



# Synthesis of nickel nanoparticles and nanoparticles magnetic films by femtosecond laser ablation in vacuum

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## Abstract

We report on the generation of Ni nanoparticles and the synthesis of magnetic nanoparticles films by 100 femtosecond (fs) Ti:sapphire laser ablation in vacuum. The nanoparticles sizes have been studied by atomic force microscopy, showing a mean size of  $\approx 40$  nm. Vibrating sample magnetometry analysis has been performed to characterize magnetic properties of the deposited nanoparticles layers. The hysteresis loops clearly indicate that the Ni nanoparticles film behaves as a system of isolated magnetic particles. Our results evidence the potentiality of fs laser ablation, in vacuum, for the generation of nanoparticles and the deposition of nanoparticles films of magnetic materials.

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## 1. Introduction

Nanoparticles and nanoparticles magnetic films represent an interesting research area in modern nanotechnology. The magnetic properties of nanoparticles are found to be almost invariably different by their bulk counterparts of the same composition due to intimate coupling of magnetism with structure,

surface effects, and thermally activated processes [1–6]. These particles are of great scientific interest in developing a better understanding of magnetic phenomena and for applications in different fields, such as permanent magnets and magnetic recording [7,8]. Future high-density magnetic recording relies on the ability to produce fine particles with high coercivity. This is possible with nanoparticles because particles smaller than the width of a domain wall ( $\approx 50$  nm) are always in a single-domain state, with the magnetization directed along the anisotropy axis and a high coercivity above the onset of superparamagnetism (size  $> 10$  nm) [9,10].

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A variety of techniques have been applied for the preparation of nanosized particles. Among others, pulsed laser deposition (PLD) offers a versatile experimental tool with the peculiarity of transferring, under specific experimental conditions, the target stoichiometry to the deposited films [11]. PLD in the nanosecond laser pulse duration regime has been established as a flexible technique for nanoparticles production, provided that ablation occurs into a background gas [12,13]. More recently, femtosecond (fs) laser ablation from solid targets has shown its potentiality as a viable route to the generation of nanoparticles of different materials, even in high-vacuum conditions [14–16].

In this paper, we report on the synthesis of nanostructured nickel nanoaggregates by fs PLD in vacuum as well as on the analysis of their magnetic properties (coercive field, saturation field, and remanence ratio). To emphasize the peculiarities of the fs PLD nanoparticle films, we have compared their magnetic behavior with that of composite films constituted by Ni microparticles mechanically diluted into a non-magnetic silicone matrix. The main outcome of the present study is the demonstration that fs PLD in high vacuum offers the possibility to produce films of Ni nanoparticles characterized by interesting, peculiar magnetic properties.

## 2. Experimental

The experimental setup used in the present experiment was already described elsewhere [16, 17]. Briefly, the laser source is a 1 kHz Ti:sapphire oscillator–amplifier system emitting at 780 nm ( $\approx 120$  fs full width half maximum) with 1 mJ maximum energy per pulse. The laser pulse energy and the repetition rate delivered to the target were changed by means of neutral density filters and an external Pockels cell, respectively. The laser beam was focused to a spot size  $\approx 2 \times 10^{-4}$  cm<sup>2</sup> on the sample surface, at an incidence angle of 45°. The Ni target (99.9%) was mounted on a rotating holder in a vacuum chamber evacuated to a residual pressure of  $\approx 10^{-5}$  Pa. Prior to the deposition experiments, the target surface was cleaned by irradiation with a large number of laser shots, and a circular ablation track was formed on the surface.

The samples were deposited at room temperature onto mica substrates. The substrate, located about 30 mm away from the target surface, was held parallel to the Ni target and central to the laser-produced plume. By carefully selecting the number of laser shots delivered to the target, two different regimes of deposition were obtained: (a) less than one layer of Ni nanoparticles; (b) films composed of many layers of Ni nanoparticles with a typical thickness of the order of 1  $\mu$ m.

The size and shape of the nanoparticles were examined by an atomic force microscope (Digital Instruments Nanoscope IIIa) equipped with a sharpened silicon tip with a radius of less than 10 nm. The images of the surface profiles were obtained by operating the atomic force microscope (AFM) in the tapping mode, with a scan size and rate of 2  $\mu$ m and 2 Hz, respectively.

The magnetic properties of the thick Ni nanoparticles samples at room temperature were obtained from hysteresis loops recorded in a vibrating sample magnetometer (VSM, Maglab 9T, Oxford Instruments) operating at a vibration frequency of 55 Hz and at a fixed temperature of 300 K. Prior to the measurements, the nanoparticles film was reduced to a sample of 4 mm  $\times$  3 mm and accurately demagnetized. The hysteresis loops were obtained by using a field rate of 0.2 T/min up to the magnetic saturation. The magnetization curves were measured in the plane of the sample along both the longitudinal and transversal main axis leading to similar results (having taken into account the demagnetizing factors).

In order to perform a comparison with other magnetic systems, we have prepared a sample of nickel microparticles uniformly dispersed inside a non-magnetic matrix (4%, v/v). This sample was obtained by using commercial powders (99%, Sigma–Aldrich).

From the hysteresis loops, the coercive field ( $H_c$ ), the saturation field ( $H_s$ ), and the remanence ratio  $M_r/M_s$  ( $M_r$  and  $M_s$  being the remanent and saturation magnetization, respectively) were obtained for each sample.

## 3. Results and discussion

Deposition of Ni nanoparticles samples with a thickness of less than one layer was carried out to

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