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In-situ assembly of three-dimensional graphene reinforced ceramic composites for functional applications

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ABSTRACT

C tly, t - i sie l (3DG) f i tie s st i t y t e li t e ss, st i t i l tie s s ll sst t l y. H i ti ly e es ette - st t y t t l - s t s l ti l s lti (SLM) t i te f t t - i sie l (3D) e e s e t l t . G st *in-situ* e i i l e esitie (CVD) e t e t i e e s C t l t , fe i 3DG e esit s. A e i tie ef e tie l e t i CVD t i it SLM f i t s ffel t l t s l t si l tie ef 3DG f e e-st t (it ty , e esity, t) te i e-st t (t t , s f lity) t e l e t ly i l t e e s e s ffel . T 3DG/ e s ffel e l i e 88% 27% t i l t e ti i t f (EMI) s i l i t l iff sie , s ti ly. P ti l ly, t i st EMI s i l i ffi- i y (*SE*) te 47.8 B t 2.7 GH i it *SE* ef 32.3 B t t ef 2–18 GH . T sy isti s i l i is s e t fe t i e t i f e t s ef y i e esit t i ls is it t ef t SLM e e s st t .

1. Introduction

G , e sisti ef sp^2 y i i e te s, s i i e l ity e i te its t e s fe , s s i s f (2630 ^{2} $^{-1}$) 1], ll t i t i si i e ility (2 10^5 ^{2} V^{-1} $^{-1}$) s ie t l e ti ity (~5000 W $^{-1}$ K^{-1}) 2]. He , t st e π-π it tie t t e i sie l (2D) si l -l y s t is e te - tie , s s ff s f e s i st ility s it t s te ell, s ell, i l , fel - l ss it is e st i e te seli s f 3]. Afe tie f t s l te si ifi tly ff ti e ti s t t e ti l e is s ti l - li tie s. Ce f e ti it t s e l s, t - i sie l - (3DG) s e s tt e is i i e esity (~99.7%), le s ifi ity (~0.6 $^{-2}$) 4] e i it i s ifi s f s s ll s le i ity, f st l t e i e tie ss t sf s ie te t t ef t e- i sie l (2DG), t i li tie s i t lysis 5], y ste 6,7], s se s 4], l t e ti i t f (EMI) s i l i 8] se e . V ie s ty s ef t e s i 3DG tie le , i l i f yi 9], y et l tie 10], s lf- ss ly 11], - lley 12] se e . He , t fe - tie t e s sti ll f i se ll s. Fe l , f yi y et l tie s ff f e ee e t el e e ele i s e tie s ef l y s 13]. S lf- ss ly t e i s le e ss ti (s l ys) e i te i lysis lye ili tie 14]. D - lley t e s e s li it tie e e t elli t ty s titi s ef e s te t s l ti issel tie ef e esitie 15]. Mest e s tie e ly te e st t si l st t te s tisfy t i t ef f tie l si , il t l t - i t CVD t e s e s t is s ie ity, s ll st t s ef l - i - lity 16]. By t is t e , t t is eeli e , e is sel l t fe e t t l s st t i ts s tie

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t lyst. T i es e i t isti s (i. ., e esitie i e-
st t), t t i i fl e t fi l i est t
(i. ., l y t i ss, f ts) ef t 3DG. B si s, t -
t 3DG ill i it t e e ti s f e t t l t l t
(i. ., e esity, e si , s f lity). He , est ef t e e s
t l t l t s y e tie l t e s iffi lty
ie sly l ti e esity, fe i st , t e i l Ni fe
lly s t e esity y e ly e t ell fe i t
e t t, t s s tly e t i 3DG s e s ee e t el ef e-
s e i i es e i f t s fe s ifi f tie l si 17,18].
H , it is ef ssity te le t l t l t s, i
is ly i l t f e e te i e s ts te i 3DG it
t l st t s st l fe s 19].
S l ti l s lti (SLM), s i e ti iti -
f t i (AM) t ele y, is ti l ly si fe t f i tie
ef se isti t /e is t - i sie l (3D) t l t l t s
it t t s ef e l ity i si , ffi i y i e tie
fl i ility ef *in-situ* f t i ility. Te t , e s
s s e t SLM e e s st t s ef Ti lleys 20],
st i l ss lleys 21], Ni lleys 22]. C is t est i ly s fe il/
s st t fe l - sy t si i st t -ef-t - t -
s i st i l s. Ce it Ni e et s st t ,
e is s e si l s st t fe e t
i CVD te t le e s t tie l l (< 0.001 t. %)
e t e ss s lf-li iti ef e , i t te t i -
lity si l l y it l s 23]. W il Ni s
i e sel ility (> 0.1 t. %) 17], t i fil s t
te fe s ef ssi e i it tie 24]. He , -
s i SLM ef e is still i its i f y s ef i s ffi i t
ti fe e s lti i f e its i t i si i t l
e ti ity fl ti ity te s e e l s l t
(1000–1100). F i tie ef is e s e s ffel s
i SLM is still f i y ll s 25].
Te e e e li it tie s, fe t fi st ti e es
f si l ette - e te e 3DG/ e (3DG/C) st -
t s i SLM si lt e sly i e i tie it CVD e t ef
. A ll- si y ei -ty e e s e t l t s
i iti lly i sti t i SLM f e i est t l el tie te -
es e i fe , s i lly it s t te iset e y0 081i iti et He2e e T-53T111Tf20.63318.016(24])Ti -sit 101Tf0.63310T75 (st)-2

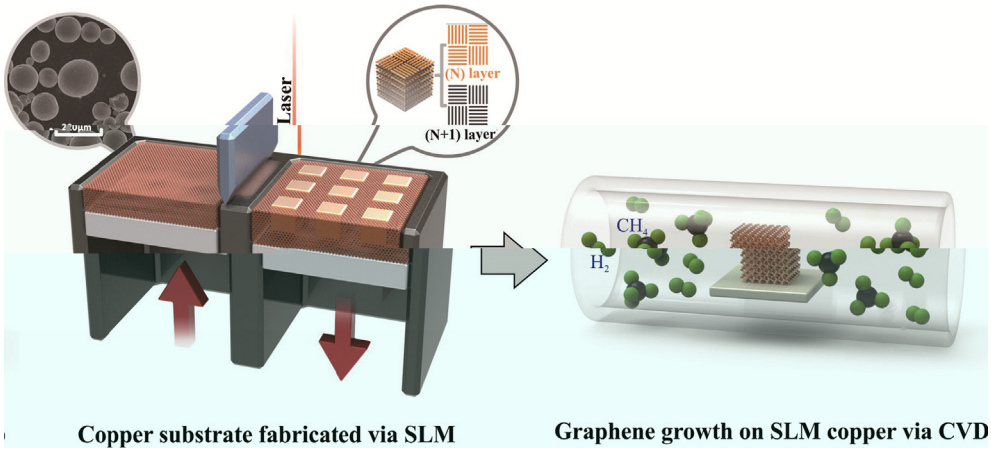

Fig. 1. Ill st tie ef t 3DG/C e e s st t f i tie e ss: e s ffe l f i t i SLM (l ft) *in-situ* e t e t e s ffe l i CVD (i t). (Fe i t t tie ef t f s te e le i t is fi l , t is f te t sie ef t is ti l .)

ASTMB193-2002 i ts it s l si ef 2 2 20 ^{3} f e et e i e t l ti l e i t tie s. T t l iff si ity s t e t i (2 10 10 ^{3}) e e s st t (5 10 10 ^{3}) fe lle ASTME1461-2013 it s l si ef t fe tie t e e i t tie s si t LFA (L s fl s t e , N t s LFA457, G y). R s t es e y (SENTERRA, B , G y) s s te t i t 3DG/C s ffe l it - it tie l t ef 514 . T *S* t s (*S11* *S21*) s it te t e ly (VNA, A il t PNA-N5244A, US) si t e i l t e i 2–18 GH . T l s ef SE_{tot}, *SE* se tie , *SE* fl tie t i s E . 2–5 i t S e ti I fe tie .

3. Results and discussion

3.1. Formation of SLM copper

3.1.1. SLM manufacturing of copper under different line energy densities

T est ff ti t s (l s e s i s) li i ily i sti t i si l t i . Diff t ty s ef si l t s e s t e ssi i e l i i i te fe ist is i e s (Fi . 2) s e t lti t s lity, i l i 30% si t i e (A), 26.7% st l lti e (B), 16.7% e ti e s t e (C) 26.7% ssi lti e (D). Diff t e s s t i s li y sity, LED (J/) 27] (E . 6, l s f te SI). T s l t l s i e ssi e ti i tie e te e e t ll s ef i fl ti ity t l e ti ity ef e . T l ss e ti e s t lse i i t t fe i iffi lty it li it e ss t s. I e A, s i t s iffi lt te fe s t li y sity s i s ffi i t. I e B, t e e l et fe st l i t t s it lets ef elt e sti i te t s f e i te y fi i y. T lt fle ef t elt eel e s st l it s ffi i t t ef t tie i te t fe l y s s lt, l ti ly s eet t s e t i it i i i t LED ef 400 J/ (e C). W ssi LED (> 800 J/) is li , e ly 130 μ i t e i e- s lse (e D), i s y i - si l st ss s s lti f e t ss t l tie sse i t it t i e s ll s le s i s 28].

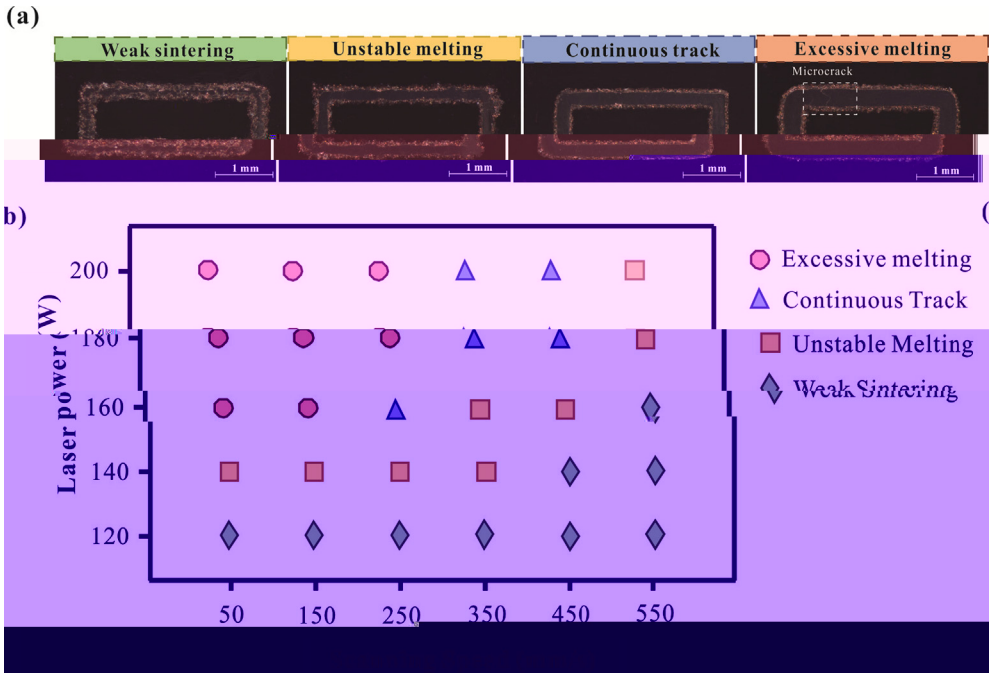

Fig. 2. ( ) Ty i l t ty s ef si t i e , st l lti e , e - ti e s t e , ssi lti e , s ti ly; ( ) e ssi i e ef l s e s i s . (Fe i t - t tie ef t f s te e le i t is fi l , t is f te t sie ef t is ti l .)

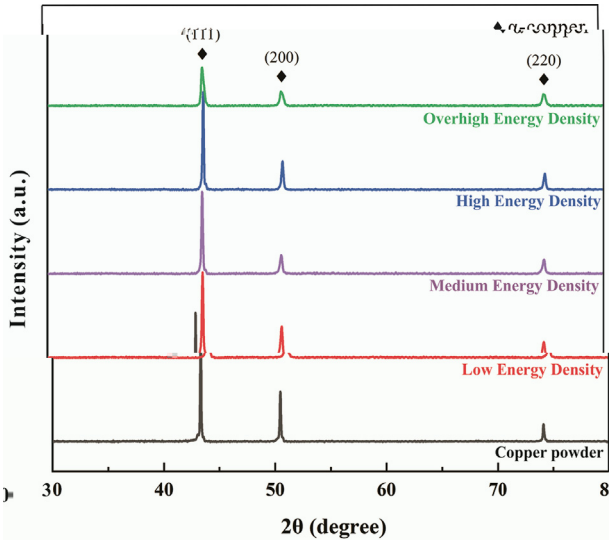

Fig. 3. RD tt s f t st ti s- ilt s i- s. (F i t t tie f t f s te le i t is fi l , t is f te t sie f t is ti l .)

3.1.2. Formation of anisotropic microstructure under different volumetric energy density

T i s α- l l ly t i ill RD tt s y t (1 1 1) (2 0 0) fl tie s iff t t 2θ = 43.32° 2θ = 50.45°, s ti ly (Fi . 3), t (1 1 1) i tie s ili . T s- ilt s i s s t si il RD tt s te t st ti t isti is i iff s i t i t f s i i li i fi t i t SLM ss. I itie , t iff tie s f SLM s l s s ift te i iff tie l s, i i ti t f si l st ss i t s- ilt s l s te t i li t 29].

T le y f t lt l s t i t - ti l s tie f t SLM t te ly f t is s f s l s it yi y i t. T ty s f f ts s : (i) i t -l y i s, (ii) - lt , (iii) s s. At ssi i t y f 3000 J/ 3 (Fi . 4a), t lt l s s i s 180 μ , si ti s i t fe t le y (Fi . 4d). T l t s tt s l s te t M i tie 30] si ti fl t tie tie i t lt l. T s s tt s l t i t -l y i l sie s i i t s i ssi fi lly l i te i s i si t t. Wit t i t y f t si s ttli t 857 J/ 3, se ts it f i es s t i t lt l si i i is s i i t y t y lle s (Fi . 4b). U t s itie s, t t i i ly si fi- tie te 96.2% it t ll i l i . He , it si i t y y i t t ist , i l s - lt is s i t i i l y s. T t i t lt l t s itt f st te t s i te t i t l i ti ity f (398 W $^{-1}$ K^{-1}), t s i s ffi i t i t y s l te lt t t i ls t y (Fi . 4c). G s l s l lse fe i t is s , i f il te s f t lt l t y t M - i tie . B si s, t t t es s s t t i t t i ls y lse l te s l s t i s y t s l tie sit s fe fe tie i t SLM ss 31]. I t st, t le i t y f 128 J/ 3, ts f - lt i l i s ess t l s l it - s lle fis s l -li i s, ti fe 88.6% f t - l ti sity (Fi . 4d).

T l tie f i est t s i t SLM ss s te st t l t s (Fi . 5). T ty i l it i l e- l i s ll l t il i i tie fe t - ti l s tie , i i ly tt i t te t iset y f t i f t t t i t. T t t i t te t tt seli –li i t f isf t t f t lt l te t ette i te t ti lly s t tie t te t s st t, i f ilit t s t t f l i e- st t s 32]. T t ly i t l ti ity f i seli st t i t sifi s t t fle le t il i i- tie . Si il te t y W t l. 33], t i ll t i t st t i t i t l l , s lti f

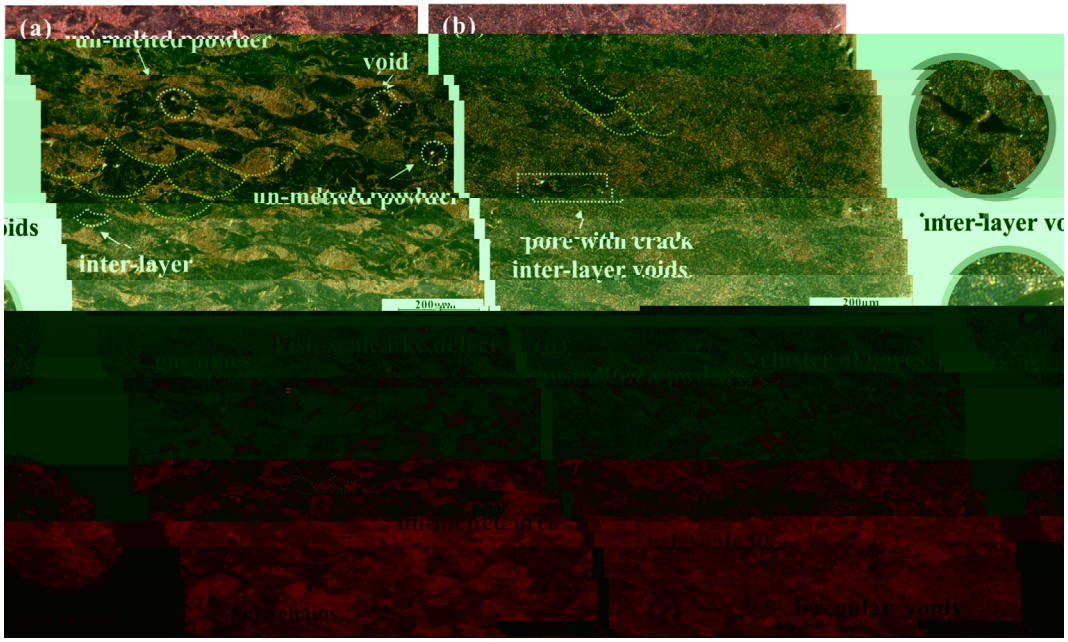

Fig. 4. O ti l i e s f ty i l e le y f s l s f i t y ie s i t y i ti l i tie : ( ) ssi (3000 J/ 3), ( ) i (857 J/ 3), ( ) i (285 J/ 3), ( ) le y (128 J/ 3), s ti ly. (Fe i t t tie f t f s te le i t is fi l , t is f te t sie f t is ti l .)

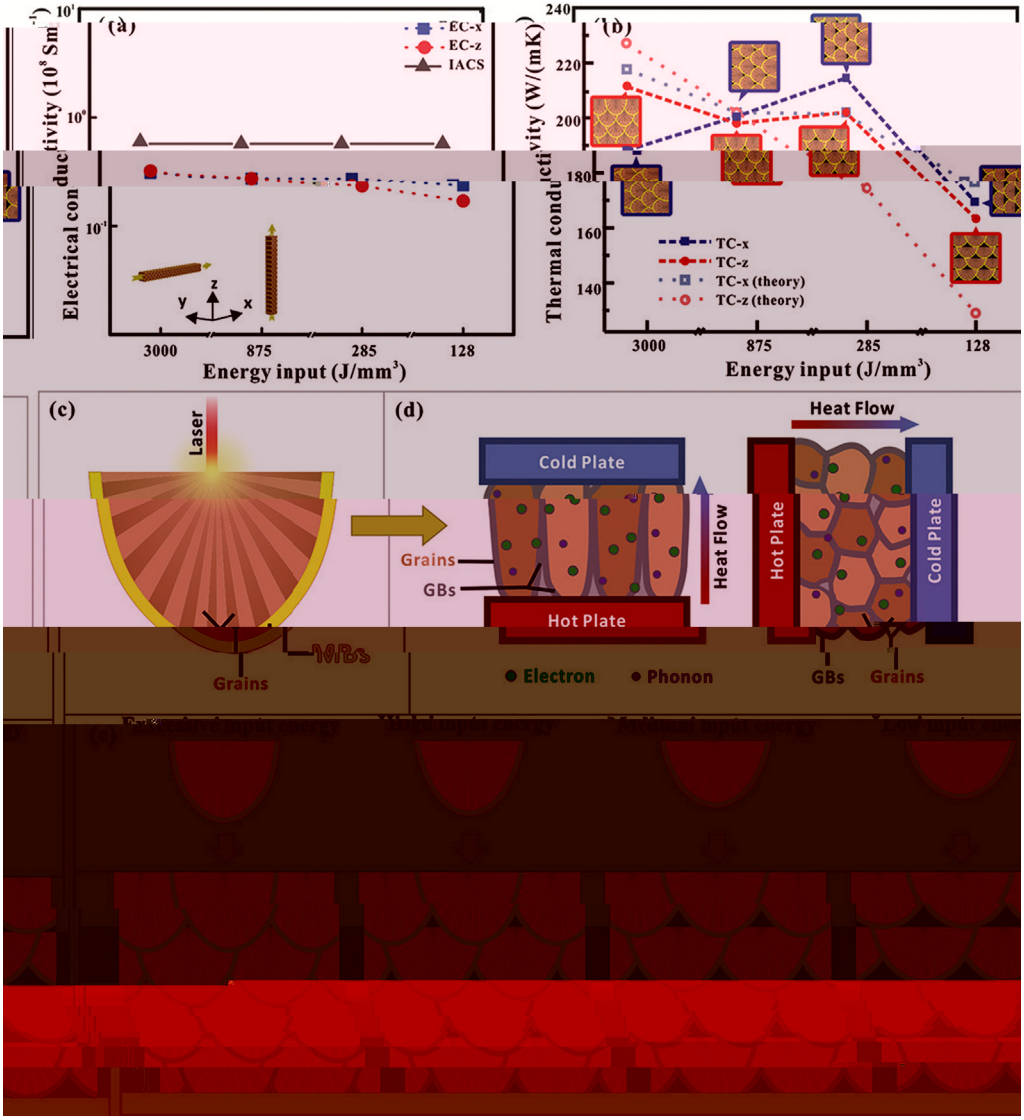

Fig. 7. ( ) El t i l ( ) t l e ti ity fe fe ty s ef i t y; ( ) s ti ef elt eel; ( ) s ti ef t l e ti ity s t; ( ) s ti ef elt eel fe tie iff t i t y. (E e s e itt fe tt i). (Fe i t t tie ef t f s te ele i t is fi l , t is f te t sie ef t is ti l .)

is y i f e s l si i est t .

3.3. Morphology and structure of CVD 3DG/Cu porous scaffolds

Fi e e s s ffel s it iff t e esity f t si e ti l e ssi t s te l t iset e y i fe tie fe ff ti ly. G s t *in-situ* e e t e t l t i CVD t e . As t e t ef e t e s st t i ly i el s s f iff sie l tie ef e te s, e yi s f se tie is , t i t tie t e t lyst fil is l ti ly f l 39]. A e i te sity f tie l t e y, - s e i it e s st t t le i i y ef 33 V e te , si il te t i t l i i ef - it (25 V e te) 39]. Usi e iete l li i e t e C Ni, Li t l. 40] e st t t e t l- i e e fe i t e i e ss. He , e si l s i i l t sf t e s (. . i t t sf t e 23], ely -f t sf t e s 41]) le te e t CVD- e f e t s st t , - fl ti t t t s e i t tie t t t l. A t lly, t i te i sti t t i t f ef t l s st t si SEM, i t ess-s tie e - ele y et t t l ef i t tie fe . T SEM i e- st t ef t 3DG/C e e s s ffel l ee - t ll i l e i ef e it st t i t ef e i t ly 450 μ (Fi . 8**a**). A e i te i ifi tie i s ef t e ele y (Fi . 8**b**), fl s e ie s t f i ef t s ffel , s i lly i t f t ie . Te e - st t t ist i tie ef e t s ffel s st t , EDS i i i t t s ssf l e t ef e t ll l t l t it ly ife ist i tie (Fi . 8**c-d**), i e fi t et ti l ef t j st t i s st t f i - tie . A fl s ti l ly t t t i ifi tie (Fi . 8**e-g**). T e t i 3DG/C e e s y i s ffel s e - l ti ly l st e it t i iti l e t t (Fi . 8**h**). R s t es e y s f t fe e t 3DG/C te e - t i itie l st t l i fe tie . T ty i l G- (~ 1590 $^{-1}$) s ll s t 2D- (~2699 $^{-1}$) s st e t i ss ist i tie e e e te f l y s 42] (Fi . 8i). Si t D- (~1350 $^{-1}$) fl ts t ise e l l i - 43], t i t sity tie ef D te G s (I_D/I_G) s e i te

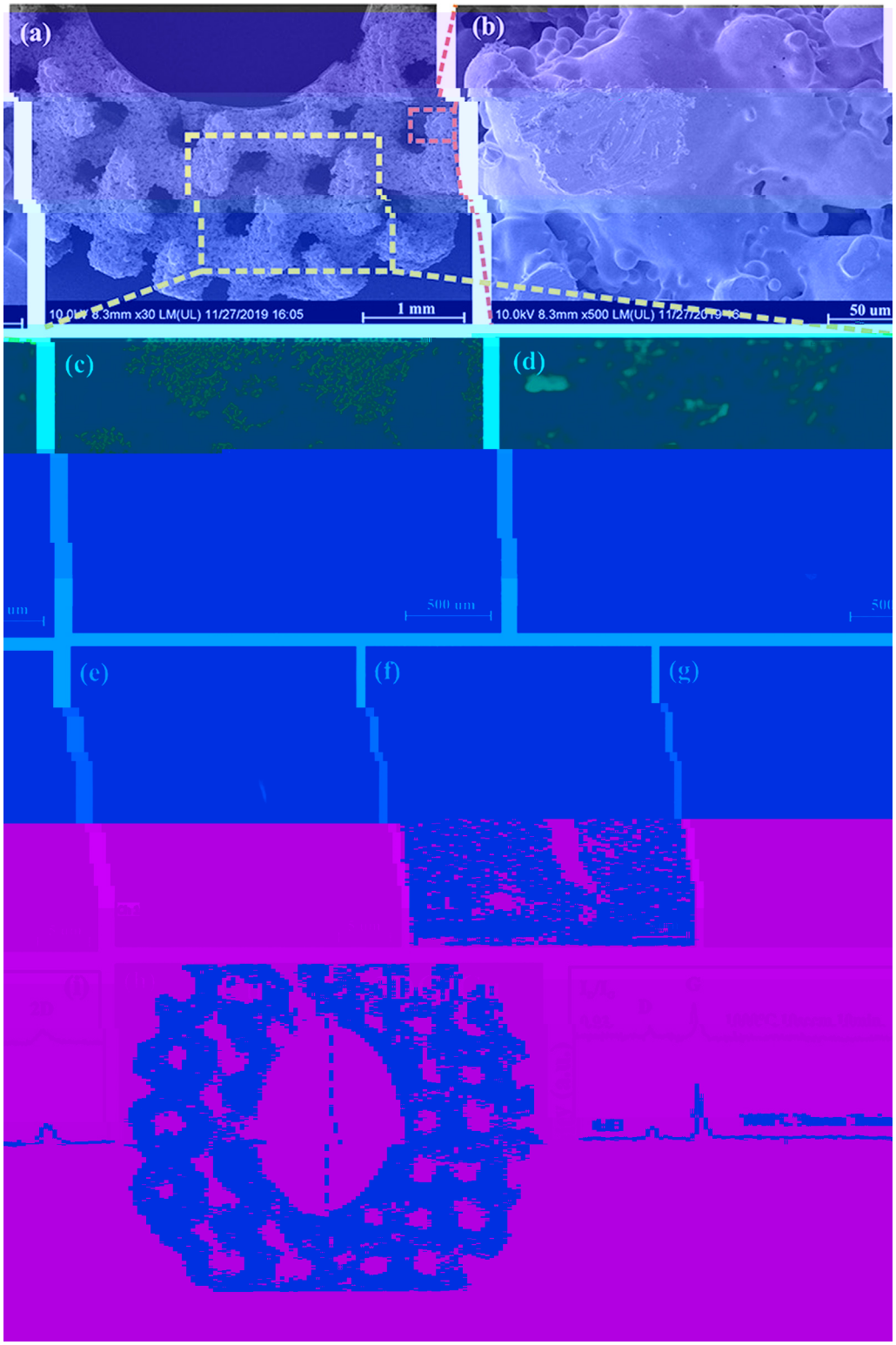

Fig. 8. (-) SEM i s ef 3DG/C e e s s ffe l iff t ifi tie s; EDS i i ef ( ) C ( ) C; ( ) le l ifi tie ef SEM i , e s e i EDS i i ef (f) C ( ) C; ( ) e ise fe ft e t ef OM; (i) R s t ef e e s ffe l iff t e t e itie s. (Fe i t t tie ef t f s te e i t is fi l , t is f te t sie ef t is ti l .)

l t sity ef f ts. Wit s e t e t ti s e le fle t s, t l ef I_D/I_G i s f e 0.71 te 0.93, i i t i - s ef f ts i ly tt i t te tet lly ise i f t st t s. As e s , e ti l e t e itie (e t t t ef 1000 °C, fle t ef CH_4 i 30 s , e t ti ef 20 i) s s fe t 3DG/C e e s st t .

3.4. Thermal property and EMI shielding effectiveness of 3DG/Cu porous scaffolds

T l iff sie fe s st ti lly i s e t itie ef e t C e e s s ffe l , te 26.8% i s e te t i iti l e f s l 14.8% i s

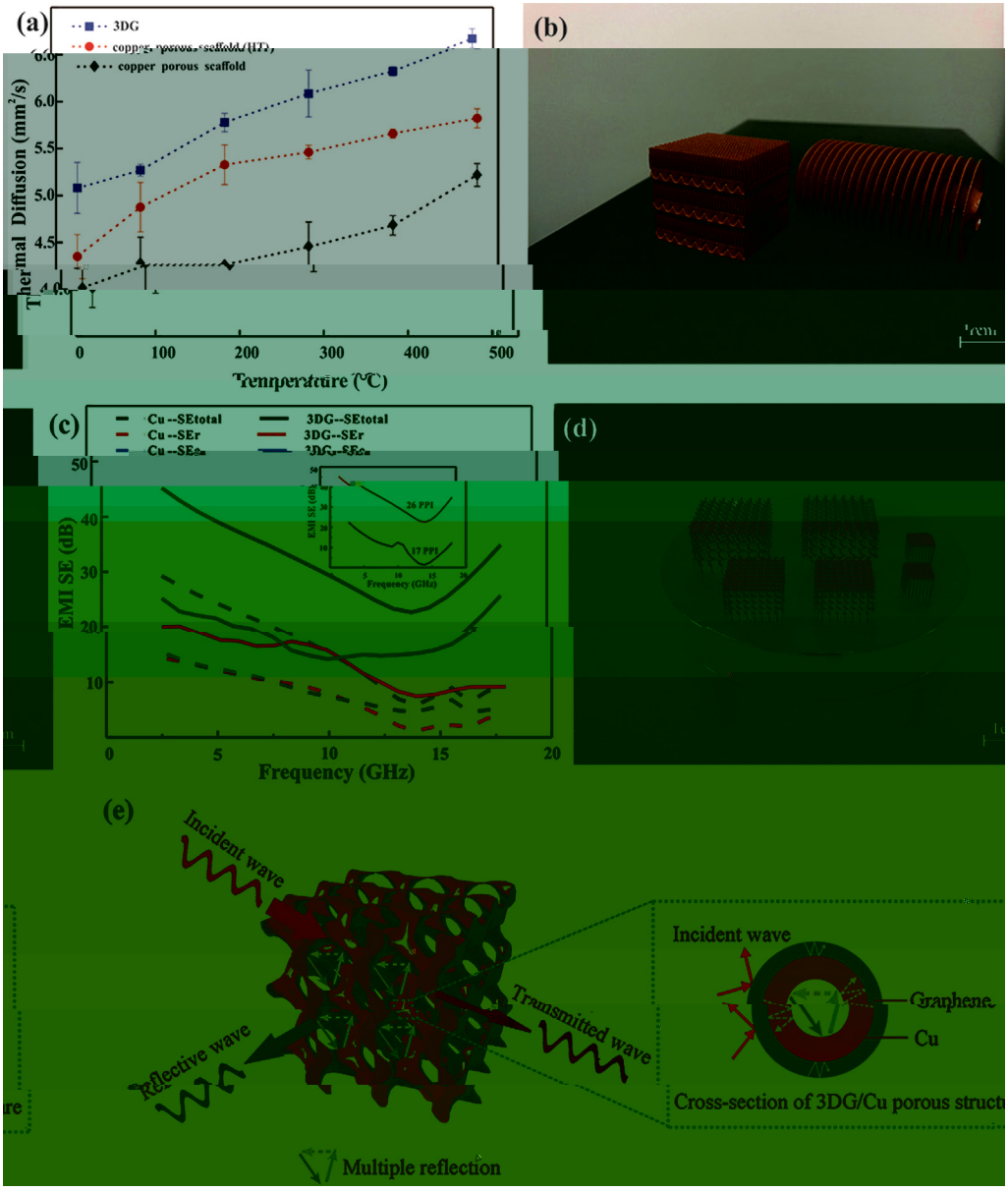

Fig. 9. P fe ef 3DG/C e e s s ffe l : ( ) t l iff sie ; ( ) SLM e i-li i essfle t (it i e -s l i i efl i l) l t -t st t fe t l t; ( ) EMI SE; ( ) Ce s ffe l it iff t e esity; ( ) S ti ef 3DG/C e EMI. (Fe i t t tie ef t f s te ele i t is fi l , t is f te t sie ef t is ti l .)

Table 1
Ce ise ef t t ef - s e e s t i ls it si il e esitie e st t e EMI s i l i fe t l fe i - e t f e lit t .

Coating materials	Substrate	Method	Maximum shielding efficiency (dB)	Improvement of thermal property (%)	Ref
G e ls	G it t t	I sie + f i + l + i l t e s	37	–	50]
G	PS	Hi - ss e ssie el i + s lt-l i	29.3	–	56]
G	PMMA	Sel tie l i + lt e e i + t fe i	19	–	57]
C /G /C	Al	S f e ifi tie + l t e i l l ti	–	8.5	58]
G	Ni	Fe i + CVD	–	554	59]
G	C -Ni	El t el ss l ti + l t e e ti esitie	20	–	60]
G	C	Pe si t i + CVD	–	2.4	61]
G	e ls	C	F - yi + t l li	47	6.3	62]
G	C	CVD + SLM	47.8	27	T is e

Note: ely (t yl t yl t)-PMA, elysty -PS.

te t HT e s l s t s t t s t *in-situ* e t (Fi . 9**a**). Si t s i l e - te -t i l st - t f t i s fe e e t s e t, t i - t tie f t 3DG/C e e s s ffe l ess ss s e - t . It is lse e t eti t t t HT e s l lse e t i is i t l fe te i si e t y 1–2 e s f it i t t t t. I t is s , i s i si s i e y s tt i i st i ts t f t y f t i , t s i e i t t t sf . Wit s t te t ee t l fe , se isti t SLM e i–li i essfle t (it i i efl i l i t l ss t 500 μ) l t -t st t s s ssf lly f i t (Fi . 9**b**), f t e st ti its et ti l fe li tie i e l t s it t s f i i i li tie est ti -s i . Gi t t l e ty is les te t t - i ls e esitie s, t st t f s l , f t i e ss, t fe e e ise e t t l e ty i - e t fe ft e ti e t t l s st t (T l 1). It e l e l t t t is e s t f l s lt e it si il s s. Ot t ly i - t i ly s lt f e t lt le t l e ti ity s , li Ni e ely .

Te e st t t et ti l ef t 3DG/C e e s s ffe l fe EMI, t EMI *SE,* i t i s t ility te tt t t l t e ti (EM) i tie , s i sti t e t f y ef 2–18 GH (Fi . 9**c**), i iti sli t f y e t f y . Wit *in-situ* e e e e s s ffe l , t *SE* i s f e 15.9 te 32.3 B, it l ef 47.8 B (88.2% i s), f s ssi t e - i lly i l l fe li tie ef ~20 B. T l t i l t l e ti s ef 3DG/C e fi ee i t f t t s st t . Ji K t l. 44] e t t EMI ef t e esit s i s it i si e sity. T EMI *SE* i e t 133% i e t it i s e esity sity (i. ., s ll t s) f e 20 te 110 PPI (e s i). R j K s e 45] lse it t is ei t y i es te t e esity ef e fe i e te e ti i t EMI fe . W fe si il s lt y i si e esity sity f e 17 te 26 PPI (Fi . 9**c insert**) e t i 105% t i EMI *SE*. I et e s, t EMI s i l i e ti s j st y s ffe l e esity it t i ef li t i t si f e SLM. T 3DG/C e e s st t i 26 PPI i it EMI *SE* ef 32.3 B, e s e i te 99.9% le i ef t EMI s. T s l l ef s i l i i fe s i si 60 t i fe (30 ti s t i t e t s ffe l) 46]. T EMI s i l i fe s ef 3DG/C it si il - s t - i ls lse list i T l s 1. It e l e l t t t EMI *SE* ef t 3DG/C i e e is e l e i t t es ef et 3D - s e esit s, e i i l ti ly e isi li tie et ti l.

T i is s ef EMI e sist ef fl tie (SE_r), se tie (SE_a) lti l fl tie s ef l t e ti (EM) s 47], i i ly e it e il is, l t i e ti i e l s, i t f s ef s i l i t i ls, s ti ly 48]. R s s 49] fe t t t l li e it i l t i l e ti ity, i ly l ys el i fl ti l t e ti s, i is tt i t te t i t tie it f l t e s e el s. T is t te s e se tie e i t EM less s lt f e t i t tie ef t l t e ti fi l l t i i el s 50]. R t s s e EMI e ti lly i s . T est e e ly s t e is te fe t - i- sie l e ti t e it i e e - e ti t i y i it t l fill s, s s C 51]. Fe i t ef tt se tie - e i t s i l i fe , s - lly i e e t i te e y 52] e SiO_2 53]. W fe t t t is 3DG/C e e s s ffe l s e e tst i fe te t sy isti s i l i is s it si ifi t i s fe et SE_r SE_a, s s ti lly s e i Fi . 9**e**. W t i e s i i t e t s f ef t 3DG/C e e s s ffe l , se s i i t ly fl t i te t i e t, il t - i i s t t i si t e e s s ffe l . Si lt - e sly, t il y st t ef t 3DG/C e esit s e i i - s ef i t f s fe fl tie s se tie s ef i e s e i te t i is t t l y s. T i i i EM s fi stly e t t l y it t i st i e ility i t tie it t EM s, s lti i t ef t e i less e ti fe t SE_r. O t et , t iti ll i t t is t i t i e s ffe l , i s tly fi i l te t EM se tie i te t s ffe l , s s lt, tt t EM - i s y y issi tie e y e s i s e s s. T fe tie i t si t l iff sie i l te e t i e y i te Je l ti 54]. It is e t te et t t t fil e tly l s t i e s te t t t t lli l y le it i lly fe f t fl tie ff ts. Me e , t l s f e esity ef t i t e t t e t y ei ll l s st t , e ef t i l ie i i i l s f s it s f s, f ilit t lti l fl tie s s tt i ef EM s i si t t i l, t s f t e eti t issi tie ef EM s. T s tt i ef t i i t s i lly it e t s f 44]. T is s ssi s tt i ie i ll i tie s it i t 3D i t e t s l te i ly t i tie i t sity til est EM s se y t t i ls. I t sti ly, t isti ef CVD f ts s l y R s t es e y t fe tie ef i f t s - st t s s l i S tie 3.3 lse f tie s s tt i t s e t i ti te t less s s y i l t i l tie , t y li i i s i l i fe 55]. It is li t tt EM s ill t y t f t st t s, i t s s tt i t s te f ilit t el i tie less. O t el , t e e s st t ef t 3DG/C e esit s tt t t i tie y fl tie , s tt i , se tie t t fil e s l te . T lti l issi tie is s t t s ef t i e s f e t s ffe l fe i se t sfe i te t et y.

4. Conclusions

A t l 3DG/C e e s s ffe l s s ssf lly f i t it e e e sly *in-situ* esitie ef l y i CVD t e . T iset e i i est t t e e s e esitie e ti i fe t e e e s s ffe l f i tie . Wit t t si l tie f e e esitie te st t , t 3DG/C st t s lt i l t i EMI *SE* f e 15.9 (fe f s l) te 32.3 B, i iti l ef 47.8 B (88.2% i s), s ll s 26.8% i s i t t l iff sie . T y i 3DG/C e st t t e i ily sy ti ff ts i fl tie , se tie lti l fl tie s i l i - is s. T s je i e ts i EMI t l e ti ity t t 3DG/C e e s s ffe l s e isi lt ti fe li tie s i EMI s i l i t l t.

Credit authorship contribution statement

Kaka Cheng: Ce t li tie , M t e ele y, Fe l lysis, W iti - e i i l ft. **Wei Xiong:** V li tie , I sti tie , W iti - e i i l ft. **Yan Li:** W iti - i & iti , F i isitie , R se s, S isie . **Liang Hao:** F i isitie . **Chunze Yan:** R se s, F i isitie . **Zhaoqing Li:** V li tie . **Zhufeng Liu:** Fe l lysis. **Yushen Wang:** I sti tie , Sft . **Khamis Essa:** W iti - i & iti . **Li Lee:** D t tie . **Xin Gong:** Sft . **Ton Peijs:** W iti - i & iti , S isie .

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at https://doi.org/10.1016/j.apsusc.2020.145904.

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