On the properties of new graphene nanoplatelet-silicon nitride composities consolidated by spark plasma sintering

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Silicon nitride (Si₃N₄) based materials are ceramics with a high potential for many applications owing to their interesting properties, such as the high hardness, the high temperature strengths, the good behavior under wearing conditions and the extraordinary toughness that can attain if convenient microstructures are developed [1]. Si₃N₄ is an electrical insulating material and regarding its thermal conductivity, a quite range of values can be achieved, again depending on the microstructure and the phases present in the material (α/β phase ratio) [2]. It would be valuable to explore how these properties may be altered by the addition of the new graphene nanostructures presently available in relatively large amounts. In this work we deal with the challenging preparation of silicon nitride – graphene nanoplatelets (GNPs) composites, taken special care in achieving dense materials with homogeneously dispersed GNPs. This was not an easy task and in fact some degree of platelets agglomeration was unavoidable.

To prepare the dense Si_3N_4 -GNPs composites the spark plasma sintering (SPS) technique, in short a pressure assisted sintering furnace in which the heating is provided by a pulsed direct current supplied to the graphite die that contains the powdered mixture, was used because allows very high heating rates (~100 °C/min) and very short residence times at high temperatures. Composites with a range of GNP concentrations were equally processed (3-15 wt%). The preferential orientation of the platelets (less than 60 nm thickness, Fig 1 a-b) with the a-b graphene plane perpendicular to the sintering axis was favored by the applied pressure, generating certain degree of anisotropy in the properties of the composite.

Raman spectroscopy, used to control damage of the GNPs, allowed the observation of orientation by D peak integration and made possible the identification of few layer graphene stacks in the composites. Local electrical response was investigated by conducting scanning force microscopy showing differences in conductivity between the plane and cross section surfaces of the composites, signaling further the GNP orientation effect. Preliminary thermal conductivity studies also evidenced the role of the GNPs in this property and the orientation effect.

Mechanical properties of these composites has been determined by Vickers indentation tests, In general a decrease in hardness, elastic modulus and toughness was verified, but data need to be interpreted with precaution as residual stresses in the specimens were quite large as the spall off often revealed.

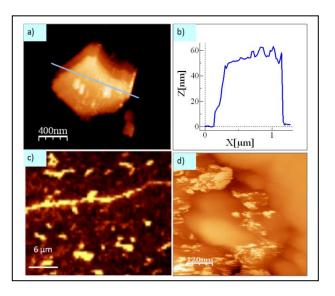


Figure 1.

a) Topography of graphene nanoplatelet and b) correspondent height profile, c) Confocal Raman image scan of a GNP-Si₃N₄ composite cross section showing platelet edges in bright color, d) Merged images of current map and topography of a Si3N4 grain surrounded by GNPs.

References

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