Development and Study of manufacturing method of few layers graphene dispersed solution for wet coating

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In the portable terminals such as the smart phone, transparent conductive film is used as a component of touch panel. The rigid and transparent conductive film is used abundantly by sputtering ITO (Indium Thin Oxide) on PET film at present. However, its conductive film may be destroyed through flexible using. Therefore, research and development of transparent conductive material with flexible mechanical characteristic substitute the ITO was actively carried out. It has the excellent potential with material substitute the ITO because the graphene has the flexible mechanical characteristic with optical transparency and electrical conduction property. In recently, a large number of synthesis of graphene have been reported; method for copying the graphene synthesized in thermal CVD on the surface of the copper foil to the PET film [1,2] and method for reducing of substrate coated graphite oxide (graphene oxide) by using solution dispersed it [3,4]. It is possible that the graphene showing comparatively few layers selectively grows by the thermal CVD on the surface of the copper foil [5]. However, following problems have been indicated, 1) the graphene of scale leaf which depends on the polycrystal texture of the copper foil is formed, 2) The graphene is selectively and heterogeneously formed in the specific crystal orientation (Cu 111 plane) in the copper foil surface, 3) it is necessary to fix the graphene of the scale leaf in acrylic resin. In addition, there is a problem of dissolving the copper foil by the acid solution in order to separate the graphene from the copper foil. And, high temperature processing at about 1000 ^{o}C under inert atmosphere is necessary in the method for coating the graphite oxide (graphene oxide) to obtain the graphene with the high crystallinity. Therefore, application to the PET film is difficult, when its technique was applied.

In this study and development, following items were examined to resolve those problems; 1) It is large production of the graphene showing few layers without catalyzing metal substrates such as the copper foil. 2) the graphene make dispersion without agglutination in the organic solvent. 3) the graphene is coated on PET film by using the wet coating process.

CVD reaction to obtain the graphene was prepared though its calcinations of raw material derived from polyester resin heat-treated moderately which adjusted remaining hydrogen content under the high-pressure and isotropic gas pressure [6]. H_2 and CH_4 generated from raw material calcined have formed the graphene without catalyst. It was possible to form the few layers graphene (graphene flower) showing flower state on circumference of raw material calcined by using gas such as H_2 and CH_4 which was generated from raw material calcined. Fig. 1 (a) and (b) show interesting morphology of its graphene flower. Fig. 2 shows TEM observation of graphene flower. The heat-treatment time necessary for obtaining 500g graphene flower lump by small-scale experimental equipment are about 4 hours. Obtained it were atomized by using the rotary mixer in 2-propanol solution. Afterwards 0.8 mg/ml solution was obtained by sonication and following centrifugal separation treatment. Similar solutions dispersed graphene flower were also obtained in 2-methoxyethanol, PGMA (Propylene glycol monomethyl ether acetat) and NMP (N-methylpyrrolidone) solvent selected through Hansen solubility parameter test. 2-methoxyethanol and 2-propanol as organic solvent with lower boiling point and high volatility were selected, and then spray and dip coating by using its solution were carried out on the copper foil or PET film having preliminary heating to obtain the graphene through transferring to the substrate. Fig. 3 shows few layers graphene on PET film. From obtaining results, since the agglomeration for graphene is so large, evaporation rate of its solvent on the substrate is slow. In the case in which its dispersed solution was oversupplied, its configuration is changed in the roll, when the graphene independently exists. In the meanwhile, it agglutinates in the graphite stacking, when it exists in multiplicity. We found that the graphene layer is independently formed on the substrate by adjusting heat-treatment temperature of the substrate, amount of the dispersed solution and supply rate of the dispersed solution. In addition, we will be discussed and examined mass-productiveness of transparent conductive film by using roll-to roll method through wet coating process with optimization of the substrate heating system and accurate control of supply of dispersed solution.

References

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Figures

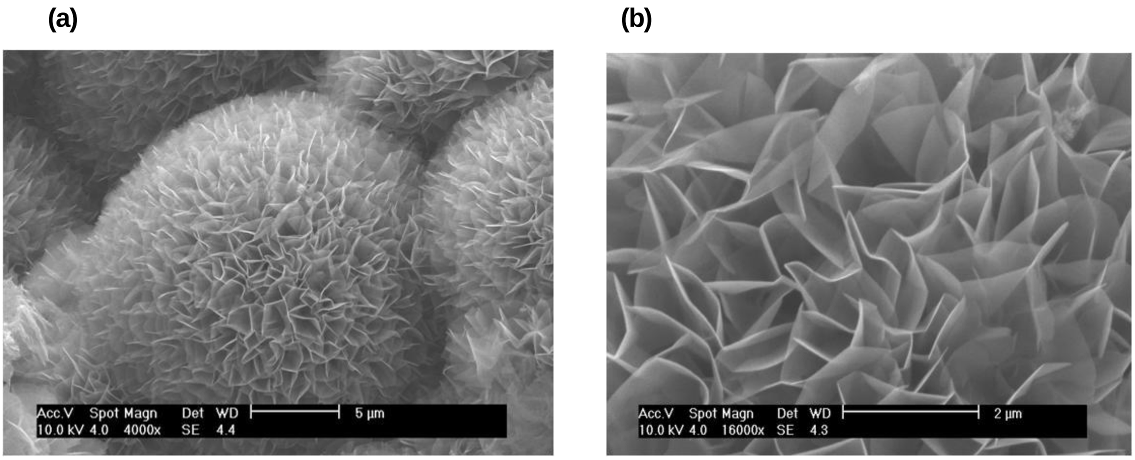

Fig. 1 Morphology of graphene flower(a) and extended one(b)

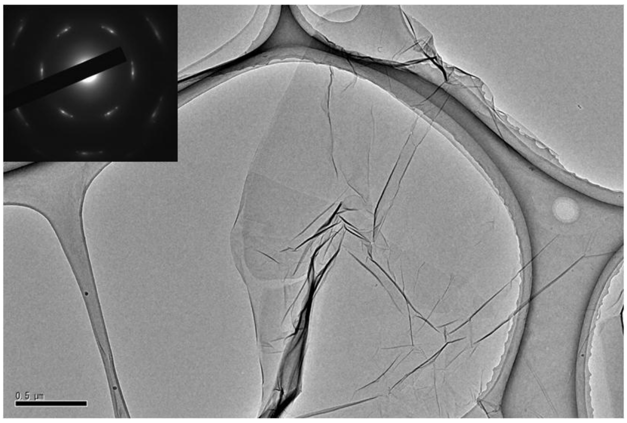

Fig. 2 TEM observation of graphene flower and SAED pattern

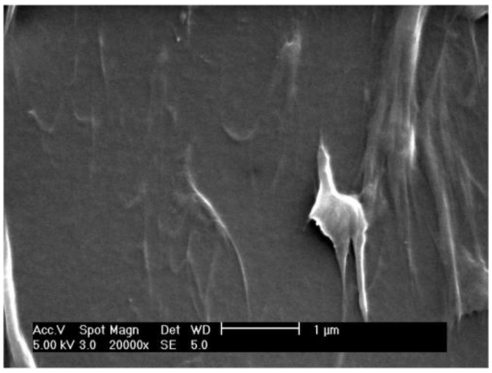

Fig. 4 Few layers graphene on PET film prepared through wet processing