STRUCTURAL METALLIC MATERIALS BY INFILTRATION

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Standard Form 298 (Rev. 8-98)  
Prescribed by ANSI Std Z39-18
Infiltration
The Infiltration Process

Fluid Matrix

Reinforcement Preform
The Infiltration Process

General Characteristics for metals:
- high capillary forces

<table>
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<th>Material</th>
<th>Temperature (°C)</th>
<th>Surface Tension (N/m)</th>
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<tr>
<td>Polyethylene (PE)</td>
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<td>Polyethylene oxide (PEO)</td>
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<tr>
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<td>PE I</td>
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<td>-</td>
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<td>0.03 to 0.04</td>
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<tr>
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<td>Na₂SiO₃</td>
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<tr>
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<tr>
<td>CaSiO₃</td>
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<tr>
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<tr>
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<tr>
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<td>970</td>
<td>0.92</td>
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<tr>
<td>Au</td>
<td>1070</td>
<td>1.13</td>
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The Infiltration Process

General Characteristics for metals:
- high capillary forces
- low viscosity
The Infiltration Process: Spontaneous Infiltration

Place preform and metal in a furnace. Infiltration proceeds. Composite is solidified. The infiltrated composite.
The Infiltration Process: *Squeeze Casting*

1. **Preform preheating and placement**
2. **Metal pouring**
3. **Ram movement initiation**
4. **Infiltration**
5. **Solidification**

The infiltrated selectively reinforced cast composite component
The Infiltration Process: Pressure Infiltration

(a) (b)
The infiltration Process

IN GENERAL

• Net-shape, rapid.

• Produces defect-free material if well engineered...

• ...with considerable flexibility in the material choice if pressure is used to drive the metal.

• Hence, well suited for the production of model multiphase materials.
50% ceramic in 50% metal
A few good reasons to add ceramic to a metal or an alloy
A few good reasons to add ceramic to a metal or an alloy

• Increase wear and abrasion resistance;

• Increase the specific elastic modulus \( (E/\rho) \) above 26 MJ·kg\(^{-1}\);

• Tailor certain physical properties: thermal conductivity, coefficient of thermal expansion, ...

• Increase the tensile strength (with ceramic fibers)
A few good reasons NOT to add ceramic to a metal or an alloy

• Lower ductility;

• Lower toughness;

(...frequently with consequences on strength.)

The volume fraction ceramic $V_f$ is therefore generally kept below 25-30% in structural particle reinforced metals.
Why a high volume fraction ceramic might be desirable
Why a high volume fraction ceramic might be desirable

- The incremental benefit increases with the fraction ceramic;
According to Christensen’s 3-phase self-consistent model ($E = 70$ GPa and $\nu = 0.345$ for Al, and $E = 390$ GPa and $\nu = 0.22$ for Al$_2$O$_3$).
Why a high volume fraction ceramic might be desirable

• The incremental benefit increases with the fraction ceramic;

• Particle clustering.
Influence of Particle Clustering:

...a somewhat extreme example, but a real one.

*Gravity cast Al-356 / SiCp*
Particle Reinforced Aluminium by Infiltration
Particle Reinforced Aluminium by Infiltration

Ceramic particles and a cast metal ingot are packed, in that order, into an alumina crucible.
Particle Reinforced Aluminium by Infiltration

Pressure infiltration then combines the two into an ingot of composite
Three Matrices

• 99.99% pure Al

• Al-2wt.% Cu
  (as-cast, T4 and T6)

• Al-4.5wt.% Cu
  (as-cast, T4 and T6)
Three Reinforcement Types

Angular $\text{Al}_2\text{O}_3$  
Polygonal $\text{Al}_2\text{O}_3$  
Angular $\text{B}_4\text{C}$
Infiltrated Particle Reinforced Aluminium

Angular $\text{Al}_2\text{O}_3$

Polygonal $\text{Al}_2\text{O}_3$

$\text{B}_4\text{C}$
Tensile Behaviour
Tensile Behaviour

Effect of reinforcement chemistry

Boron carbide

Angular alumina

Pure Al matrix

σ (MPa)

ε
Effect of reinforcement chemistry (for equal volume fractions)

Tensile Behaviour

Effect of particle size

Pure Al/angular alumina)
Comparing $\text{Al}_2\text{O}_3$ with $\text{B}_4\text{C}$:
- $\Delta \text{CTE}$ is 1.3 times higher for $\text{B}_4\text{C}$;
- the experimental slope is 1.25 times higher.

$\left(\frac{\sigma_m}{\alpha \mu b}\right)^2$ vs $\frac{1}{\lambda}$

The Size Effect
Tensile Behaviour

Effect of reinforcement shape and quality

Pure Al matrix; 10µm Al₂O₃
Tensile behaviour

Illustrating the influence of particle type

Al-Cu 4.5% wt. alloy reinforced by about 50% Al$_2$O$_3$ particles

![Graph showing tensile behavior with stress vs. strain for Al-Cu alloy with different particle types]
Damage
Damage

1 - Particle fracture followed by void nucleation in the matrix at particle cracks
Damage

2 - Matrix voiding at sites of high stress triaxiality
Damage

Measurement:
- Young’s modulus evolution with strain
- Derived damage parameter: $D_E = 1 - \frac{E}{E_0}$
Damage

Measuring the rate of damage accumulation
Link between Damage and Tensile Ductility

\[ \varepsilon_f = \frac{n}{d \ln(D_E)} \left( 1 - \frac{d \ln(D_E)}{d\varepsilon} \right) \]
Fracture Toughness
Toughness

• $J_R$ method for pure Al composites using precracked CT specimens (ASTM E-1737);
• Unloading compliance method used to monitor crack growth

Partial unloading used to measure specimen compliance and compute crack extension
Toughness

- Construction line
- Region of qualified data
- 1.5 mm exclusion line
- 0.2 mm offset line
- $J_{GT}$
- $J_{0.2mm}$

Crack extension $\Delta a$
$J_{GT}$ corresponds to the onset of marked crack advance

(pure Al/25 μm $\text{Al}_2\text{O}_3$ polyg. composites)

Crack front marked by fatigue, specimen #2

Crack front marked by fatigue, specimen #3
Toughness

Polygonal Al$_2$O$_3$ particles/pure Al: influence of particle size
Toughness

$B_4C$ particles/pure Al: influence of particle size

![Graph showing the influence of particle size on toughness](image)
Toughness

Equal size: influence of reinforcement nature and quality
Toughness

Alloyed matrix composites were characterized in small-scale yielding using chevron-notched specimens (ASTM E-1304)

Consistency: $J$-integral test data for Al-Cu matrix composites are between 2 and 27% lower than chevron-notched test data.
Toughness

Solution-treated condition

Particle size [μm]

$K_{Iv}$ [MPa m$^{1/2}$]

Al-Cu 2% / angular Al$_2$O$_3$

Al-Cu 4.5% / angular Al$_2$O$_3$

Al-Cu 2% / polygonal Al$_2$O$_3$

Al-Cu 4.5% / polygonal Al$_2$O$_3$
Strength/Toughness Combination
Overall summary of data:

Strength/Toughness Combination

- Pure Al matrix
- Al-Cu2% matrix (T6)
- Al-Cu4.5% matrix (T6)

Graph showing:
- $K_{IV}$ or $K_{eq-GT}$ [MPa m$^{1/2}$]
- UTS [MPa]

Data points:
- 25 μm, polyg.
- 15 μm, polyg.
- 35 μm, ang.
- 5 μm, ang.
Strength/Toughness Combination

- Pure Al matrix
- Al-Cu2% matrix
- Al-Cu4.5% matrix

UTS [MPa]

$K_{lc}$, $K_{Jeq}$ [MPa m$^{1/2}$]

Materials:
- 25 µm polygonal Al$_2$O$_3$
- 15 µm polygonal Al$_2$O$_3$
- 35 µm angular Al$_2$O$_3$
- 5 µm angular Al$_2$O$_3$
- Pure Al matrix
- Al-Cu2% matrix
- Al-Cu4.5% matrix

Types:
- 7175-T73
- 7075-T73
- 2024-T8
- 2124-T8
- 7075-T73
- 2024-T8

Graphical representation showing the relationship between strength (UTS) and toughness ($K_{lc}$, $K_{Jeq}$) for various aluminum materials and their combinations.
Toughening mechanisms
Toughening mechanisms

What makes these composites tough?

• A first very simple mechanism: $K \propto \sqrt{(G.E)}$
  and $E$ is 2.5 times higher than for Al alloys.

• Still, corresponding $G/(J)$ values near 10 kJ/m$^2$ are high.

• There is significant $R$-curve behaviour: these $K$ values are for near-steady crack advance.
Fracture micromechanisms
Fracture micromechanisms

Particle fracture

Pure Al/ 30 µm angular Al₂O₃
Fracture micromechanisms

Particle fracture

Pure Al/ 30 µm angular Al₂O₃
Matrix void growth

Fracture micromechanisms

Pure Al/10 μm polygonal $\text{Al}_2\text{O}_3$
Fracture micromechanisms

- Voids nucleate between particles
- Final void size scales with average particle size
Local fracture energy estimation
Local fracture energy estimation

Pure Al composites: 3-D fracture surface topography measurement
Local vs. total fracture energy

Global fracture energy, $J_{GT}$ [kJ/m²]

Local fracture energy, $2\gamma_{pz}$ [kJ/m²]

Pure Al matrix composites
Toughening mechanisms

Observation of crack tip plasticity using a photoelastic coating:

\[ \varepsilon_1 - \varepsilon_2 \approx 0.2\%: \]

pale yellow - orange fringes
Toughening mechanisms

(Al/35 µm ang. Al₂O₃)
In other words, the total fracture energy:
\[ J = 2\gamma_{pz} + W_p \gg 2\gamma_{pz} \]

- \( 2\gamma_{pz} \) is the local « process zone » or « cohesive law » fracture energy;

- \( W_p \) is the energy dissipated in the surrounding macroscopic plastic zone
Toughening mechanisms

Tvergaard and Hutchinson  (JMP$ vol. 40 (1992) 1377)
Cohesive Zone Model :

\[ \Gamma_0 = \int_0^{\delta_c} \sigma d\delta \]

Fig. 1. Traction–separation relation for fracture process.
Toughening mechanisms

Tvergaard and Hutchinson (JMP volume 40 (1992) 1377):

\( \Gamma_{ss} \): steady-state toughness
\( \Gamma_0 \): local fracture energy \((2\gamma_{pz})\)
\( \sigma_y \): composite yield strength
\( \tilde{\sigma} \): peak-stress of the cohesive law
\( N \): strain-hardening coefficient

\( \frac{\Gamma_{ss}}{\Gamma_0} \) vs. \( \frac{\tilde{\sigma}}{\sigma_y} \)
Toughening mechanisms


Fig. 7a,b. Meshes at two stages of deformation for \( \sigma_y/E = 0.003, n = 10, H_0/B_0 = 0.25, R_0/B_0 = 0.01 \). a Initial mesh; b \( \varepsilon_1 = 0.522 \) and \( V/V_0 = 2.50 \cdot 10^5 \).

Fig. 8. Average true stress and void volume growth vs. average logarithmic strain, for \( H_0/B_0 = 1 \) and \( R_0/B_0 = 0.01 \). With remeshing.
Metal sponge
The replication process

- Molten metal
- Open-cell pattern
- Gas pressure
- Composite
- Open-cell foam
- Pattern removal
- Machining
The replication process

Cold Isostatic Pressing (CIP) + sintering for 40 µm (32-45 µm) powder: 45 min. at 750°C.
The replication process

1. **Al ingot**
2. **Al₂O₃ crucible**
3. **NaCl preform**
4. **Al – NaCl composite**

Diagram components:
- Thermocouple
- Pressure-Vacuum
- Cooling coils
- Molten metal
- Preform
- Furnace
- Insulation
- Pressure vessel
- Chill
The replication process

Machining:
conducted prior to salt removal by dissolution on the (brittle) NaCl-Al composite;

Dissolution:
- in distilled water.
- below 50 µm, degassed water with forming gas (H₂ + N₂) bubbling (to minimize corrosion problems)
Commercial NaCl powder, sieved to:

- 32-45 µm (40 µm);
- 63-90 µm (75 µm);
- >250 µm (ave. 400µm).
Replicated Foams

$\text{NaCl \ 400 \ \mu m ,} \ \ V_f \ \text{Al} = 16 \ %$
Replicated Foams

75 µm, Vf Al = 16 % (fracture surface)
Replicated Foams

NaCl 20-32 µm, Vf Al = 18% (fracture surface)
Mechanical Properties
Mechanical Properties

Compression; microcellular AA1199, 400 µm NaCl

Engineering Compressive Stress (MPa) vs. Engineering Compressive Strain

Compression curves for microcellular AA1199 with 400 µm NaCl, showing stress-strain behavior at different densities: 0.670 g·cm⁻³, 0.672 g·cm⁻³, and 0.680 g·cm⁻³.
Influence of Density

Compression; microcellular AA1199, 400 µm NaCl
Influence of Density

Tension; microcellular AA1199, 400 µm NaCl

Engineering strain [%]

Engineering stress [MPa]

- Vf = 30%
- Vf = 21%
- Vf = 13%
Influence of Density

Evolution of $E_0$ with $Vf_{Al}$, 400 $\mu$m NaCl

Compression:

$E = 33 Vf^2$

Influence of Density

Evolution of $\sigma_{2\%}$ with $Vf_{Al}$, 400 $\mu$m NaCl

Size Effect

$\Phi_{\text{NaCl}} = 40\mu m$

$\Phi_{\text{NaCl}} = 75\mu m$

$\Phi_{\text{NaCl}} = 400\mu m$

$\Phi_{\text{NaCl}} = \text{Average NaCl grain size in the preform}$

$V_{\text{Al}} = 30\%$

Engineering stress [MPa] vs. Engineering strain [-]
Sources of hardening at small cell sizes:

• Geometrically necessary dislocations when cooling after infiltration
  $\text{CTE}_{\text{Al}} = 23.6 \cdot 10^{-6} \ [\text{K}^{-1}]$
  $\text{CTE}_{\text{NaCl}} = 44 \cdot 10^{-6} \ [\text{K}^{-1}]$

• Oxidation during salt dissolution (hydroxide formation)
Damage

Al foam 16%, made with NaCl 63-90 µm
Damage

Before necking, $E$ decreases with $e$ while $R$ increases linearly with $e$.

This implies **damage build-up** during foam tensile deformation:
(the modulus would otherwise increase),

taking the form of **foam strut tensile deformation and failure**
(since the resistance increases linearly with strain before the peak).
Damage

Visualisation by X-Ray Microtomography:

At ESRF, in collaboration with:

- Ariane Marmottant, Luc Salvo, Rémy Dendiével
  (INPG Grenoble, France)

- Eric Maire
  (INSA Lyon, France)
Tensile test coupled with X-ray Microtomography

466_3

Stress axis (Z)

Salt: 400 µm
Vf preform = 75 %
Pinfiltration = 155 bars
Tensile test coupled with X-ray Microtomography

466_4

Stress axis (Z)

Salt: 400 µm
Vf preform = 75 %
Pinfiltration = 155 bars
Tensile test

467_0

Salt: > 250 µm
Vf preform = 75 %
Pinfiltration = 1 bar
Tensile test

467_1

Stress axis

Salt: > 250 µm
Vf preform = 75 %
Pinfiltration = 1 bar
Tensile test

467_2

Stress axis

Salt: > 250 µm
Vf preform = 75 %
Pinfiltration = 1 bar
Tensile test
467_3

Stress axis

Salt: > 250 µm
Vf preform = 75 %
Pinfiltration = 1 bar
Tensile test

467_0

Salt: > 250 µm
Vf preform = 75 %, Pinfiltration = 1 bar
Tensile test

Salt: > 250 µm
Vf preform = 75 %, Pinfiltration = 1 bar
Tensile test
467_2

Salt: > 250 µm
Vf preform = 75 %, Pinfiltration = 1 bar
Tensile test

Salt: > 250 µm
Vf preform = 75 %, Pinfiltration = 1 bar
Damage as seen in the SEM

Far from fracture zone

Fracture surface

NaCl 75µm, VfAl = 31% NaCl 75µm, VfAl = 28% NaCl 400µm, VfAl = 25%

NaCl 75µm, VfAl = 28%
Microstructural tailoring
Microstructural tailoring

Influence of NaCl Sintering:
T sintering = 755 °C; V_f = 66%; particle size: 63-90 µm

$t = 0$ [h]

$t = 2$ [h]

$t = 9$ [h]

$t = 25$ [h]
Microstructural tailoring

Influence of NaCl sintering

NaCl 63-90 µm, no sintering
Vf Al = 18%

NaCl 63-90 µm, sintered 24h@750°C
Vf Al = 18%
Microstructural tailoring

Commercial powders

Precipitated powders

Sieving 63 - 90 µm

Sieving > 250 µm

(a few µm in diameter)
Microstructural tailoring

Influence of Infiltration Pressure (preform 75% dense)
Conclusion

Infiltration: definition, engineering advantages, usefulness in research;

High $V_f$ ceramic particle reinforced metal: can be made relatively tough, strong and ductile.

Open-cell aluminium foams (sponges): exploration of processing/microstructure/property relations for this class of materials.
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