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Iron and iron oxide nanoparticles obtained by ultra-short laser ablation in liquid

A. De Bonis[a,*], T. Lovaglio[a], A. Galasso[a], A. Santagata[b], R. Teghil[a]

[a] Dipartimento di Scienze, Università della Basilicata, Viale dell'Ateneo Lucano, 10 – 85100 Potenza, Italy
[b] CNR-ISM, U.O.S di Potenza, Zona Industriale di Tito, 85050 Tito Scalo (PZ), Italy

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ABSTRACT

Laser ablation of an iron target in water and acetone has been carried out using a frequency doubled Nd:glass laser source (pulse duration of 250 fs and frequency repetition rate of 10 Hz). The observation of the nanostructures formed in the laser irradiated region of the metallic target and fast shadowgraphic analysis of the laser induced cavitation bubble have been performed in order to correlate the size distribution of the obtained nanoparticles to the dynamics of the ablation process. The composition, morphology and oxidation state of the synthesized nanoproducts have been investigated by XPS (X-ray Photoelectron Spectroscopy), TEM (Transmission Electron Microscopy) and microRaman spectroscopy. The experimental data support a relationship between the nanoparticles size distribution and the femtosecond laser ablation mechanism, while the chemical and structural characteristics of the nanoparticles can be tuned by varying the liquid medium.

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1. Introduction

The scientific and technological interest for the synthesis of metal nanoparticles (NPs) is continuously increasing due to the different physical and chemical properties that nanosized materials exhibit compared to the bulk form. Among the large number of physical and chemical methods for the production of NPs, the Laser Ablation in Liquid (LAL) technique presents some advantages such as the possibility of obtaining bare NPs and tuning the physical and chemical characteristics of the obtained materials simply by varying experimental parameters such as focusing condition or the liquid medium used [1,2]. Moreover, it has been showed that using an ultra-short laser source it is possible to obtain nanoproducts in metastable phases, such as nanodiamonds [3–5]. During the ablation of a solid target in liquid a high pressure – high temperature laser induced plume expands into the liquid, perpendicular to the target surface. The interaction of the expanding plume with the surrounding liquid induces the formation of cavitation bubbles [1,6]. The collapse of the bubble generates local conditions of high pressure and high temperature inducing a second ablation effect [7–9]. Despite the experimental simpleness, the dynamics of the ablation in liquid process is not definitively clarified. In order to relate the properties of the products to the many experimental parameters, a multi-technique approach it is required.

In recent years, LAL has been widely used to obtain metallic NPs ranging from noble metals to Ti, Pd to composite systems [2,5,10,11]. Among the metallic NPs, iron and iron oxides are very interesting materials in science and technology for their applications spanning from biomedical science to catalysis, photoelectrochemical cells or magnetic devices. Amendola et al. studied the ablation of an iron target in different organic solvents using a nanosecond laser source showing the formation of different magnetic nanostructures, whose composition is influenced by the solvent [12]. More recently, Iwamoto et al. showed that when a nanosecond laser irradiates an iron or iron oxide target in a polyvinyl pirrolidone (PVP) solution, wustite (FeO) and magnetite (Fe_3O_4) NPs are formed, irrespective form the target composition [13]. On the other hand, irradiating a Fe_2O_3 powder in water, Pandey et al. showed that it is possible to enhance the oxide NPs crystallinity and relative crystalline size [14]. However, magnetic iron oxide NPs present hydrophobic surfaces which push towards their agglomeration and large clusters are formed. In order to avoid this drawback, the use of cationic and anionic surfactants is needed.

The laser ablation of iron in different liquids has been widely reported, using nanosecond or laser sources [12–14]. In this paper we present, for the first time to our knowledge, the dynamics of ultra-short ablation of an iron target immerged in distilled water and acetone, with the general purpose to increase the understanding of the role of the solvent in the ultrashort ablation process and in

* Corresponding author. Tel.: +39 0971206249.
E-mail address: angela.debonis@unibas.it (A. De Bonis).

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the NPs formation. Moreover, well dispersed iron oxide NPs have been obtained and their physical-chemical properties have been carefully investigated.

2. Experimental

The laser ablation process was performed by an ultra-short Nd:glass pulsed laser source ($\lambda = 527$ nm, $\tau = 250$ fs, 10 Hz repetition rate). The laser beam was focused perpendicularly to the surface of the metal target by a 40 mm focal length plano-convex lens mounted on a micrometric stage. The laser energy reaching the target surface was 3 mJ, for a fluence of 15 J/cm^2. The Fe target (Aldrich, purity 99.9%, 0.5 mm tick), contained in a 2 cm × 5 cm quartz cuvette, was covered by a 18 mm liquid column in order to avoid the evaporative explosion of the liquid surface layer which could occur at the focusing conditions here reported. Bidistilled milliQ water and acetone (99% Aldrich) have been used as liquid media.

Fast shadowgraphic set-up used to characterize the cavitation bubbles has been described elsewhere [7]. Scanning Electron Microscopy images were acquired by a Jeol - JSM-7100F instrument.

High resolution TEM (HRTEM) Fei-TECNAI G2 20 TWIN, operating at 200 kV was used to study both the dimension and distribution of the NPs obtained by laser ablation. To this aim, some drops of a sample solution were placed on a holey carbon coated copper grid.

XPS spectra were acquired by a Theta Probe spectrometer using monochromatized MgKα radiation. The acquired XPS spectra were analyzed using an home-made curve-fitting program, Googly, described by Castle et al. [15], which allows the simultaneous fitting of peaks in the form of a Voigt function and their associated background in a wide energy range.

Micro-Raman measurements were performed using a HORIBA LabRam 800 HR apparatus equipped with a edge filter, a He–Ne laser ($\lambda = 632.8$ nm) and an Olympus microscope (objectives: 10x/50x/100x). The laser spot size impinging on the samples surface was about 10 μm in diameter when the 100x microscope objective was used. A spectral resolution of about 4 cm^{-1} was obtained by a holographic grating with 600 lines/mm.

3. Results and discussion

3.1. Ablation mechanism

During the laser ablation of a solid target in liquid a large amount of energy is released on the solid in a very short time scale. As a consequence, a rapid increase of pressure and temperature at the solid liquid interface can be expected. The emission of an intense pressure wave is the most efficient mechanism path followed by the target for releasing energy. The laser induced plasma acts like a piston against the liquid environment, generating a shock wave (SW) that, generally, expands at a velocity comparable with the speed of sound through the liquid [6,7,16]. A SW front velocity of 1198 and 1445 m/s has been evaluated during the ablation of the iron target in acetone and water, respectively. Behind the SW front, the fast pressure gradient and the heat-exchange of the laser induced plasma plume with the surrounding liquid induce the formation of a cavitation bubble, that contains vapor, gases and NPs [1]. The clusters present in the bubble can interact each other in a condensation process giving origin to NPs with the polycrystalline structure.

The bubble, formed about one microsecond after the laser shot, expands reaching its maximum radius and finally collapse. After the collapse, the rebound of the bubble can be usually observed.

In Fig. 1 the evolution with time of the radius of the bubbles obtained during the ablation of iron in water and acetone is reported. Since the cavitation bubble dynamics is dependent from the density of the liquid medium [7], the bubble in acetone is characterized by larger radius and lifetime (580 μm; 95 μs) than in water (440 μm; 60 μs). Moreover, the rebound of the bubble has been observed only when the ablation is carried in acetone. It has been showed that the temporal evolution of cavitation bubble oscillations induced by ultra-short laser ablation of metallic target in liquid is in good agreement with the well-known Rayleigh-Plesset model [17]. Nevertheless, the maximum radius reached by the bubbles during its further oscillation periods is lower than the predicted one [7]. It has been proposed that this effect could be related to a not full adiabatic dynamics of the bubble with a dissipation of energy during its shrinking phase. Consequently, it can be expected that an amount of energy could be released to the metallic target surface during the collapsing process [7].

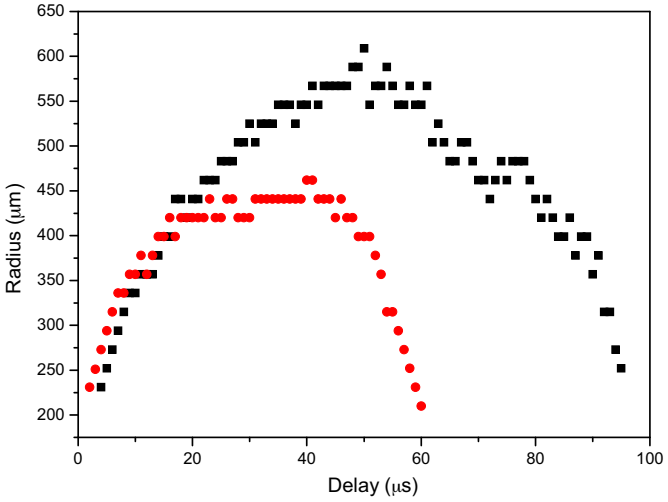

Fig. 1. Temporal evolution of the radius of the first CB produced by ablation of a Fe target in water (●) and acetone (■).

3.2. Nanoparticles characterization

High resolution TEM, microRaman and XPS analysis allow to fully characterize the products obtained by LAL in the two different solvents. In Fig. 2a and b TEM micrographs of the NPs produced in both solvents and the respective size distributions are reported. All NPs are characterized by a spherical shape and exhibit a bimodal size distributions, as already observed for metallic NPs obtained by ultra-short LAL [7,8]. In particular the two distributions evaluated for NPs obtained in water and in acetone have a mean diameter of 9 and 25 nm and of 11 and 29 nm, respectively.

3.3. NPs produced in acetone

TEM images, reported in Fig. 3a and b, show that when the ablation is performed in acetone, a core-shell structure is often evident. The inter-planar distance observed for the shells surrounding the nanoparticles has been evaluated to be of 0.35 nm, in good agreement with the (2 0 0) distance in bulk graphite [12]. Where the graphitic shell is present, it has a thickness of less than 10 nm. The core of the nanoparticles presents crystalline domains where the interplanar distance is of 0.25 nm that solely corresponds to the distance of the (1 1 0) planes in α-Fe_2O_3 [18,19]. Since Raman spectroscopy is useful to differentiate various iron oxides, this technique has been used to characterize our NPs and the presence of hematite (α-Fe_2O_3) has been proved. In the Raman spectrum reported in Fig. 3 c signals at 231, 291, 403, 604 and 1301 cm^{-1}, related to hematite, are evident [18,20]. The wide band between

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