



3D printing strong and conductive geo-polymer nanocomposite structures modified by graphene oxide



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ABSTRACT

Preparation of inks with proper rheology performance is the key for extrusion based 3D printing. In this work, extrusion based 3D printing graphene oxide (GO)/geopolymer (GOGP) nanocomposite was reported for the first time. The addition of GO in geopolymeric aqueous mixture (alumiosilicate and alkaline-source particles) dramatically changes its rheology properties, and enable the 3D printing that cannot be realized solely by geopolymer. The 3D printing GOGP structures exhibited high mechanical properties with compressive strength higher than 30 MPa, and electrical conductivity of 10^2 S/m was achieved after annealing, which is among the highest conductive ceramic nanocomposites. We expect that the rheology modification mechanism proposed in this study will facilitate the 3D printing of diverse materials, as well as the understanding of the interaction between GO and hydrophilic particles.

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1. Introduction

As first reported in 1950s, geo-polymer is an inorganic material with a non-crystal structure formed by long-range and covalently bonded atoms, mainly silicon, aluminum and oxygen [1–3]. Such chemical structure is reminiscent of long-chain structured polymers and macromolecules, and allows geo-polymers to be molded readily at room temperature, easily to be mixed with various additives to form composites with desired functionality. For this reason, geo-polymers have been employed in many different areas, ranging from fire- and heat-resistant coatings and adhesives, medical applications, high-temperature ceramics, new binders for fire-resistant fiber composites, toxic and radioactive waste encapsulation to cementing components of concrete [4–8].

In previous studies, performance characterization and related mechanisms for different combination of raw materials of geo-

polymers are mainly focused, and the investigation from structure or architecture level is still very limited [9,10]. Processing geopolymer, or geopolymer nanocomposite material, into architectures can not only make the best use of the given material in terms of mechanical property, but also provide new functions that inherited from the nanoparticles. Graphene is a very promising candidate. Indeed, the use of graphene to modify the properties of ceramics has recently attracted extensive attention [11–15]. Graphene consists of sp^2 bonded carbon atoms in a plan, and displays remarkable properties, such as high electrical conductivity and high mechanical strength and excellent chemical stability [16–18]. For applications in nanocomposites, graphene oxide (GO) is generally used as the precursor for graphene, because GO can be easily synthesized in a large quantity and reduced to graphene afterward [19–21]. More importantly, the grafted abundant hydrophilic functional groups make GO highly solubilized in water and even interact strongly with matrix materials [22,23]. For example, Walker et al. reported that the toughness of ceramic can be increased by ~235% at 1.5 vol% of graphene, due to the strong graphene/ceramic interface and the extrinsic bridge effects of graphene [24]. Ranjbar et al. investigated graphene reinforced geopolymer and improved the compressive and flexural strength of the geopolymer by 1.44 and 2.16 times, respectively, with 1 wt% of

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2. Experimental

2.1. Materials

GO was synthesized by modified Hummer's method, as described elsewhere [20]. The concentration of the obtained GO gel after dialysis is ~ 45 mg/ml. Dry Na-geopolymer powder were supplied by Institute for Advance Ceramics (Harbin Institute of Technology), with composition of $\text{SiO}_2/\text{Al}_2\text{O}_3 = 4$ and $\text{SiO}_2/\text{Na}_2\text{O} = 4$ (mole ratio). All other chemical materials are obtained from Sinopharm Chemical Reagent Co., Ltd.

2.2. Preparation of GO/Geopolymer mixture

In a typical synthesis, geopolymeric suspensions was first prepared by mixing two parts, alkaline-source particles and aluminosilicates particles (ASOPs). Alkaline-source particles are quickly dissolved in water, because of their high solubility. After stirring for 20 min, the prepared GO suspensions were injected into the ASOPs suspensions at temperature below 5°C . For the preparation of GOGP_1, GOGP_2, GOGP_3 and GOGP_4, the mass ratios of ASOPs: GO suspensions: DI water were 1:4.4:0.53, 1:2.2:0.53, 1:1.1:0.53, and 1:0.88:0.53, and the corresponding volume (weight) percentage are 4.5872 vol% (4 wt%), 5.66 vol% (5 wt%), 10.71 vol% (10 wt%),

The technology of 3D printing allows us to build complicated structures in a very efficient way, and currently, it has been investigated extensively in various areas, such as energy storage, nacre-biomimetic materials and tissue engineering [27–29]. Among all the reported 3D printing technologies, extrusion based technique is very unique, mainly due to its versatility for the selection of materials and low cost operation [30,31]. Here, for the first time, we report the extrusion based 3D printing of geopolymer/GO nanocomposite structures (Fig. 1 and video 1 in the supplementary information). We show that the addition of GO can dramatically change the rheological properties of geopolymer precursor, and enable the 3D printing of the ink based on GO/geopolymer (GOGP) mixture that cannot be realized by pure geopolymer. The GO nanosheets anchor themselves in geopolymer and encapsulate individual geopolymer grains to resist being pullout and at the same time, form a continuous 3D network throughout the whole nanocomposites. In addition, we show that highly conductive GOGP nanocomposites can be obtained after sintering.

Supplementary video related to this article can be found at

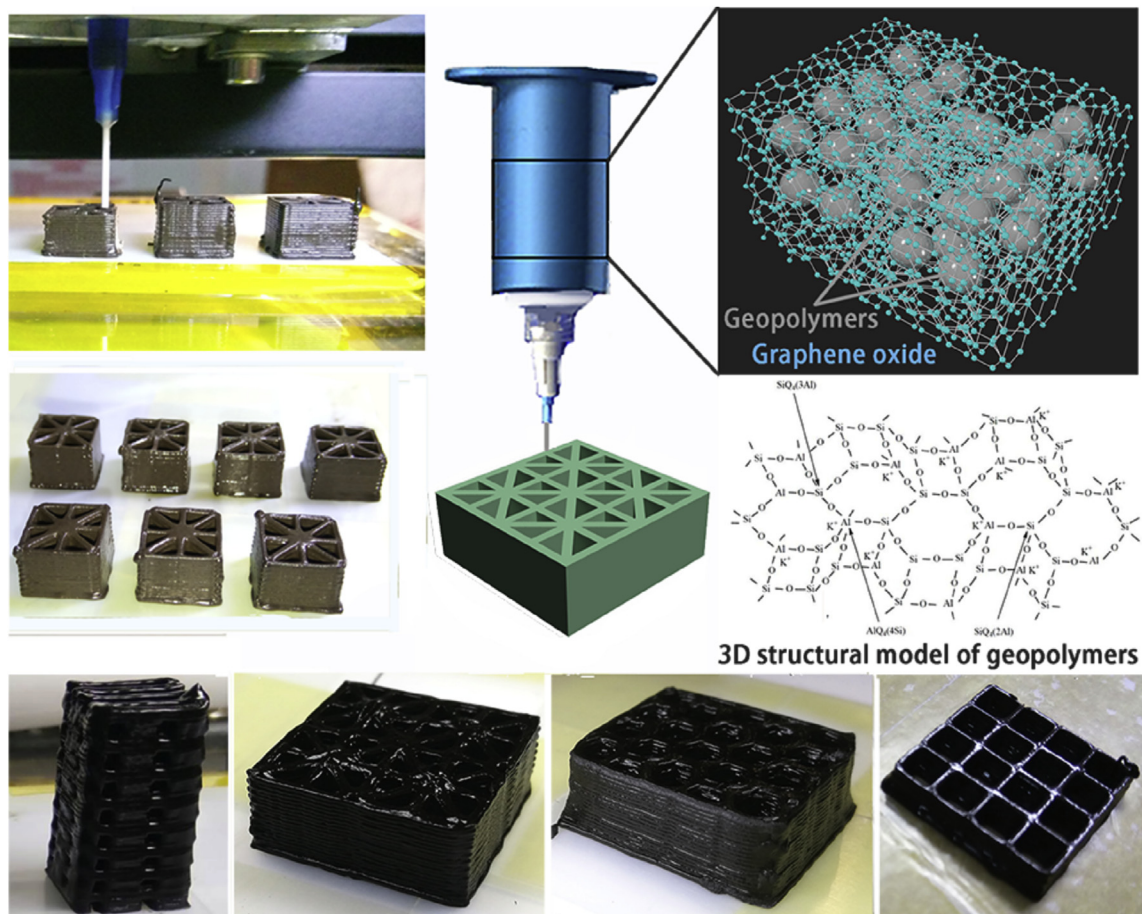


Fig. 1. Illustration of the 3D printing process and some 3D printed structures. The colors of the printed samples turn from brownish to blackish when the GO loading increased. The chemical structure of geopolymer is also showed. (A colour version of this figure can be viewed online.)

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