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TEM INVESTIGATION OF DC SPUTTERED CARBON-NITRIDE-NICKEL THIN FILMS

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Deposition of carbon nitride (C-N) and carbon-nitride-nickel (C-N-Ni) films onto glass, NaCl and Si(001) substrates was carried out in a dc magnetron sputtering system. Carbon was deposited from high-purity (99.99%) pyrolytic graphite target, 50 mm in diameter, positioned at 10 cm from a resistance-heated substrate holder. C-N-Ni films were grown by a small Ni plate mounted on the graphite target. The base pressure of the deposition chamber was $\sim 7 \times 10^{-7}$ Torr. Films were grown at a substrate temperature of 20-700 °C, in pure N₂ at partial pressures of 1.9 -2.2 mTorr and the substrates were held at ground potential. The typical film thickness of 15-30 nm was deposited on all the substrates at a magnetron current of 0.2 and 0.3 A, which resulted in a deposition rate of 1.5-2 nm/s.

Structural characterizations were performed by high-resolution transmission electron microscopy (HRTEM) using a JEOL 3010 operated at 300 kV and a 200 kV Philips CM 20 electron microscope equipped with a Ge detector Noran EDS system. The N content of the C-N samples prepared at room temperature was 22-24% by EDS measurement and showed a decrease to 6-7% at elevated temperatures up to 700 °C. The N concentration in the C-N-Ni films was higher: $\sim 38\%$ at RT and $\sim 9\%$ at 700 °C. The Ni concentration of C-N-Ni samples was 5-6% and 0.3-0.4% in samples deposited at RT and 700 °C respectively. The low Ni content in the latter is attributed to a decrease of the sticking coefficient of the carbon co-deposited Ni at elevated temperatures.

All the samples were found amorphous, or amorphous-like by the selected area electron diffraction (SAED) patterns, however, according to HRTEM, the structures differed from each other:

- homogeneous amorphous structures, showing patterns on a size scale about 1 nm. This is characteristic to low temperature deposited C-N and C-N-Ni films.
- amorphous matrix with bent or curly fringe patterns arranged in more or less separated areas of 10-15 nm typical size. This was found in high temperature C-N films.
- fullerene-like structures, such as tortuous bundles of fringes arranged in onion or fingerprint-like patterns. This shows up in the C-N-Ni samples deposited at 700 °C. This we explain by the effect of the co-deposited metal species, which, can catalyze the growth of arranged carbon and carbon-nitride structures.

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