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Capillary pressure in graphene oxide nanoporous membranes for enhanced heat transport in Loop Heat Pipes for aeronautics

Cosimo Buffone [a,*], Jerome Coulloux [b], Beatriz Alonso [c], Mark Schlechtendahl [d,e], Vincenzo Palermo [f], Amaia Zurutuza [c], Thomas Albertin [b], Simon Martin [b], Marco Molina [g], Sergey Chikov [a], Rolf Muelhaupt [d,e]

[a] Microgravity Research Centre, Universitè Libre de Bruxelles, Avenue F.D. Roosevelt 50, 1050 Bruxelles, Belgium
[b] Atherm, Z.I. – 1 rue Charles Morel, 38420 Domène, France
[c] Graphenea, Tolosa Hiribidea, 76, 20018 Donostia, Gipuzkoa, Spain
[d] Freiburg Materials Research Center, FMF, Albert-Ludwigs-University of Freiburg, Stefan-Meier-Str. 21, 79104 Freiburg, Germany
[e] Institute for Macromolecular Chemistry, Albert-Ludwigs-University of Freiburg, Stefan-Meier-Str. 31, 79104 Freiburg, Germany
[f] Institute for Organic Synthesis and Photoreactivity, CNR – National Research Council of Italy, Via P. Gobetti 101, 40129 Bologna, Italy
[g] Selex ES, Viale Europa, 20014 Nerviano, MI, Italy

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ABSTRACT

We describe a novel application of graphene-based materials to enhance heat transport in sintered metal wicks, which are the core components for Loop Heat Pipe (LHP) evaporators. Standard metal wicks limit the applicability of LHP to about 8–10 m of transport length and around few meters of gravitational head. This is due to the typical average pore size (about 1 μm) in the sintered metal wicks made of nickel or titanium, which are the most commonly used materials. The idea of the present work is to bond a layer of graphene on top of the wick facing the vapor side of the LHP evaporator. The much smaller pore sizes of graphene (around tens of nanometers) would produce a significant increase in capillary force, while at the same time minimising the pressure drop due to its microscopic thickness (few microns). The wicking height (i.e., capillary rise of a liquid inside a pore) measurements demonstrate that there is an improvement of at least more than 3.5 times when the graphene coating is used, compared to the standard nickel sintered powder wick. This means that the heat transfer of a graphene LHP could work in a spatial range in excess of 28–35 m, which would allow breakthrough applications such as anti-icing of aircraft wings and propellers, as well as wind turbines that cannot be addressed by standard LHP technology.

1. Introduction

Heat Pipes (HP) are passive heat transfer devices capable of transferring heat between two remote locations kept at different temperatures [1,2]. HPs are evacuated vessels inside which a working fluid undergoes evaporation and condensation; due to the very large latent heat of evaporation, a small quantity of working fluid is required to transfer large heat loads. In addition, because evaporation and condensation are nearly isothermal processes, there are relatively small temperature gradients in the HP with limited thermal stresses of the materials involved. The core of the HP is the wick structure which provides capillary pressure to overcome all the pressure losses inside the HP. There are different types of wicks for HPs [1,2], the most commonly being based on: 1. Groove arrays; 2. Screen meshes; and, 3. Sintered powders. The best performing wicks are made of sintered powder because the average pore size is much smaller than that of grooves or screen meshes. However, the manufacturing process for sintered powder is more complicated and costly.

HPs have been used in a variety of applications, from electronic cooling [3], to aerospace [4–6], to core cooling for nuclear power plants [7], just to mention but a few. There are some important limitations with standard HPs which are connected with the fact that vapor and liquid are in intimate contact and at high loads this can result in reaching the "entrainment limit" during which the evaporator is starved of liquid that cannot return from the condenser by capillary action. The other limitation is connected with the fact that in standard HP the wick is present all over its length, which limits the shape that the HP can have and makes it much heavier. These important limitations were overcome in the '80s by the invention of Loop Heat Pipes by Maidanik and co-workers [8], who developed this concept for space applications.

* Corresponding author.
E-mail address: cbuffone@ulb.ac.be (C. Buffone).

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Loop Heat Pipes (LHP) [9,10] can transport heat over distances of 8–10 m compared to a few meters for standard grooved and sintered HP, and up to few meters against gravitational head compared with less than a few cm for grooved HP and less than 1 m for sintered HP, when working against gravity. LHP are more complex to predict and much more complicated to build, resulting in higher manufacturing costs compared with standard HP technology. However, the LHP allows in certain circumstances to control the evaporator temperature [10], something that is not possible with standard heat pipes that operate in a "constant conductance" mode. LHP have been also used in demanding high g-force environments such as aboard fighter jets. The most complex and costly part of a LPH is the evaporator where the wick structure resides. The complicated network of superficial grooves renders the LHP evaporator a very challenging part to make using the current manufacturing techniques (mainly sintering and machining).

The limitation on transfer distance has kept to date LHP technology out of applications such as anti-icing of aeroplanes and aeroengines where there is a lot of waste heat generated inside the core of aeroengines which could be used to heat up cold spots such as nacelle lips, Outlet Guide Vanes and even wing leading edge of aircrafts. If the improvement in capillary pressure is substantial, then LHP could be also used to anti-ice propeller blades, which typically suffer dramatically from icing because propeller aircrafts fly relatively lower than turbojets, just at the same height where subcooled droplets are more common in the atmosphere.

The revolutionary work of 2004 by Geim and Novoselov [11,12], demonstrated the possibility to process and manipulate thin sheets with mesoscopic lateral size and atomic thickness. This has allowed the production of a new class of 2-dimensional (2D) materials, where billions of sheets shall be stacked over each other to form highly anisotropic membranes and coatings. New electronic and chemical properties shall be achieved by creating complex multilayered structures of chemically functionalized graphene sheets [13–15] or by stacking together different kinds of inorganic, 2-dimensional sheets [16–18].

Such kind of materials features a highly interconnected, 2D porous structure, where both the chemical nature of the 2D walls and the average pore size can be tuned [19].

Molecules of liquids diffusing through these 2D pores will feature new properties [20,21]. To this aim, exciting results have been recently published on the capillary transport of gases and small molecules inside 2D materials such as graphene or graphene oxide (GO).

Geim and co-workers demonstrated that GO membranes can block very effectively some gases and be selectively permeable to others, such as water [22,23] or small ions [24]. These barrier properties are due to the 2D multilayer nature of GO membranes, which creates highly tortuous, 2D diffusion paths of the molecules in between the sheets.

Furthermore, a recent work suggests that selective transport of protons is possible *through* the atomic hexagonal voids of single sheets of boron nitride [24]. Applications still more futuristic regards the use of a single sheet of graphene, capable of withstanding particular high pressures, with well-defined holes to desalinate seawater [25,26] or to sequence DNA fragments extremely fast [27].

This work describes the processing of different types of graphene onto a nickel sintered wick coupon and measure the wicking height using a standard method. We present Scanning Electron Microscope characterization of the standard nickel wick and of the wick coated with graphene. We demonstrate that the addition of graphene improves significantly the capillary pressure generated by the wick by a factor of at least 3.5, which opens up many more opportunities for LHP in aerospace and ground based applications.

2. Wick coupons integrated with graphene

We have tried two different methods of integrating graphene inside a porous wick structure made of Nickel. The first approach was to suck inside the wick coupon a graphene solution and the second method was to bond only one surface of the wick coupon with graphene oxide. The different wick coupon samples with different types of graphene are summarised in Table 1.

Fig. 1 shows the nickel wick coupon (having 12 mm diameter and 45 mm length) coated with graphene slurries only on top of the upper circular surface, that is the one where the wicking height will be measured.

2.1. Method 1: coating by vacuum suction inside the wick

Graphene was dispersed by sonication (Bandelin Sonopuls K76, Berlin, Germany, 30 min sonication at 40% amplitude) in Acetone (5 g L^{-1}) and further processed with a high pressure homogenizer at 1000 bar (GEA NIRO SOAVI NS100 1L 2K, Parma, Italy). The dispersion was then diluted to different concentrations before being poured into the setup in which the wick was fixed as in Fig. 2A. The setup was used with a Buchner funnel attached to vacuum pump. The graphene solution was sucked through the wick. Depending on the solutions concentration and the total amount of graphene used a graphene layer formed on top of the wick (Fig. 2B–D). Samples with different graphene types were produced and analyzed by SEM (ElectroScan Corp. Environmental Scanning Electron Microscope Mod. 2020, Wilmington, MA, USA). We tested two different materials for this method: (A) thermally reduced graphite oxide [28,29] (TRGO) and, (B) milled graphene with different functionalization [30,31]. TRGO was synthesised based on a modified Hummers [31,32] process, i.e. oxidation of graphite (KFL 99.5) by KMnO4 in sulfuric acid milieu as reported previously [28]. The purified GO was dried and ground and then thermally reduced by rapid heat treatment (750 °C) in a rotary tube furnace (Nabertherm, Lilienthal, Germany) under nitrogen atmosphere. The milled graphene was instead produced by milling Graphite under argon and carbon dioxide pressure using a planetary ball mill PM 100 from Retsch, Haan (Germany). The milling chamber was filled with graphite (9.3 g) and 50 ZrO2 balls (Yttrium stabilized, diameter 10 mm) both were dried in vacuum at 60 °C. The chamber was evacuated and then pressurized with argon or CO2 (13 bar), respectively. Milling was performed at 250 rpm for the duration of 48 h. Here we present only the most promising results obtained with TRGO.

2.2. Method 2: deposition of GO slurry on the wick

We used a graphene oxide slurry that was synthesised using a modified Hummers method [33–35] and investigated in different applications [36–38]. The as-prepared slurry was then deposited on top of the wicks, the surface was activated prior to the deposi-

Table 1
Graphene coated wick coupons.

Sample number	Material used	Coating method
#1	TRGO	Vacuum suction (method 1)
#2	TRGO	Vacuum suction (method 1)
#3	TRGO	Vacuum suction (method 1)
#4	GO graphenea	Slurry deposition (method 2)
#5	GO graphenea	Slurry deposition (method 2)
#6	GO graphenea	Slurry deposition (method 2)