Available online at www.sciencedirect.com

ScienceDirect

Physics Procedia 83 (2016) 36 – 45

Physics Procedia

9th International Conference on Photonic Technologies - LANE 2016

# Synthesis and characterization of Pd nanoparticles by laser ablation in water using nanosecond laser

M. Boutinguiza[a], M. Meixus[a], J. del Val[a], A. Riveiro[a] R. Comesaña[b], F. Lusquiños[a], J. Pou[a,*]

[a]*Applied Physics Department, University of Vigo, EEI, Lagoas-Marcosende, 36310 Vigo, Spain*
[b]*Materials Engineering, Applied Mechanics and Construction Department, University of Vigo, EEI, 36310 Vigo, Spain*

## Abstract

Colloidal solutions of Pd nanoparticles were produced by laser ablation of solids in liquids (LASL) technique. Two different lasers operating at 1064 and 532 nm of wavelength to ablate a Pd foil submerged in water were used. The properties of the obtained nanoparticles were studied and the influence of the wavelength on the nanoparticle size was discussed. The nanoparticles formation mechanism is discussed and compared with the results obtained in previous work using continuous wave (CW) and long pulses lasers. The obtained nanoparticles were characterized by means of transmission electron microscopy (TEM), high resolution transmission electron microscopy (HRTEM) and UV/VIS absorption spectroscopy. The obtained nanoparticles consisted of crystalline Pd nanoparticles with rounded shape and strong tendency to agglomeration. The size distribution of the nanoparticle range from few nanometers to 40 nm together with the presence of occasional large particles when the infrared (IR) laser was used.

Peer-review under responsibility of the Bayerisches Laserzentrum GmbH

*Keywords:* laser ablation; palladium nanoparticles; nanosecond laser

## 1. Introduction

Nanomaterials having one or more dimensions in the range of 1-100 nm are considered a bridge between the atomic and bulk materials which is revolutionizing science, technology and industry, thanks to the phenomena related to the increased surface to volume ratio. In the last decades metal noble nanoparticles such as Au, Ag, Pd,

---

* Corresponding author. Tel.: +34-986812216 ; fax: +34-986812201 .
*E-mail address:* jpou@uvigo.es

Peer-review under responsibility of the Bayerisches Laserzentrum GmbH
doi:10.1016/j.phpro.2016.08.005

etc. have attracted attention of researchers due to their unique physical, chemical and electronical properties obtainable at nanoscale, different from that of bulk material (see Krutyakov et al. (2008), Menéndez-Manjón et al. (2011), or Boutinguiza et al. (2015)).

Pd is a transition metal with the lowest melting point and the lowest density of the platinum group of metals, showing interesting characteristics such as stable electrical properties, high resistance to wear, and exceptional catalytic properties. These properties make palladium a very appealing material being used mainly as catalyst in lots of processes (see the review by Astruc et al. (2005)). In particular the novel properties of Pd nanoparticles encouraged investigations in many different fields, among them nano-catalysis, fuel cells by the use of Pd as hydrogen storage, sensors, etc. Hamasaki et al. (2014) used Pd nanoparticles with a Co3O4 support for the formulation of aryl iodides. Nair et al. (2015) demonstrated the capabilities of Pd nanoparticles to enhance hydrogen storage when using nitrogen rich carbon material and Mijowska et al. (2015) used Pd nanoparticles in an electrochemical enzymatic biosensor to detect glucose.

There are a lot of different techniques and methods for producing Pd nanoparticles and nanostructured Pd based materials which can be classified in chemical and physical groups. Chemical reduction which typically uses reducing agents to generate nanoparticle with different sizes and composition; for instance Nguyen et al (2010) synthesized Pd nanoparticles with different shapes and sized by polyol method. Hydrothermal method is an easy and low cost technique for producing nanoparticles and enables altering the crystalline structure and the composition of the resulted particles by adjusting parameters such as temperature, pressure, or precursor concentration. Chang et al. (2009) obtained ZnO modified with various contents of Pd through hydrothermal method. However, many of these techniques use precursors and solvents, or imply chemical reactions which can contaminate the obtained nanoparticles, besides the lack of stability of the obtained product as well as low uniformity and dispersivity of the nanoparticles.

Physical techniques involve the use of precursor metals to obtain nanomaterials without the formation of new substances, but by the molecular rearrangement. Different physical techniques have been used to produce Pd films and Pd nanoparticles. Joshi et al. (2009) reported the use of Pd nanoparticles obtained by sputtering for hydrogen detection, while Slavcheva et al. (2014) reported the physical and electrochemical characterization of Pd films obtained by sputtering. Among the wide spectrum range of physical methods, LASL has emerged last years as an efficient technique in the generation of metal nanoparticles due to chief advantages like obtaining stable and dispersed nanoparticles and nanocolloids together with the absence of contaminants. Cristoforetti et al. (2012, 2013) obtained and characterized Pd nanoparticles in water and other organic solvent using pulsed laser, while Nishi et al. (2013) reported the preparation of monodispersed Pd nanoparticles at the water-air interface using pulsed laser. Marzuna et al. (2015) controlled the size of the nanoparticles by using a saline solution. In previous work we had explored synthesizing crystalline nanoparticles using continuous wave (CW) laser as well as millisecond pulsed laser, Boutinguiza et al. (2014). In the present work we report the results of an experimental work devoted to obtain Pd nanoparticles by laser ablation of a palladium target in water using nano and picosecond lasers with different wavelengths.

## 2. Materials and methods

Palladium foils with 99.99% of purity were cleaned and sonicated to be used as laser ablation targets. The foils were fixed to a bottom of a glass vessel and filled with milli-Q water up to 1 mm over the upper surface of the Pd foil (more details can be found in Boutinguiza et al. 2011a). Two different lasers sources have been used to ablate the targets. First system was a diode-pumped Nd:$YVO_4$ laser providing pulses at wavelength of 532 nm, 0,30 mJ of pulse energy and pulse duration of 10 ns; while the second laser source consisted of a picosecond diode-pumped Nd:$YVO_4$ laser delivering pulses at 1064 nm of wavelength, 800 ps of duration and 0.03 mJ of pulse energy. Processing parameters used with both lasers are listed in table 1. In all experiments laser beam was focused on the upper surface of the target to give a fluence of 0.2 to 2 J/cm$^2$, and was kept in relative movement with respect to the metallic plate at 5 mm/s of scanning speed in the case of nanosecond laser and at 20 and 40 mm/s when the picosecond laser was used These conditions enabled similar overlapping for both lasers with similar interpulse distances, 0.25 μm in the case of nanosecond laser and 0.10 and 0.20 μm for the picosecond laser. The processing