Journal of Alloys and Compounds 651 (2015) 357–362

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Ultrafast atmospheric-pressure-plasma-jet processed conductive plasma-resistant Y_2O_3/carbon-nanotube nanocomposite

Chih-Hung Wu, Jian-Zhang Chen*

Graduate Institute of Applied Mechanics, National Taiwan University, No. 1, Sec. 4, Roosevelt Rd., Taipei City 10617, Taiwan

A R T I C L E I N F O

Article history:
Received 28 July 2015
Received in revised form
11 August 2015
Accepted 12 August 2015
Available online 15 August 2015

Keywords:
Atmospheric pressure plasma jet
Y_2O_3
Screen-printing
Carbon nanotube
Composite
Nanoporous

A B S T R A C T

We developed an ultrafast sintering process for a conductive plasma-resistant Y_2O_3/carbon-nanotube composite using an atmospheric-pressure-plasma jet (APPJ). The processing time can be as short as 3–5 s. The incorporation of carbon nanotubes (CNTs) significantly improves the conductivity. A N_2 APPJ reacts violently with the CNTs and carbonaceous materials in the screen-printed pastes, enabling ultrafast processing. The synthesized films show great erosion resistance to low-pressure CHF_3 inductively coupled plasma (ICP). The conductivity remains at a similar level after exposure to the CHF_3 ICP for 30 min. This coating can serve as a protective layer in a low-pressure plasma environment. The high conductivity (~0.01 S cm^{-1}) is advantageous in preventing arcing or charging effects in the low-pressure plasma environment.

1. Introduction

Yttria (Y_2O_3) is a high hardness, mid-infrared transparent, high-dielectric-constant (high-k) material that has been applied in many different fields [1–6]. Owing to its high hardness and transparency in the mid-infrared spectrum region, it has been used for mid-infrared transparent windows and missile domes [1,2,7,8], mid-infrared waveguides [9,10], and anti-wear anti-reflection coating for mid-infrared lenses [11,12]. The high-k property makes Y_2O_3 suitable for use as the gate dielectrics of transistors and capacitors [3–6,13]. Y_2O_3 is also an anti-plasma-erosion material that can be applied in a plasma processing chamber [14–19]. When used in a plasma environment, the high insulating property of Y_2O_3 may cause surface charging and arcing. Therefore, some techniques have been used to improve or control the conductivity of Y_2O_3 [15,18,19]. Carbon-doping has found to be an effective method to significantly improve the conductivity of Y_2O_3 [15].

Y_2O_3 coatings have been developed using several techniques including sputtering [20–22], aerosol deposition [14], sol–gel [23,24], and e-beam evaporation [25]. In this study, we have developed an ultrafast synthesis method for the preparation of Y_2O_3/carbon-nanotube (Y_2O_3/CNT) composites. A N_2 atmospheric pressure plasma jet (APPJ) is used for the rapid sintering of the screen-printed Y_2O_3/CNT pastes. The vigorous interaction between the energetic N_2 APPJ and the carbonaceous materials/organic compounds in the pastes enables the ultrafast processing capability [26–28]. CNTs are added to the Y_2O_3 nanoparticle screen-printing pastes to modulate the conductivity. A Y_2O_3/CNTs composite with high conductivity (0.01–0.1 S cm^{-1}) can be synthesized in 3–5 s using APPJs. In comparison, conventional furnace calcination would take 10–15 min at ~400–500 °C.

2. Experiment details

The Y_2O_3/CNTs pastes were prepared by a modified version of a procedure reported in literature [29]. Ethyl cellulose (5–15 mPa s, #46070, Fluka) and ethyl cellulose (30–50 mPa s, #46080, Fluka) were dissolved in ethanol to form two separate 10 wt% solutions, called EC1 and EC2, respectively. 2.8 g of EC1 and 2.2 g of EC2, 4 g of terpineol, and 5 ml of ethanol were mixed with 1 g of Y_2O_3–1 wt% CNT or Y_2O_3–5 wt%CNT powder (Y_2O_3, diameter: 32–36 nm, purity: 99.9%), (CNT, diameter: 40–90 nm, length: >10 μm, purity: 99.5%). These organic compounds provide essential viscosity for the screen-printing pastes. After mixing all the ingredients, the paste was stirred at 300 rpm for 24 h. Subsequently, the mixture was processed under low pressure at 50 °C in a rotary-evaporator for

* Corresponding author.
E-mail address: jchen@ntu.edu.tw (J.-Z. Chen).

http://dx.doi.org/10.1016/j.jallcom.2015.08.085
0925-8388/

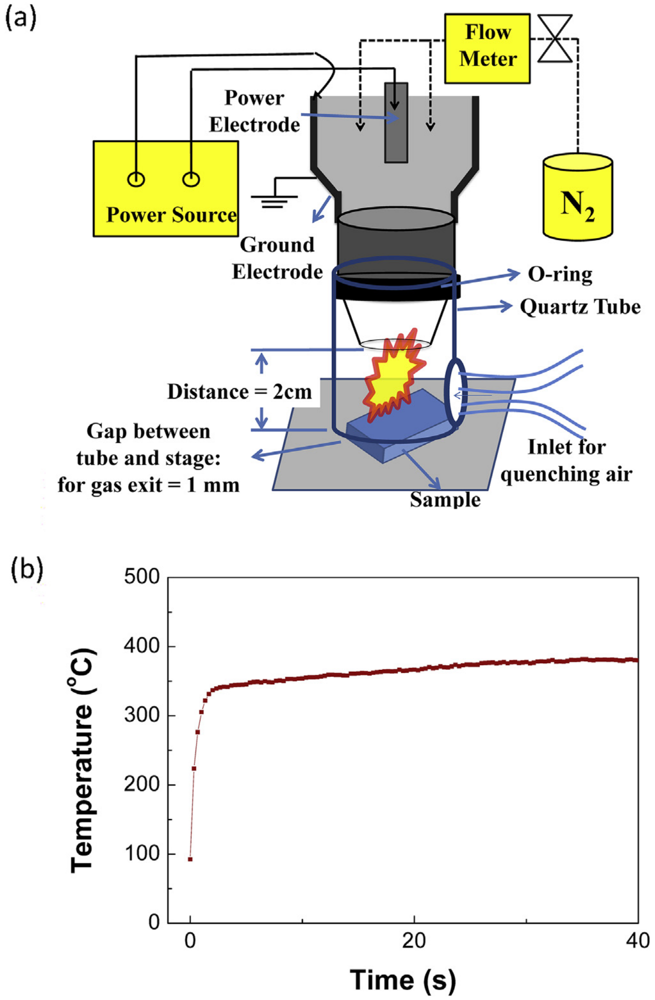

Fig. 1. (a) Schematic of APPJ system. (b) Evolution of substrate surface temperature upon APPJ operation.

6 min to extract excess ethanol.

The pastes were deposited on a Corning Eagle-XG glass substrate using the screen-printing technique. The thickness of the nanoporous Y_2O_3/CNT composite layer was ~8 µm. The printed pastes were sintered by N_2 APPJ for 3, 5, 10, and 30 s. Fig. 1(a) shows a schematic of the APPJ apparatus. A Pyrex™ tube with a side hole was installed at the exit of the jet to adjust the participation of environmental air in the reaction. The distance between the Pyrex™ tube exit and the nozzle of the plasma jet was 2 cm. The APPJ operation conditions were as follows: applied voltage, 275 V; N_2 flow rate, 38 slm; on/off duty cycle, 7/33 µs. Fig. 1(b) shows the temperature evolution under APPJ exposure. The temperature increased rapidly to 341 °C; then, it increased steadily to 377 °C within 30 s. Table 1 lists the temperatures at specific times. The fast removal of the organic compounds was made possible by the synergetic effect of the APPJ and temperature. To prepare the

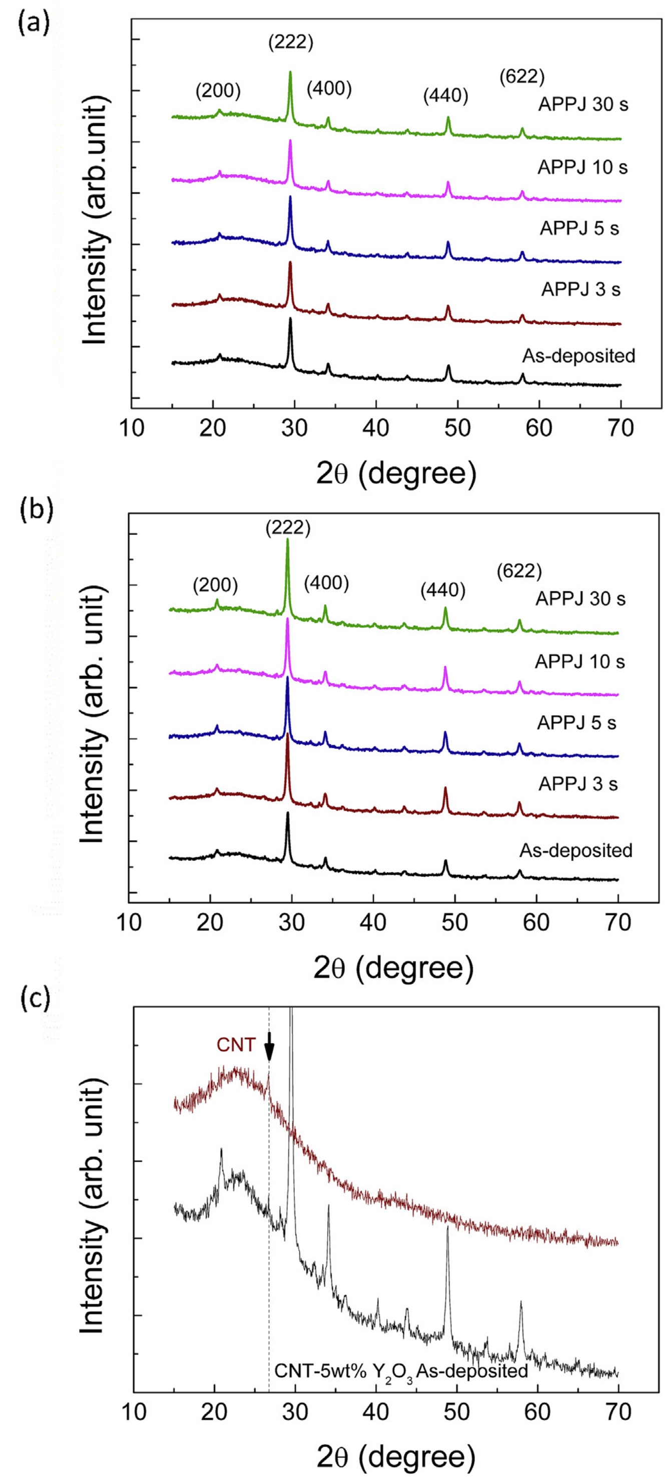

Fig. 2. XRD patterns of (a) APPJ-sintered Y_2O_3–1 wt%CNT, (b) APPJ-sintered Y_2O_3–5 wt%CNT, and (c) as-deposited pure CNT and Y_2O_3–5 wt%CNT thin films.

Table 1
Substrate temperatures during APPJ operations.

APPJ treatment time (s)	Temperature (°C)
3	341
5	345
10	353
30	377

Table 2
Conditions in plasma erosion experiment.

Parameter	Value
CHF_3 flow rate (sccm)	50
Chamber pressure (Pa)	1.2
Plasma power (W)	100
Exposure time (min)	6, 30