

FRACTIONATION AND CHARACTERIZATION OF Pd NANOPARTICLES IN THE Pd - C COMPOSITE FILMS OBTAINED BY PVD METHOD



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The results of fractionation and structural investigation of Pd nanoparticles in the Pd-C nanocomposite films are presented. The investigations were performed with electron microscopy methods (SEM, TEM). The films were obtained in PVD process.

Production of composite films

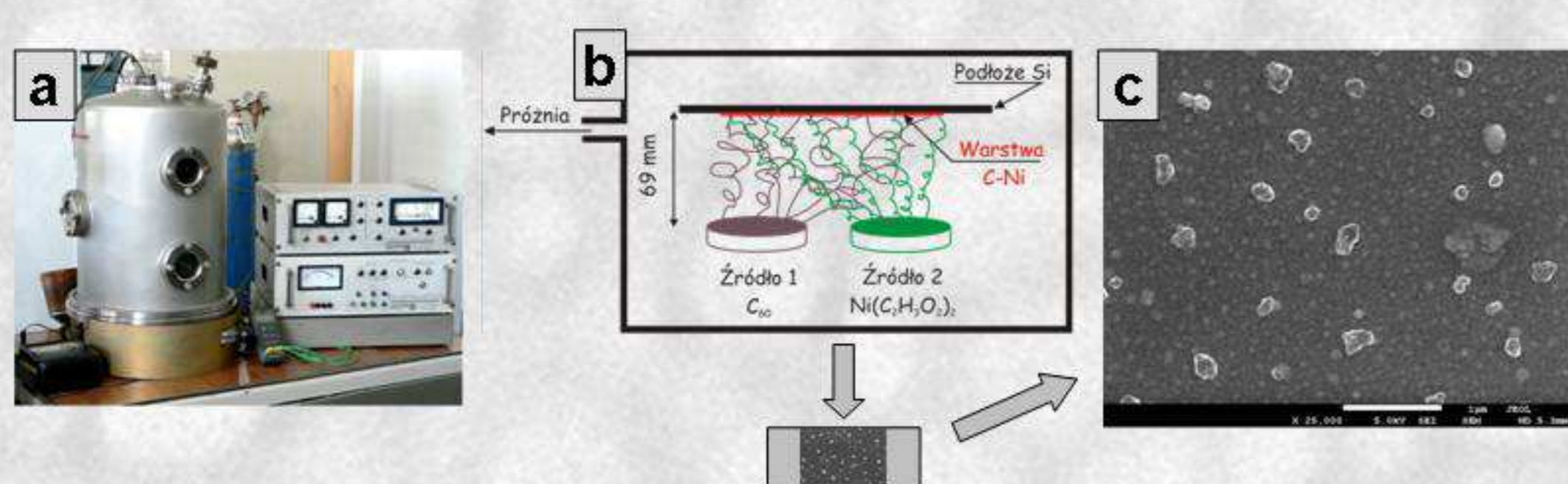


Fig. 1. Proces PVD; a) PVD set-up, b) schema, c) PVD film

The PVD process was originally elaborated in Tele & Radio Research Institute and procedure base on PVD process from two separated sources: one containing pure 99.95% fullerene C₆₀, and second source containing palladium acetate. In a result of the technological procedure we obtained Pd-C films on different substrates (Fig. 1).

Samples preparation

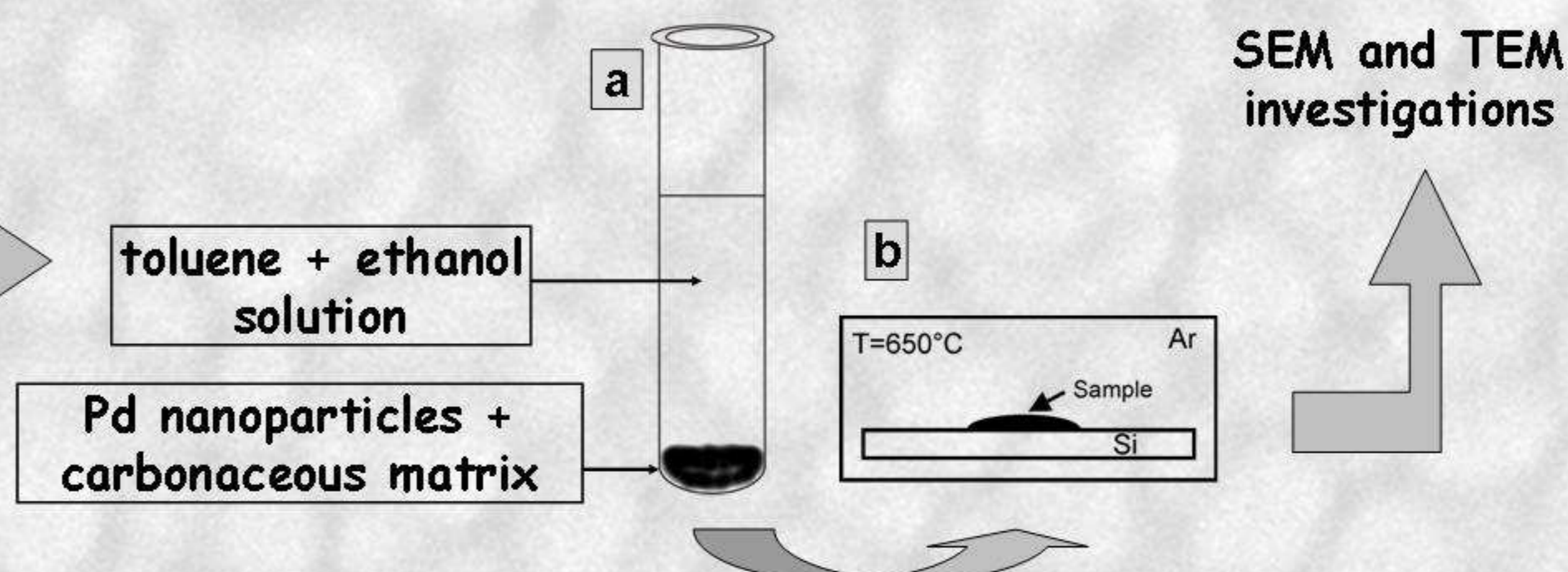


Fig. 2. a) Sedimentation process b) annealing in Ar atmosphere

This films were removed from the substrate and dispersed in toluene and ethanol solution (50/50). Subsequent fractions of Pd nanoparticles of different sizes were separated by sedimentation process. Next, the suspension was dried, deposited on silicon plate and annealed at 650°C in an atmosphere of argon for 30 minutes (Fig. 2).

SEM and TEM investigations

The size and structure of these Pd nanoparticles were studied with scanning electron microscopy (SEM)- fig. 3a, 4a, 5a and transmission electron microscopy (TEM) - fig. 3b, 4b, 5b. SEM and TEM images showed uniformly distributed Pd nanoparticles with different sizes. The analysis of the size distribution lead to conclusion, that the Pd nanoparticles can be assigned to classes (defined by the average size): small - 23nm, medium - 40nm, big - >98nm (Fig. 3c, 4c, 5c). This analysis was performed using visual impression of the distribution of size data i.e. histograms of the size of Pd nanoparticles were applied to set a mentioned classes.

HRTEM images showed polycrystalline character of Pd nanoparticles (Fig3d, 4d, 5d).

Conclusion

Sedimentation method allows for effective fractionation of the dominant Pd nanoparticles size in the Ni - C films, obtained by PVD method. According to a study of the high resolution TEM method, Pd nanoparticles have polycrystalline structure.

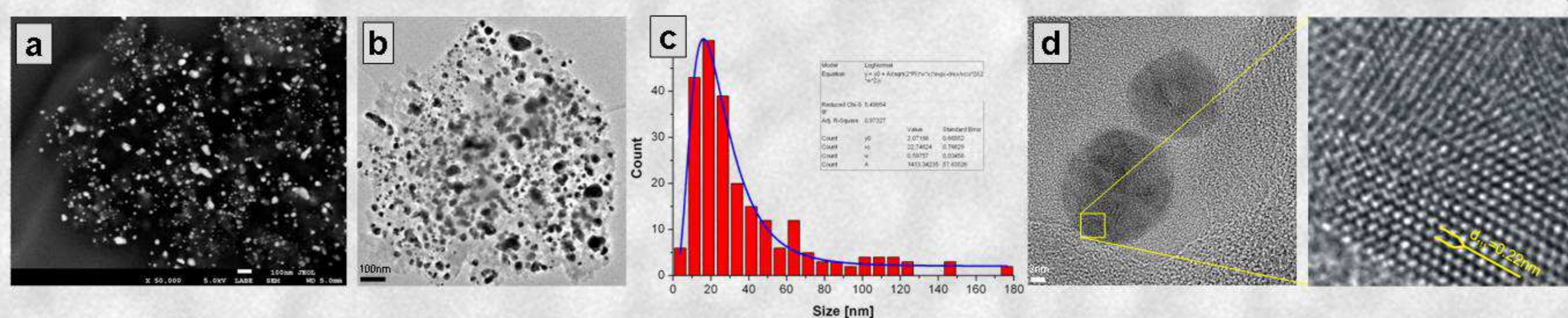


Fig. 3. Small size of Pd nanoparticles a) SEM image b) TEM image, c) histogram of Pd nanoparticles, d) structure of Pd nanoparticles

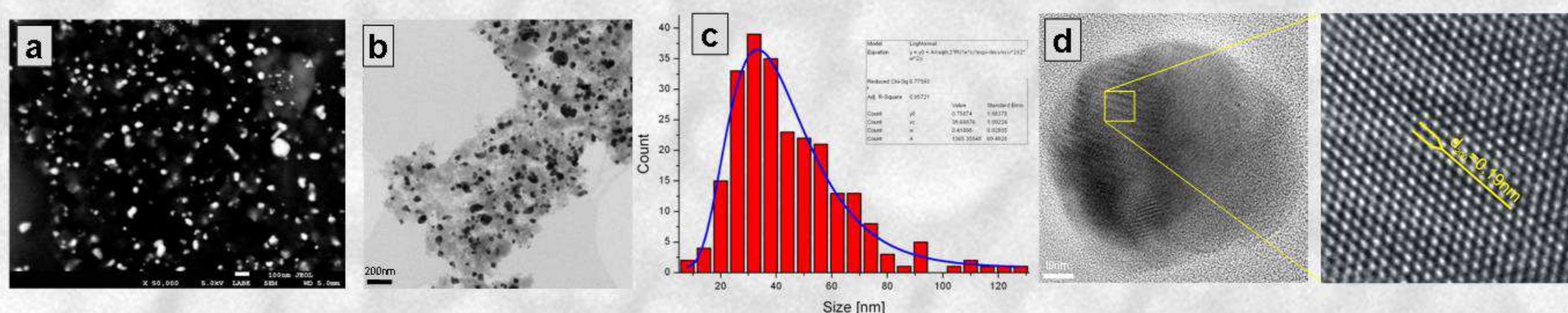


Fig. 4. Medium size of Pd nanoparticles a) SEM image b) TEM image, c) histogram of Pd nanoparticles, d) structure of Pd nanoparticles

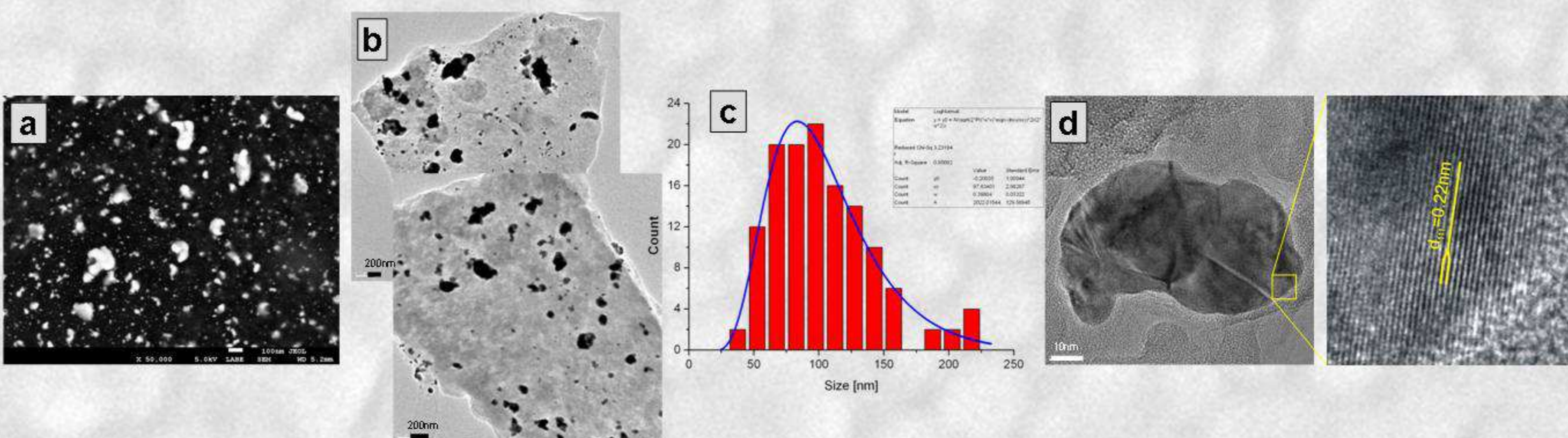


Fig. 5. Big size of Pd nanoparticles a) SEM image b) TEM image, c) histogram of Pd nanoparticles, d) structure of Pd nanoparticles