





Department of Industrial Engineering

Ph.D. Course in Chemical Engineering (XII Cycle-New Series)

FROM GRAPHENE SYNTHESIS TO APPLICATIONS

Supervisor *Prof. Maria Sarno* **Ph.D. student** *Claudia Cirillo*

Scientific Referees

Prof. Paolo Ciambelli Prof. Adolfo Senatore Prof. Massimiliano Polichetti

Ph.D. Course Coordinator *Prof. Paolo Ciambelli*

Abstract

Graphene is the first two-dimensional (2D) atomic crystal available to us. Since its discovery in 2004, graphene has captured the attention and the imagination of worldwide researchers thank to its supreme properties.

Catalytic chemical vapour deposition (CCVD) is a widely employed method to synthesize large areas graphene on metal foil or to cover nanoparticles (NPs) with carbon coating. Exfoliation of graphite is largely used for the massive production of flakes of graphene.

In such light, this thesis work has been focused to develop industrial scalable processes starting from research at lab scale on graphene formation.

The first part of investigation has been addressed to fabrication of highquality graphene films on Ni foil using CVD at ambient pressure. Critical parameters including Ni thickness, cooling rate, and polycrystalline crystallographic orientation have been explored to understand the graphene formation mechanism and to obtain controlled carbon growth. We have studied the effect of operating conditions such as the synthesis time and feed composition, as well as the key role played by H_2 .

The placement of graphene on arbitrary substrates is key for applications. A study of graphene transfer from metal foil on specific support was also realized.

Subsequently, core-shell few layer-graphene-coated metal nanoparticles (GCMNP) were synthesized by CVD. Different synthesis operating conditions were investigated to achieve a good control over the coverage of GCMNPs and to understand the mechanism of GCMNP formation and carbon coverage. The reactor outlet gas was continuously monitored on-line during the catalyst activity. Several techniques were utilized to characterize the catalyst and the reaction products and to correlate their properties with the reactor operating conditions.

Magnetic properties of the core-shell few layer graphene-coated magnetic nanoparticles were also studied.

Parallel, few layer graphene oxide (GO) nanosheets were prepared by a very fast modified Hummers method and largely characterized. The tribological behaviour of GO in mineral oil was investigated under a wide spectrum of conditions.

Finally, the preparation of graphene and multilayer graphene sheets by liquid phase graphite exfoliation in N-methylpyrrolidone (NMP) was carried out. A one step massive very pure thin flakes production with an high monolayer yield was obtained.