DYNAMIC FRACTURE TOUGHNESS of TaC/CNTs/SiC CMCs PREPARED by SPARK PLASMA SINTERING

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Motivation

TaC/CNTs/SiC

Material for aerospace & ballistic armors

Oxidation

Impact

Fracture

TaC

- High elastic Modulus,
- High strength

CNTs
Work Focus

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

- Geary et al., dynamic fracture toughness for glass reinforced polymer
- Samborski, facture 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

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## Powder Property

| material   | density (g/cm³) | average size          | purity, % |
|------------|----------------|-----------------------|-----------|
| SiC        | 3.216          | 800 nm                | >99       |
| TaC        | 13.9           | 1000 nm               | >99       |
| MWCNTs     | 2.1            | Dₒ<20 nm, Dᵢ: 4 nm, L: 1-12 μm | >99 wt   |
| B₄C        | 2.51           | 45-55 nm              | >99       |
Powder Mixture

Mixing Process:

CNTs in mixed powder

Higher magnification
Stage 1:
\( \dot{Q} = 133^\circ C/min, 9\text{min} \)

\[ T = 1200^\circ C \]

Hold 3min

\[ P = 30 \text{ MPa} \]

Stage 2:

\[ P = 90 \text{ MPa} \]

\[ T = 1800 \text{ °C} \]

Hold 10 min

Densification 98.4%
Sample Fabrication

SiC 91 wt%

Sintering Aids
B₄C 1 wt%

ultrasonication

CNTs 4 wt%

TaC 4 wt%

Compacting

CNTs/TaC/SiC Powder Mixtures

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

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

*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

\[ 0.12 \leq \frac{a}{W} \leq 0.30 \quad 4 \leq \frac{S_0}{W} \leq 10 \]

- \( S_0 \): 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
To obtain a constant loading rate

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

\[ \varepsilon_s(t) \approx \frac{-2c}{L_s} \varepsilon_r(t) \]

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

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

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

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

\[ P_{max}: \text{ maximal dynamic force} \]

\[ S_0: \text{ the three-point test fixture outer span} \]

\[ B: \text{ side to side dimension of the test specimen} \]
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

- Energy Absorbed (mJ) vs. Time (μs)
  - $E_{cri}$
- Loading Force (N) vs. Time (μs)
  - $P_{max}$ fracture initiation
  - Loading history
  - The stress intensity factor history
Strain Rate Effect

- $\dot{\varepsilon} \uparrow, E_{max} \uparrow$
- $\dot{\varepsilon} \uparrow, K_{IC} \uparrow$
- $\dot{\varepsilon} \uparrow, P_{max} \uparrow$
## Fracture Toughness

| Impact Energy ($E_i$, mJ) | Vickers Hardness ($HV$, GPa) | Strain Rate ($\dot{\varepsilon}$, 1/s) | Max Energy Absorbed ($\Delta U_A$, mJ) | Max Loading Force ($P_{max}$, N) | Fracture Toughness ($K_{IC}$, Mpa • m$^{1/2}$) |
|--------------------------|-----------------------------|----------------------------------------|----------------------------------------|-------------------------------|---------------------------------|
| Static                   | 24.55 ± 1.32                | /                                      | /                                      | /                             | 3.88 ± 0.28                     |
| dynamic                  |                             |                                        |                                        |                               |                                 |
| 445                      | /                           | 51.0                                   | 49.2                                   | 85.4                          | 4.71 ± 0.17                     |
| 790                      | /                           | 69.8                                   | 63.5                                   | 97.7                          | 5.45 ± 0.14                     |
| 1235                     | /                           | 90.4                                   | 86.1                                   | 149.8                         | 8.36 ± 0.09                     |

*Note: Different failure modes.*
Toughening Mechanisms

(a) Fiber pullout
(b) Interfacial debonding
(c) Crack bridging
(d) Crack deflection

Surface flaw relax stress field

Main radial cracks

Crack slows down

Secondary radial cracks

50 μm

relieve residual stress
decrease crack opening
Toughening Mechanisms

SEM image

- Interaction between cracks and CNTs
- Interface has a lower toughness
- Perpendicular cracks reach the interface and propagate along the interface
- Perpendicular to axial direction of CNTs
- Restrain crack opening
- Reduce driving force for crack propagation
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!

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