1 to a few hundred grammes).

The interest in this paper lies primarily in the precise measurement of absolute density values by weighing samples in air and in a liquid. Sample weights considered are of the order of about 100 g or less.

The experimenter using the technique of measurements in air and in liquid must be aware of three sources of errors common to immersion techniques [4]: the adherence of air bubbles to the sample surfaces, often invisible to the unaided eye, causing an artificial increase in volume and buoyancy; the effects of surface tension on the wire supporting the immersed specimen; and temperature fluctuations. The immersion fluid should have a low surface tension (good wetting properties), a low vapour pressure, and a high density. Frequently used liquids are water or diethyl phthalate. The liquid used is usually distilled before use to remove impurities and trapped gases.

3. Analysis

The density of an MMC material containing a volume fraction of fibre, V_f , and matrix, V_m , in addition to a fraction of voids, V_g , is given by

$$\rho_c = \rho_m V_m + \rho_f V_f + \rho_g V_g \quad (1)$$

where ρ is the density and the subscripts refer to matrix, fibre, and gas, respectively. Other than these three phases, no other material is assumed to be present in significant amounts to affect the composite density. From volume conservation

$$V_f + V_m + V_g = 1 \quad (2)$$

which, when used in Equation 1, gives

$$\rho_c = \rho_m(1 - V_f) + \rho_f V_f + (\rho_g - \rho_m)V_g. \quad (3)$$

However, $\rho_g \ll \rho_m$ and $\rho_g \ll \rho_f$; hence, Equation 3 reduces to

$$\rho_c = \rho_m(1 - V_f) + \rho_f V_f - \rho_m V_g. \quad (4)$$

Equation 4 shows that the density of an MMC material is reduced by a quantity $\rho_m V_g$ due to the presence of voids containing gases. The manner in which these voids come about is of no concern here. The fact that these gases may be at very high pressure is not significant to this analysis. Tsai and Hahn [5] show an expression for the composite density based on the mass fraction of the constituents.

In hydrostatic weighing of a specimen, the specimen is weighed in air, W_a , and in a liquid, W_l . The weight in air is

$$W_a = g(M_c - \rho_a v_c), \quad (5)$$

and the weight in a liquid is

$$W_l = g(M_c - \rho_l v_c) \quad (6)$$

where g is the acceleration due to gravity, ρ_a and ρ_l are the air and liquid densities, respectively, and M_c and v_c are the true composite mass and volume of the specimen, respectively. Since $M_c = \rho_c v_c$, Equations 5 and 6 give

$$\rho_{cm} = \frac{W_a \rho_l - W_l \rho_a}{W_a - W_l} \quad (7)$$

where ρ_{cm} stands for the measured composite density. The densities given by Equations 4 and 7 are equal, thus relating the fractions of the MMC constituents to measurements.

Equation 4 can be solved for V_f and V_g , giving the following relations:

$$V_f = \frac{1}{\rho_f - \rho_m} [\rho_{cm} - \rho_m(1 - V_g)] \quad (8)$$

and

$$V_g = 1 - \frac{\rho_{cm}}{\rho_m} - \left(1 - \frac{\rho_f}{\rho_m}\right) V_f. \quad (9)$$

These equations show that either V_f or V_g must be

measured independently of the other. It is shown below that the same specimen can be used to measure V_f and V_g independently.

3.1. Case of zero void fraction

In the absence of voids, Equation 8 or 9 reduces to

$$V_f = \frac{1}{\alpha} \left(1 - \frac{\rho_{cm}}{\rho_m} \right) \quad (10)$$

with $\alpha = 1 - \rho_f/\rho_m$. Thus to obtain the fibre volume fraction, it is necessary to measure only the composite weight in both air and liquid and use Equation 7 to determine ρ_{cm} . The value of α is known from the matrix and fibre properties. Because of the insignificance of the term $W_1\rho_a$, Equation 7 reduces to

$$\rho_{cm} = \frac{W_a\rho_l}{W_a - W_1}. \quad (11)$$

The uncertainty in V_f is calculated directly [6] from

$$\Delta V_f^2 = \left(\frac{\partial V_f}{\partial \rho_{cm}} \right)^2 \Delta \rho_{cm}^2 \quad (12)$$

where ΔV_f and $\Delta \rho_{cm}$ are the uncertainties in the volume fraction and the composite density, respectively. Using Equation 10 to calculate the partial derivative, dividing both sides of Equation 12 by Equation 10, and taking the square root results in

$$\frac{\Delta V_f}{V_f} = \left(\frac{\rho_m}{\rho_{cm}} - 1 \right)^{-1} \frac{\Delta \rho_{cm}}{\rho_{cm}}. \quad (13)$$

Bowman and Schoonover [7] have achieved a precision of 0.0001% in determining the density of a 10 g silicon crystal using a mechanical balance with a 1 microgram sensitivity and water as the liquid. It is expected that under routine conditions in a metallurgical laboratory or a quality control laboratory, the weight of a specimen of MMC material can be determined to a precision of between 0.01 and 0.001%. It can be shown (see Appendix) that $\Delta \rho_{cm}/\rho_{cm}$ can be determined to a precision of 0.06 to 0.006%, respectively, using the expected precision in weighing. Assuming the more conservative value, Equation 13 becomes

$$\frac{\Delta V_f}{V_f} = 0.06 \left(\frac{\rho_m}{\rho_{cm}} - 1 \right)^{-1} \quad (14)$$

where $\Delta V_f/V_f$ is in per cent. The value of ρ_{cm} is obtained from Equation 7. The uncertainty in the volume fraction was estimated for boron/aluminium and graphite/aluminium as a function

of the volume fraction using Equation 14. The following values were used: $\rho_m = 2.7 \text{ g cm}^{-3}$, $\rho_f = 2.47 \text{ g cm}^{-3}$ (boron), and $\rho_f = 1.66 \text{ g cm}^{-3}$ (graphite), and Equation 1 with $V_g = 0$ was used to compute ρ_c for each case. The results are shown in Fig. 1 as $|\Delta V_f/V_f|$ against V_f . For some MMC systems where $\rho_f > \rho_m$ giving $\rho_{cm} > \rho_m$, the inverse term in Equation 14 will be negative.

Fig. 1 shows that the uncertainty in the volume fraction of fibre in boron/aluminium or graphite/aluminium is less than 0.5% using a standard hydrostatic weighing technique with a precision of 0.01%. This figure also shows that the uncertainty in V_f depends upon the fibre volume fraction through the term $(\rho_m/\rho_{cm} - 1)^{-1}$. This term causes the value of $|\Delta V_f/V_f|$ to decrease with increasing V_f , as shown in the figure.

3.2. Case of non-zero void fraction

The above analysis relates the void fraction and the fibre volume fraction as shown in either Equation 8 or 9. These two quantities cannot be separated with only one measurement. However, two sequential experiments can be carried out with the same specimen. The first experiment consists in determining W_a and W_1 , thereby yielding the composite density. The second experiment consists in etching away part of the specimen matrix (approximately 25 to 75%) leaving the fibres intact. The etched specimen is again weighed in air and in the liquid giving the new values W'_a

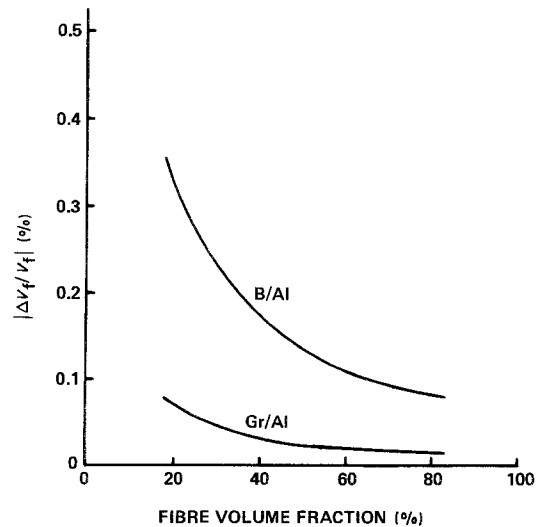


Figure 1 Uncertainty in the fibre volume fraction as a function of fibre volume fraction for boron/aluminium and graphite/aluminium calculated from Equations 14 and 1 with $V_g = 0$.

and W'_1 , respectively, from which the density ρ'_{cm} is calculated from either Equation 7 or 11. In this process, the void fraction remains essentially unchanged. For purposes of this analysis, it is assumed that etching does not change the void fraction significantly. This assumption implies that the voids are generally small and uniformly distributed throughout the matrix.

For the first measurement before etching, Equation 9 is

$$V_g = 1 - \frac{\rho_{cm}}{\rho_m} - \alpha V_f \quad (15)$$

where ρ_{cm} is defined by Equation 11 and α as for Equation 10 and V_f is the fibre volume fraction before etching. After etching, Equation 9 becomes

$$V_g = 1 - \frac{\rho'_{cm}}{\rho_m} - \alpha V'_f \quad (16)$$

where V'_f is the fibre volume fraction after etching away part of the matrix,

$$\rho'_{cm} = \frac{W'_a \rho_1}{W'_a - W'_1} \quad (17)$$

and V_g in Equations 15 and 16 is unchanged according to the above assumption.

The specimen has been etched to remove a fraction n of matrix material so that the new matrix volume fraction is

$$V'_m = n V_m \quad (18)$$

where $0 < n \leq 1$. From Equation 2, the new fibre volume fraction is

$$V'_f = 1 - V'_m - V_g = 1 - n V_m - V_g \quad (19)$$

using Equation 18. Using Equation 2 again in Equation 19 gives

$$V'_f = (1 - n)(1 - V_g) + n V_f \quad (20)$$

which relates to measured values of V_f and the unknown void fraction. Using Equation 20 in Equation 16 after some rearranging gives

$$V_f = \beta(1 - V_g) - \frac{\rho'_{cm}}{\rho_m} \quad (21)$$

where

$$\beta = n - \left(1 - \frac{1}{\alpha}\right) \quad (22)$$

Substituting Equation 15 into Equation 21 gives

$$V_f = \frac{\rho_{cm} - \rho'_{cm}}{\rho_m(1 - \beta\alpha)} \quad (23)$$

TABLE I Values of α and β for a number of metal matrix composite material systems

Composite	ρ_f (g cm ⁻³)	ρ_m (g cm ⁻³)	α	β^*	$\alpha\beta$
B/Al	2.47	2.7	0.085	3.745	0.318
Gx/Al	1.66	2.7	0.385	2.845	1.095
B/Ti	2.47	4.51	0.452	2.644	1.195
Gx/Mg	1.66	1.74	0.046	3.862	0.178
SiC/Al	3.20	2.7	-0.185	4.555	-0.843
Al ₂ O ₃ /Al	3.98	2.7	-0.474	5.422	-2.570

* β was calculated from Equation 22 using $n = 0.25$.

which yields the fibre volume fraction in the presence of an unknown void fraction from two independent sets of measurements from the same specimen. All quantities in Equation 23 are either known (ρ_f, ρ_m) or measured (ρ_{cm}, ρ'_{cm}, n). Note that the quantity n must also be known. An expression is derived below to yield that quantity from measured weights of the specimen before and after etching. Table I shows values of relevant parameters for a number of MMC systems.

It is necessary to determine the value of n with reasonable precision so that β and, hence, V_f and V_g can be determined. The weight of the specimen in air before and after etching gives the values W_a and W'_a , respectively. Their difference gives the mass of matrix lost in etching, or

$$W_a - W'_a = \rho_m \Delta v_m \quad (24)$$

where $\Delta v_m = v_m - v'_m$, the matrix volume change. From Equation 18, $v'_m = n v_m$ so that $\Delta v_m = (1 - n)v_m$. Substituting this value in Equation 24 and solving for n gives

$$n = 1 - \frac{1}{v_m \rho_m} (W_a - W'_a) \quad (25)$$

where v_m is the unknown matrix volume before etching. Now, v is the total specimen volume so that $v_m = v V_m$. To estimate V_m from measurements, Equations 2 and 10 can be combined, setting $V_g = 0$ to yield

$$V_m = 1 - \frac{1}{\alpha} \left(1 - \frac{\rho_{cm}}{\rho_m}\right) \quad (26)$$

where ρ_{cm} is the composite density before etching. The assumption $V_g = 0$ in deriving Equation 26 may not be too good. It is shown below that the uncertainty in n affects the uncertainty $\Delta V_f/V_f$ when the matrix contains voids. Substituting Equation 26 in Equation 25, recalling that

$v_m = vV_m$, gives

$$n = 1 - \frac{W_a - W_a'}{v\rho_m \left[1 - \frac{1}{\alpha} \left(1 - \frac{\rho_{cm}}{\rho_m} \right) \right]} \quad (27)$$

where all quantities are determined directly from measurements or tabulated data. The volume v is simply the product of the three specimen dimensions.

The method presented above applies to any fibre reinforced composite metal. In the case of short fibre or whisker or platelets, the fibres that were present in the etched matrix must be carefully collected and weighed in air and in the liquid with the remaining unetched composite.

3.3. Determination of void fraction with V_f known

If V_f is known from an independent measurement, then Equation 21 can be solved for V_g to yield

$$V_g = 1 - \frac{1}{\beta} \left(V_f + \frac{\rho'_{cm}}{\rho_m} \right) \quad (28)$$

where the quantities on the right-hand side of Equation 28 are obtained from two sets of independent measurements.

The void fraction can be determined by another experimental method if the fibre volume fraction is known from other measurements. Instead of etching the specimen, indentations are made into the matrix totalling a volume Δv . The specimen is weighed in air and in liquid before and after indentation to obtain the values W_a , W_1 , and W_1'' . If the specimen matrix contains voids, indentations will reduce these voids to zero at the indentations. Equation 9 can be written in terms of actual material volumes, or

$$\frac{v_g}{v} = 1 - \frac{\rho_{cm}}{\rho_m} - \alpha \frac{v_f}{v} \quad (29)$$

where v_g and v_f are the actual void and fibre volumes and ρ_{cm} is obtained from weight measurements before indentation. After indentation, the reduced volume results in a buoyancy change, thus altering the material density measurement to

$$\rho_{cm}'' = \rho_1 \frac{W_a}{W_a - W_1''} \quad (30)$$

and W_1'' is related to the volume change, Δv , by

$$W_1'' = K(v - \Delta v) \quad (31)$$

where $K = g(\rho_{cm} - \rho_1)$. Thus, Equation 29 has a

second form

$$\frac{v_g'}{v} = 1 - \frac{\rho_{cm}''}{\rho_m} - \alpha \frac{v_f}{v}. \quad (32)$$

Multiplying Equations 29 and 32 through by v and adding gives

$$v_g + v_g' = [2 - (\beta_0 + \beta_1)]v - (1 - \beta_1)\Delta v - 2\alpha v_f \quad (33)$$

where

$$\beta_0 = \frac{\rho_1}{\rho_m} \left(\frac{W_a}{W_a - W_1} \right) \quad (34)$$

$$\beta_1 = \frac{\rho_1}{\rho_m} \left(\frac{W_a}{W_a - W_1''} \right). \quad (35)$$

Since $v_g' = v_g - \Delta v_g$, after division by v , Equation 33 becomes

$$\Delta V_g = (1 - \beta_1) \frac{\Delta v}{v} - [2 - (\beta_0 + \beta_1)] + 2\alpha V_f. \quad (36)$$

The ratio $\Delta v/v$ can be found from Equation 31 to be

$$\frac{\Delta v}{v} = 1 - \frac{W_1''}{W_1} \quad (37)$$

so that Equation 36 becomes

$$\Delta V_g = A + 2\alpha V_f \quad (38)$$

where

$$A = (1 - \beta_1) \left(1 - \frac{W_1''}{W_1} \right) - [2 - (\beta_0 + \beta_1)] \quad (39)$$

where all quantities follow density from measurements. $V_g = V_g' + \Delta V_g$ so that using Equations 32 and 38, recalling that $v_g'/v = V_g'$ and $v_f/v = V_f$, there results

$$V_g = \left(1 - \frac{\rho_{cm}''}{\rho_m} \right) + A + \alpha V_f \quad (40)$$

thus giving the void fraction directly from four sets of weight measurements (W_a , W_1 ; W_a , W_1'') and an independent measurement of V_f .

Note that Equation 40 also gives the void fraction in a material containing no fibres ($V_f = 0$). Thus,

$$V_{g|V_f=0} = 1 - \frac{\rho_{cm}''}{\rho_m} + A. \quad (41)$$

When $V_f \neq 0$ and $V_g \rightarrow 0$, $W_1'' \rightarrow W_1$ since any specimen indentation causes material displacement without a volume change (densification is neglected here). Then, $\beta_1 \rightarrow \beta_0$ and Equation 38 reduces to

$$V_f = \frac{1}{\alpha}(1 - \beta_0) \quad (42)$$

which is identical to Equation 10.

The total volume of indentation does not need to be large. It can be shown [1] that this volume is related to the precision of the hydrostatic weighing by the expression $\Delta v = pv$, where p is the precision. In the method discussed in this paper, p is approximately 0.01%.

3.4. Error analysis for non-zero void fraction

Equations 15, 21, 23, and 27 can be represented by a functional relationship of the form $f = f(x_1, x_2, x_3, \dots)$, where x_1, x_2, x_3, \dots are measurement parameters or parameters that can be calculated from measurements. In estimating the errors for the values of V_f, V_m, V_g and n , cross-correlated terms are neglected and, therefore, covariant uncertainties are not considered. Consequently, the uncertainty in this functional relationship can be written as

$$\Delta f^2 = \left(\frac{\partial f}{\partial x_1}\right)^2 \Delta x_1^2 + \left(\frac{\partial f}{\partial x_2}\right)^2 \Delta x_2^2 + \left(\frac{\partial f}{\partial x_3}\right)^2 \Delta x_3^2 + \dots \quad (43)$$

Applying this expression to Equations 21 and 33 yields the following:

$$\frac{\Delta V_f^2}{V_f^2} = \left(1 + \frac{1}{V_f} \frac{\rho'_{cm}}{\rho_m}\right)^2 \left[\left(\frac{\Delta n}{n}\right)^2 + V_g \left(\frac{\Delta V_g}{V_g}\right)^2\right] + \left(1 - \frac{\beta}{V_f}\right)^2 \left(\frac{\Delta \rho'_{cm}}{\rho'_{cm}}\right)^2 \quad (44)$$

and

$$\frac{\Delta \beta^2}{\beta^2} = \left(1 + \frac{\alpha}{\beta}\right)^2 \left(\frac{\Delta n}{n}\right)^2 \quad (45)$$

Using the values shown in Table I for α and β , an average value of $\alpha/\beta \approx 0.04$ with a standard deviation of 0.1. Therefore, $(1 + \alpha/\beta)^2 \approx 1$ so that Equation 45 reduces to $\Delta \beta/\beta \approx \Delta n/n$. From Equation 15, it follows that

$$\frac{\Delta V_g^2}{V_g^2} = \frac{1}{V_g^2} (1 + \alpha V_f)^2 \left(\frac{\Delta \rho_{cm}}{\rho_{cm}}\right)^2 + \alpha^2 \left(\frac{V_f}{V_g}\right)^2 \left(\frac{\Delta V_f}{V_f}\right)^2 \quad (46)$$

and from Equation 27

$$\frac{\Delta n^2}{n^2} = 10 \left[\left(\frac{1}{1 - W'_a/W_a}\right)^2 \left(\frac{\Delta W_a}{W_a}\right)^2 + \left(\frac{1}{1 - W_a/W'_a}\right)^2 \left(\frac{\Delta W'_a}{W'_a}\right)^2 + \left(\frac{\Delta v}{v}\right)^2 + \left(\frac{1}{1 - \rho_f/\rho_m}\right)^2 \left(\frac{\Delta \rho_{cm}}{\rho_{cm}}\right)^2 \right] \quad (47)$$

Equations 46 and 47 can be combined to eliminate the uncertainty in the void fraction, resulting in

$$\frac{\Delta V_f^2}{V_f^2} = \frac{1}{1 - \alpha^2 V_f^2} \left[\left(\frac{\Delta n}{n}\right)^2 + A(1 + \alpha V_f)^2 \left(\frac{\Delta \rho_{cm}}{\rho_{cm}}\right)^2 + B \left(\frac{\Delta \rho'_{cm}}{\rho'_{cm}}\right)^2 \right] \quad (48)$$

where

$$A = \left(1 + \frac{1}{V_f} \frac{\rho'_{cm}}{\rho_m}\right)^2 \quad (49)$$

$$B = \left(1 - \frac{\beta}{V_f}\right)^2 \quad (50)$$

4. Discussion of numerical results

The uncertainties for an MMC material containing voids were calculated using the Equations of Section 3.4. The numerical parameters for boron/aluminium used in these calculations are: $V_f = 0.5$, $\rho_m = 2.7 \text{ g cm}^{-3}$ (aluminium), $\rho_f = 2.47 \text{ g cm}^{-3}$ (boron), $\rho_{cm} = 2.59 \text{ g cm}^{-3}$, $\rho'_{cm} = 2.65 \text{ g cm}^{-3}$, $W_a = 100 \text{ g}$, $W'_a = 61 \text{ g}$ (calculated for this example), $V_g \approx 0.02$, and $n = 0.25$. These values give $\beta = 3.75$ and $\alpha = 0.085$ so that $\alpha/\beta = 0.02$. Based on reported [4] measurement precision, $\Delta W_a/W_a = \Delta W'_a/W'_a = 0.0001$, $\Delta v/v = 0.001$, $\Delta \rho_{cm}/\rho_{cm} = \Delta \rho'_{cm}/\rho'_{cm} \approx 0.0006$ (see Appendix). Using these values in Equations 47, 48, and 46 yields, respectively:

$$\frac{\Delta n}{n} = 0.0225$$

$$\frac{\Delta V_f}{V_f} = 0.023$$

$$\frac{\Delta V_g}{V_g} = 0.058.$$

These results show that the uncertainty in the fibre volume fraction is approximately 2% for this case compared to an uncertainty less than 0.5% for the case of zero void. The dominant uncertainty in $\Delta n/n$ is the fourth term in Equation 47

so that, for practical purposes, Equation 47 reduces to

$$\frac{\Delta n}{n} \approx \frac{10}{1 - \rho_f/\rho_m} \left(\frac{\Delta \rho_{cm}}{\rho_{cm}} \right). \quad (51)$$

In Equation 48, the term in front of the bracket is of order unity. The uncertainty is dominated by the first and third term in the bracket so that Equation 48 can be simplified to

$$\frac{\Delta V_f^2}{V_f^2} \approx \left(\frac{\Delta n}{n} \right)^2 + \left(1 - \frac{\beta}{V_f} \right)^2 \left(\frac{\Delta \rho'_{cm}}{\rho'_{cm}} \right)^2. \quad (52)$$

The uncertainty in Equation 46 cannot be simplified as both terms are of the same order.

It can also be seen from the results of the numerical evaluation that $\Delta n/n$ and $\Delta V_f/V_f$ are the same magnitude. This is illustrated in Fig. 2. The uncertainties in $\Delta V_f/V_f$, $\Delta n/n$, and $\Delta V_g/V_g$ are plotted as a function of the precision of weight measurements. The differences between $\Delta n/n$ and $\Delta V_f/V_f$ are negligibly small. The uncertainty in the void fraction measurement is a factor of about 2.5 greater than for the fibre volume fraction. From Fig. 2, it can be noted that to achieve a 1% uncertainty in V_f and V_g , the balance must have a precision of 0.004 and 0.0015%, respectively.

5. Conclusions

A method for determining the fibre volume fraction, V_f , and the void fraction, V_g , in an MMC material has been described. These quantities are determined from specimen weight measure-

ments in air and in a liquid using a laboratory balance. For a material without voids, one weight measurement in air and in a liquid is sufficient to determine V_f . With a balance having a precision of 0.01%, V_f can be determined with an uncertainty of 0.5% or less. This is somewhat better than the 1 to 2% achievable with an image analysing system.

In the presence of voids, V_f and V_g can be determined by two sets of weight measurements as before: one set of measurements on the intact specimen, the second set after a fraction (0.25 to 0.75) of the matrix has been etched away. With a balance having a precision of 0.01%, V_f and V_g can be determined with an uncertainty of less than 3 and 6%, respectively. The uncertainties in V_f and V_g are nearly linearly related to balance precision, rising steeply with decreased precision.

It has also been shown that the method was theoretically feasible for determining the void fraction in materials not containing fibres. Two sets of weight measurements would be needed, one with the specimen intact, the other after indentations have been made in the specimen surface. However, in this case, the balance precision required would have to be greater than 0.01%. The feasibility of this method has not been investigated.

Acknowledgements

The author gratefully acknowledges the unfailing

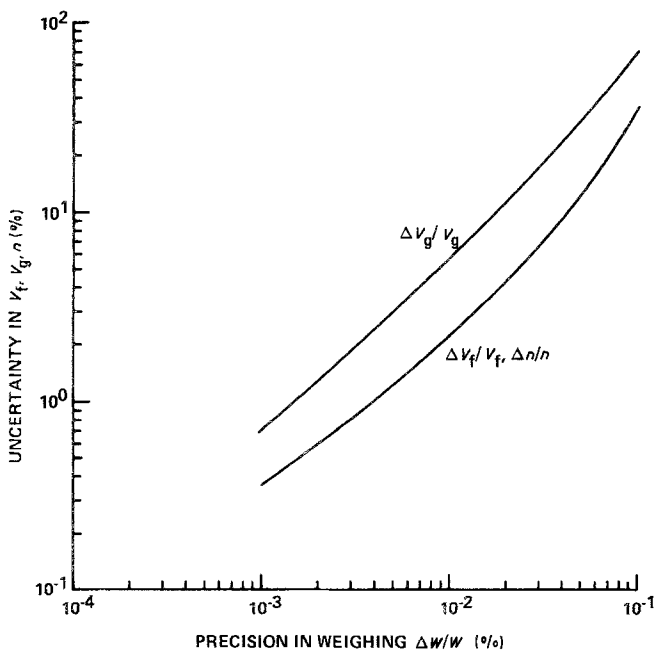


Figure 2 Uncertainties in V_f , V_g , and n as a function of precision in weighing (calculated from Equations 46, 47, and 48).

support of Mr Louis Gonzalez, Manager, MMCIAC. He is also indebted to Dr Carl Zweben of the General Electric Company for his constructive comments.

Appendix

The uncertainties in the material density obtained from weight measurements are related to weight measurement uncertainties in the following manner. Equation 17 is

$$\rho_{\text{cm}} = \frac{W_a \rho_1}{W_a - W_1} \quad (\text{A1})$$

and the uncertainty in the density, neglecting cross-correlation terms, is obtained from

$$\Delta \rho_{\text{cm}}^2 = \left(\frac{\partial \rho_{\text{cm}}}{\partial W_a} \right)^2 \Delta W_a^2 + \left(\frac{\partial \rho_{\text{cm}}}{\partial W_1} \right)^2 \Delta W_1^2. \quad (\text{A2})$$

Carrying out the partial derivatives using Equation A1, substituting the result into Equation A2, and dividing through by ρ_{cm}^2 after some rearrangement gives

$$\frac{\Delta \rho_{\text{cm}}^2}{\rho_{\text{cm}}^2} = \frac{W_1^2}{(W_a - W_1)^2} \left[\left(\frac{\Delta W_a}{W_a} \right)^2 + \left(\frac{\Delta W_1}{W_1} \right)^2 \right]. \quad (\text{A3})$$

If the weight in air and in water can be measured to the same uncertainty, then we can write $\Delta W_a/W_a = \Delta W_1/W_1 = \Delta W/W$, where W is the specimen measured weight. Then Equation A3 becomes

$$\frac{\Delta \rho_{\text{cm}}^2}{\rho_{\text{cm}}^2} = \frac{2W_1^2}{(W_a - W_1)^2} \left(\frac{\Delta W}{W} \right)^2. \quad (\text{A4})$$

In general, $W_1 \approx 0.8W_a$ is a good approximation so that $2W_1^2/(W_a - W_1)^2 \approx 32$. Consequently, Equation A4 further reduces to

$$\frac{\Delta \rho_{\text{cm}}}{\rho_{\text{cm}}} = 5.66 \frac{\Delta W}{W}. \quad (\text{A5})$$

Therefore, for an uncertainty in the weight of 0.01%, the uncertainty in the density is 0.06%.

References

1. J. E. SCHOUTENS, unpublished data.
2. E. D. KARSTEN, Martin Marietta Corporation, Denver Division, Denver, Colorado, Personal Communication, January (1982).
3. J. D. WHITTENBERGER, F. I. HURWITZ, J. J. RICCA and R. M. JURTA, *J. Mater. Sci. Lett.* 1 (1982) 249.
4. N. A. PRATTEN, *J. Mater. Sci.* 16 (1981) 1737.
5. S. W. TSAI and H. T. HAHN, "Introduction to Composite Materials" (Technomic Publishing Company, Westport, Connecticut, 1980) p. 381.
6. P. R. BEVINGTON, "Data Reduction and Error Analysis for the Physical Sciences" (McGraw-Hill Book Company, New York, 1969) p. 56.
7. H. A. BOWMAN and R. M. SCHOONOVER, *J. Res. Nat. Bur. Stand. (C)* 71C (1967) 179.

Received 18 April
and accepted 21 July 1983