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Zusammenfassung

Gefriertrocknen von keramischen Suspensionen ist ein innovatives Formgebungsverfahren zur Herstellung von porösen keramischen Materialien. Im Rahmen eines breit angelegten Verbundvorhabens zwischen mehreren Forschungseinrichtungen werden neuartige Metall/Keramik-Verbundwerkstoffe entwickelt, die durch Schmelzinfiltration solcher keramischer Formkörper erzeugt werden. Diese Verbundwerkstoffe weisen eine charakteristische hierarchische Struktur auf. Auf mesoskopischer Ebene liegen lamellare Domänen mit Größen von bis zu mehreren Millimetern vor. Die einzelnen Domänen setzen sich abwechselnd aus keramischen und metallischen Lamellen mit einer Dicke zwischen 20 und 100 µm zusammen.

Das Ziel dieser Arbeit war die experimentelle Charakterisierung der mechanischen Eigenschaften solcher Aluminiumoxid-Aluminium-Verbundwerkstoffe auf unterschiedlichen Größenskalen, d.h. auf der Ebene einzelner Domänen sowie auf der Ebene vieler Domänen.. Das elastische und plastische Verformungsverhalten der einzelnen Domänen wurde mittels Ultraschallmessungen und Druckversuchen an Miniaturproben untersucht, welche aus dem Polydomänenwerkstoff entnommen wurden. Die Schädigungsentwicklung während Druckbeanspruchung wurde mit Hilfe von mikroskopischen in-situ- und ex-situ-Versuchestechniken analysiert. Die herstellungsbedingten Eigenspannungen sowie die innere Lastübertragung unter externer Druckbeanspruchung wurden röntgenographisch mit Hilfe einer energiedispersiven Synchrotron-Experimentiermethode untersucht. Die Untersuchungsergebnisse zeigen, dass die Domänen eine ausgeprägte elastische und plastische Anisotropie aufweisen. Die höchste Steifigkeit wird parallel zur Gefriertrocknungsrichtung beobachtet, die geringste senkrecht dazu. Die elastischen Eigenschaften einzelner Domänen mit unterschiedlicher Orientierung wurden im Lichte eines Modells diskutiert, welches dreidimensionale lamellare Strukturen mit abwechselnden Schichten unterschiedlicher Dicken betrachtet. Die in der Kammer eines Rasterelektronenmikroskops durchgeführte in-situ-Druckversuche an einzelnen Domänen zeigten, dass diese ein ausgeprägt anisotrope elastisch plastisches Verformungsverhalten aufweisen. Bei Beanspruchung parallel zur Gefriertrocknungsrichtung weisen die Domänen eine hohe Festigkeit auf und zeigen ein sprödes Verhalten. Bei Beanspruchung in andere Richtungen wird das Verformungsverhalten von der weichen Metallmatrix dominiert. Die

plastische Anisotropie ist im Vergleich zur Vorhersage mit theoretischen Modellen für Laminate weniger stark ausgeprägt, was auf die Existenz von Brücken zwischen den keramischen Lamellen zurückgeführt werden kann. Die Röntgen-Diffraktionsunstersuchungen zeigen, dass im Herstellungszustand stark fluktuierende phasenspezifische Mikroeigenspannungen vorliegen, welche durch das ungleiche thermische Ausdehnungsverhalten von Metallmatrix und der keramischen Vorform erklärt werden können. Untersuchungen zur inneren Spannungsverteilung unter externer Belastung zeigten, dass der Lastübertrag von der elastisch und plastisch weicheren metallischen Komponente auf die keramische Phase am ausgeprägtesten ist, wenn die Belastung parallel zur Lamellenebene erolft. Auch hier erfolgt der Lastübertrag allerdings nicht vollständig, as auf Schädigungen der keramischen Lamellen und Grenzflächendekohäsionen zurückzuführen ist. Mit den Untersuchungen konnte erstmals ein tiefer Einblick in die Mikromechanik dieser neuartigen Metall-Keramik-Verbundwerkstoffe gewonnen werden.

Table of contents

Abbreviations

CMC:	Ceramic Matrix Composites
PMC	Polymer Matrix Composites
MMC	Metal Matrix Composites
CIP	Cold isostatic Pressing
EMA	Effective Medium Approximation
UTS	Ultimate Tensile Strength
UPS	Ultrasound Phase Spectroscopy
RUS	Resonant Ultrasound Spectroscopy
SEM	Scanning Electron Microscope
LVDT	Linear Variable Differential Transformer
RMS	Root Mean Square
GLS	Global Load Sharing
LLS	Local Load Sharing
TEM	Transmission Electron Microscope
μCT	Micro Computed Tomography
UPS	Ultrasound Phase Spectroscopy
RUS	Resonant Ultrasound Spectroscopy
SR	Synchrotron Radiation
SSD	Solid State Detector
FEM	Finite Element Modeling
FOM	Figure of Merit
EFTEM	Energy Filtered Transmission Electron Microscopy

List of symbols

d_r	Reinforcement particle size (µm)
d_c	Critical particle size (µm)
d_m	Matrix particle size (µm)
V_f	Reinforcement volume fraction
R	Reduction ratio during secondary processing of powder metallurgy route
l	Structural wavelength in freeze-casting (µm)
u	Velocity of the ice front in freeze-casting (µm s^{-1})
u_c	Critical velocity for particle entrapment in freeze-casting (µm s^{-1})
r	Ceramic particle size in freeze-casting (µm)
E	Young's modulus (GPa)
G	Shear modulus (GPa)
K	Bulk modulus (GPa)
v	Poisson's ratio
S	Compliance
S_c	Effective compliance tensor of the composite material
φ_s	Shape factor of the inclusion used in EMA method
ξ	Adjustable parameter in Halpin – Tsai equation
η	Parameter relating composite and matrix moduli in Halpin – Tsai equation
e^C	Constrained strain in the equivalent homogeneous inclusion
e^T	Stress-free transformation strain in the equivalent homogeneous inclusion
S_E	Eshelby tensor
ρ	Density (Mg m^{-3})
V	Wave velocity (m s^{-1})
C	Elastic constant (GPa)
n	Mode number in RUS
fex	Measured frequency in RUS (MHz)
fr	Fitted frequency in RUS (MHz)
$\%err$	Error in fitting a mode in RUS
k	Mode symmetry

$\bar{\varepsilon}$	Continuum mechanics average lattice microstrain
α_{hkl}	Weighting factor for continuum mechanics average lattice microstrain calculation
ε_{hkl}	Individual lattice microstrain
T_{hkl}	Texture factor
m_{hkl}	Multiplicity factor
E_{hkl}	Young’s modulus of individual lattice planes (GPa)
$\bar{E}$	Macroscopic Young’s modulus of polycrystalline material (GPa)
l_c	Critical fiber length for load transfer in short fiber reinforced composites
d_f	Diameter of the short fibers
ε	Strain
τ_d	Interfacial shear stress (MPa)
σ_u	Ultimate tensile/compressive strength
$\sigma_{m'}$	Stress in the matrix at the composite strain equal to fiber tensile strain
m	Weibull modulus
σ	Stress (MPa)
δ_{ij}, δ_{il}	Kronecker’s delta
F	Externally applied force
s	Atomic displacement resulting from external applied force
A	Curvature at the bottom of the potential energy vs. atomic distance plot
C_{ijkl}	4^{th} order stiffness tensor
S_{ijkl}	4^{th} order compliance tensor
d	Lattice plane spacing
θ	Angle between the incident X-ray and diffracting plane
λ	Wavelength
L_i	Laboratory co-ordinate system for stress analysis by diffraction
S_i	Sample co-ordinate system for stress analysis by diffraction
ψ	Angle between the normal to the family of planes to be measured and the normal to the plane of the sample
ϕ	Azimuthal angle in the plane of the sample
$d_{\phi\psi}$	Lattice plane spacing in stressed condition

d_0	Lattice plane spacing in un-stressed condition
α	Thermal expansion coefficient (/°C)
$\Delta\alpha$	Thermal expansion mismatch between matrix and reinforcement
ΔT	Temperature drop
T_{act}	Actual temperature where the stress measurement is carried out
T_0	Stress-free temperature
I	X-ray intensity after attenuation in the object
I_0	Initial X-ray intensity
μ	Linear attenuation coefficient for the material being scanned in CT (cm^{-1})
x	Length of the X-ray path through the material (cm)
V_i	Volume of an arbitrarily shaped free elastic body
S_i	Free surface of an arbitrarily shaped free elastic body
L	General form of the Lagrangian
KE	Kinetic energy
PE	Potential energy
ω	Angular frequency
u_i	*ith* component of the displacement vector
$a_{i\lambda}$	Expansion coefficients
Φ_λ	Basis functions
a	Column vector
K	Symmetric and positive definite matrix
Γ	Symmetric matrix
O	Order of the matrix
f_{calc}	Calculated eigenfrequency in RUS
f_{mea}	Measured eigenfrequency in RUS
N_p	Number of eigenfrequcies
W_i	Weighting factor
$d(hkl)$	Lattice spacing of the (hkl) family of planes (Å)
$E(hkl)$	Energy of the (hkl) family of planes (keV)
h	Planck's constant (6.626068×10^{-34} m^2 kg / s)
c	Velocity of light (3×10^8 m s^{-1})
$E_0(hkl)$	Strain free energy (keV)
ΔE	Resolution of the SSD

$\Delta\theta$	Angular divergence defined by the incident and detecting slit system
k_j, k_k	Unit wave vectors
Γ_{il}	Christoffel acoustic tensor
n_j, n_k	Direction cosines of the normal to the wavefront
ΔV	Estimated error in measuring the wave velocity in a material
dV/df	Measure of the material's dispersion
Δf	Bandwidth of the wave or the pulse in the experiment
$\omega t + k_w x$	Phase function
k_w	Wave number
f	Frequency (Hz)
V_p	Phase velocity
V_g	Group velocity
L_s	Specimen length in ultrasonic analysis (mm)
N	Number of wave periods in the sample
$\Delta\phi$	Phase difference between input and received signals in UPS (rad)
E_L	Longitudinal Young's modulus of long fiber reinforced composite
ν_{LT}	Longitudinal Poisson's ratio of long fiber reinforced composite
$K_L^{'}$	Lateral compression modulus of long fiber reinforced composite
$G_{TT'}$	Transverse shear modulus of long fiber reinforced composite
m'	Global slope of the phase-frequency spectra
V_{ij}	Shear wave velocity (m s^{-1})
σ_{VM}	von Mises equivalent stress (MPa)
σ_c	Composite crushing strength (MPa)
σ_{YM}	Matrix yield strength (MPa)
σ_f	Fiber compressive crushing strength (MPa)
τ_{mu}	Ultimate shear stress of the metallic alloy
N_a	Number of atoms in a solid
β	Domain orientation angle
$V_p^{'}$	Pore volume fraction
ρ_p	Pore density
$\frac{1}{2}S_2^{hkl}$, S_1^{hkl}	Diffraction elastic constants
Ψ^*	Strain independent direction

l_f	Load fraction in a phase
σ_{appl}	Externally applied stress
X	Compressive strength along 0 ° domain orientation (MPa)
Y	Compressive strength along 90 ° domain orientation (MPa)
τ_{LT}	Compressive shear yield strength (MPa)
β	Domain orientation angle (°)
$\sigma_{xx} = f(\theta)$	compressive strengths of the single domains at different orientations (MPa)
θ_t	Angle of tilt of a poly domain sample around direction 3 (°)
$C_{11}^{'}$	Off-axis elastic constant along freezing direction for different tilts around 3 direction (GPa)

Superscripts

u: Upper bound

l: Lower bound

Subscripts

f: reinforcement

m: matrix

c: composite

T: transverse

L: longitudinal

TT: transverse – transverse

LT: longitudinal - transverse