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Exploring the Fusion of Intelligence with Materials


Department of Mechanical Engineering and Materials Science
DYNAMIC FRACTURE TOUGHNESS of TaC/CNTs/SiC CMCs PREPARED by SPARK PLASMA SINTERING

Qiaoyun Xie

Advisor: Dr. Sylvanusa N. Wosu
Motivation

TaC/CNTs/SiC

material for aerospace & ballistic armors

oxidation

impact

facture

TaC

- High elastic Modulus,
- High strength

CNTs
Work Focus

3/4-point static ASTM → dynamic (SHPB, drop weight tower, Charpy tester)

- Geary et al., dynamic fracture toughness for glass reinforced polymer
- Samborski, fracture toughness for alumina and magnesia ceramic
- Rubio-Gonzalez, dynamic fracture toughness for composite material by Hopkinson bar

NO STANDARD MEASUREMENT

- Vickers indentation
  - static fracture toughness

- SEM
  - crack propagation

- SHPB
  - dynamic fracture toughness
# Powder Property

<table>
<thead>
<tr>
<th>material</th>
<th>density (g/cm³)</th>
<th>average size</th>
<th>purity, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>3.216</td>
<td>800 nm</td>
<td>&gt;99</td>
</tr>
<tr>
<td>TaC</td>
<td>13.9</td>
<td>1000 nm</td>
<td>&gt;99</td>
</tr>
<tr>
<td>MWCNTs</td>
<td>2.1</td>
<td>D₀&lt;20 nm, D₁: 4 nm, L: 1-12 μm</td>
<td>&gt;99 wt</td>
</tr>
<tr>
<td>B₄C</td>
<td>2.51</td>
<td>45-55 nm</td>
<td>&gt;99</td>
</tr>
</tbody>
</table>
Powder Mixture

Mixing Process:

CNTs in mixed powder

[Images showing CNTs at different magnifications]

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Spark Plasma Sintering (SPS) System

Stage 1:
\[ \dot{Q} = 133°C/min, \text{ 9min} \]
\[ T = 1200°C \]
Hold 3min
\[ P = 30 \text{ MPa} \]

Stage 2:
\[ P = 90 \text{ MPa} \]
\[ T = 1800 °C \]
Hold 10 min

Densification 98.4%
Sample Fabrication

SiC 91 wt%

Sintering Aids

B₄C 1 wt%

ultra-sonication

CNTs/TaC /SiC Powder Mixtures

Compacting

Sintering

CNTs/TaC /SiC Material
Vickers Indentation (static)

\[ HV = \frac{F}{A} \approx \frac{1.854F}{d^2} \]

- \( HV \): Vickers hardness
- \( F \): loading force
- \( A \): indentation area
- \( d \): average length of the diagonal left by the indenter

\[ K_{IC} = 0.016 \frac{E}{HV} \frac{F}{c^{3/2}} \]

- \( K_{IC} \): mode I fracture toughness
- \( E \): Young’s modulus
- \( c \): crack length from the impression center
Sample Requirement

**ASTM C1421-10 three-point bending test**

- **B** = 3 mm
- **W** = 4 mm
- **t** ≤ 0.1 mm
- **a** = 2 mm
- **S** = 20 mm
- **S₀** = 16 mm
- **3*D** = 4.5 mm

\[ 0.12 \leq \frac{a}{W} \leq 0.30 \quad \quad 4 \leq \frac{S₀}{W} \leq 10 \]

- **S₀**: three-point test fixture outer span
- **B**: side to side dimension of the test specimen
- **W**: top to bottom dimension of the test specimen parallel to the crack length
- **a/W**: normalized crack size
SHPB Set-up (dynamic)

To obtain a constant loading rate

copper pulse shaper 3.2 mm diameter × 3.2 mm thickness
strain rate, energy absorption and force measurement

\[ \dot{\varepsilon}_s(t) \approx \frac{-2c}{L_s} \varepsilon_r(t) \]

\[ F(t) = A_b E (\varepsilon_i(t) + \varepsilon_r(t)) \]

\[ E_A(t) = \frac{A_b c}{E} \int_0^t (\sigma_i^2(t) - \sigma_r^2(t) - \sigma_t^2(t)) \, dt \]

- \( \dot{\varepsilon}_s \): strain rate
- \( c \): wave velocity
- \( L_s \): specimen thickness
- \( E_A \): energy absorption
- \( A_b \): cross sectional areas of bars

- \( E \): Young’s modulus of the bars
- \( \sigma_i, \sigma_r, \sigma_t \): incident, reflected, transmitted stress
- \( F \): force at the incident bar/specimen interface
- \( \varepsilon_i, \varepsilon_r \): incident, reflected strain
Dynamic Fracture Toughness

quasi-static method expression for dynamic calculation

\[ K_{IC} = f(a/W) \left( \frac{P_{max} S_0 10^{-6}}{BW^{3/2}} \right) \left[ \frac{3(a/W)^{1/2}}{2(1 - a/W)^{3/2}} \right] \]

\[ f(a/W) = \frac{1.99 - (a/W)(1 - a/W)[2.15 - 3.93(a/W) + 2.7(a/W)^2]}{1 + 2(a/W)} \]

- \( P_{max} \): maximal dynamic force
- \( S_0 \): the three-point test fixture outer span
- \( B \): side to side dimension of the test specimen
Sample Response

TaC/CNTs/SiC CMCs

three-point dynamic fracture test:

770 mJ impact energy
Strain Wave

- Constant slope
- Constant loading rate
- Small amplitude
- Mechanical impedance mismatch
Dynamic Loading

$E_{cri}$

dynamic equilibrium

$P_{max}$

fracture initiation

loading history

the stress intensity factor history

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Strain Rate Effect

\[ \dot{\varepsilon} \uparrow, E_{\text{max}} \uparrow \]

\[ \dot{\varepsilon} \uparrow, K_{IC} \uparrow \]

\[ \dot{\varepsilon} \uparrow, P_{\text{max}} \uparrow \]
# Fracture Toughness

<table>
<thead>
<tr>
<th>Impact Energy ($E_i$, mJ)</th>
<th>Vickers Hardness ($HV$, GPa)</th>
<th>Strain Rate ($\dot{\varepsilon}$, 1/s)</th>
<th>Max Energy Absorbed ($\Delta U_A$, mJ)</th>
<th>Max Loading Force ($P_{\text{max}}$, N)</th>
<th>Fracture Toughness ($K_{IC}$, Mpa $\cdot$ m$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Static</td>
<td>24.55 ± 1.32</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>3.88 ± 0.28</td>
</tr>
<tr>
<td>dynamic</td>
<td>445</td>
<td>/</td>
<td>51.0</td>
<td>49.2</td>
<td>85.4</td>
</tr>
<tr>
<td></td>
<td>790</td>
<td>/</td>
<td>69.8</td>
<td>63.5</td>
<td>97.7</td>
</tr>
<tr>
<td></td>
<td>1235</td>
<td>/</td>
<td>90.4</td>
<td>86.1</td>
<td>149.8</td>
</tr>
</tbody>
</table>

Different failure modes

Static: $24.55 ± 1.32$ GPa, $3.88 ± 0.28$ Mpa $\cdot$ m$^{1/2}$

Dynamic: $445$, $790$, $1235$ N, $4.71 ± 0.17$, $5.45 ± 0.14$, $8.36 ± 0.09$ Mpa $\cdot$ m$^{1/2}$

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Toughening Mechanisms

- a. Fiber pullout
- b. Interfacial debonding
- c. Crack bridging
- d. Crack deflection

Surface flaw relax stress field

Crack slows down

Main radial cracks

Relieve residual stress
decrease crack opening

Secondary radial cracks

50 µm
Toughening Mechanisms

SEM image

- Crack deflection
- Crack bridging

Interaction between cracks and CNTs

- Interface has a lower toughness
- Perpendicular cracks reach interface and propagate along the interface
- Perpendicular to axial direction of CNTs

- Restrain crack opening
- Reduce driving force for crack propagation

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Conclusions

- TaC/CNTs/SiC CMCs were prepared by spark plasma sintering

- Dynamic fracture toughness by SHPB w/ copper pulse shaper was investigated based on the static ASTM C1421-10

- $K_{ICd} \ (4.71-8.36 \ \text{MPa}\cdot\text{m}^{1/2}) > K_{IC} \ (3.88 \ \text{MPa}\cdot\text{m}^{1/2})$

- SiC CMCs exhibited a more strain rate dependent property for higher strain rate

- Toughening mechanisms (CNTs deflection, CNTs bridging): restrained the crack to open and to grow further under the loading
Thank You!

Liaoyun Xie

Department of Mechanical Engineering and Materials Science