

Metals and metal oxides particles produced by pulsed laser ablation under high vacuum

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Received: 13.04.2010

Abstract

A pulsed KrF Excimer laser (248 nm, 15 mJ) was utilized to synthesize different particles on (111) Si substrate under vacuum ~ 10^{-6} torr using Aluminum (Al), Platinum (Pt), Tungsten (W), Molybdenum (Mo), Cadmium Oxide (CdO), and Yttrium Oxide (Y₂O₃) targets. Scanning electron microscope (SEM) was used to study particle size distribution on silicon substrate. The size of individual metal particles was found to be ranging from 71 nm to 2 μ m whereas metal oxides particles were found in the range of 71 nm to 1.1 μ m. The particles below 100 nm are formed by gas phase condensation of ions, atoms and molecules whereas particles greater than 100 nm size results either from the ejection of liquid droplets from the melt surface or stresses induced by laser on target surface. The advantage of particle formation due to laser ablation in high vacuum is pure particles production.

Key Words: Laser ablation, nanoparticles, high vacuum

1. Introduction

Pulsed lasers are typically used for laser ablation of metals. Pulsed laser irradiation can heat metal and metal oxides to melting or even boiling point [1] to form the particles. Formation of particles under laser ablation of solids in vaccum, gas and liquid environments has been extensively investigated [2–7]. The particles generally are found to be between nanometers to several tens of micrometers in size.

The nanoparticles (NP) have unusual optical, electronic and chemical properties that are very attractive for many applications like biotechnology, chemical catalysts, electronic and optical devices [8–16]. NP with dimensions less than 30 nm are particularly suited for these applications because of their chemical stability and presence of a resonant surface plasmon excitation in the visible spectra [17, 18]. In particular, these nanoparticles can be used as selective optical makers to diagnose different biological objects. A large variety of chemical methods are being used in the preparation of NP [19] but laser ablation is one of the preferable techniques. One advantage of laser ablation compared to other conventional methods for preparing NP is the absence of other chemical reagents. It has recently been demonstrated that laser ablation in liquids is a

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promising technique to obtain metal colloids [20, 21]. But NPs have a strong tendency to react with liquid environment to form different compounds like oxides, hydroxides and nitrides etc depending upon the type of solvent. Therefore, this problem can be largely circumvented when the laser ablation of metals is performed under high vacuum.

In the present work, we report the production and size distribution of Aluminum (Al), Platinum (Pt), Tungsten (W), Molybdenum (Mo), Cadmium Oxide (CdO), and Yttrium Oxide (Y_2O_3) particles under high vacuum using a high power excimer laser.

2. Experimental set up

Figure 1 shows the schematic of experimental setup. Pulsed Laser Ablation (PLA) has been used to deposit particles of metal and metal oxides targets (Al, Pt, W, Mo, CdO and Y_2O_3) on silicon substrate. Excimer Laser operating at wavelength 248 nm, repetition rate 20 Hz, pulse length 20 ns and pulse energy 15 mJ, was used to ablate the target materials in ablation chamber at a vacuum $\sim 10^{-6}$ torr. Laser beam was focused onto the surface of rotating target at an angle of incidence 45° and 5000 laser pulses were used for target ablation. Ablated material was deposited on silicon substrate placed 10 mm away and parallel to the target at room temperature. The size distribution of particles from different target materials is studied using a scanning electron microscope.



Figure 1. Schematic view of the experimental setup.

3. Results

In this experiment six targets Aluminum, Platinum, Tungsten, Molybdenum, Cadmium Oxide and Yttrium Oxide are used to produce particles by Pulsed Laser Ablation (PLA) on silicon substrate. SEM analyses of produced particles were performed and size of the particles was determined. The size of these particles ranges from few nanometers to microns. There are also some particles, which agglomerate to form clusters.



Figure 2. SEM images and respective bar graphs of Al, Pt, W, and Mo particles formed on silicon substrate.

Figure 2 shows SEM micrographs and their respective bar graphs of particle size distribution of different metals embedded in silicon substrate. Figure 2(a) shows the SEM micrograph and bar graph of Aluminum particles embedded in silicon substrate. The size of these small particles has been measured by dividing the whole SEM micrograph in five portions. Square areas from four corners and square area from the middle are selected so that four equally squared portions cover whole micrograph. The particle size from all these areas is calculated. Almost homogeneous distributions of different particle sizes are found in all these portions. The particle ranges from 76 nm to 2 μ m and maximum density at 600 nm. The bar graph in Figure 2(a) shows the number of particles in different sizes. This figure shows that maximum numbers of particles are between 300 nm to 700 nm. The interesting feature of this micrograph is the formation of particles under 100 nm.

Figure 2(b) shows the SEM micrograph and respective bar graph of Platinum particles embedded in silicon substrate. The overall view of this image shows that particle size ranges from 76 nm to 1.8 μ m with maximum density at 100 nm. The cluster formation is also observed. The interesting feature of this micrograph is the formation of particles of size ~76 nm also. Figure 2(c) shows the micrograph of tungsten particles and respective bar graph. Tungsten particles are distributed uniformly on the whole silicon surface. There is also some cluster formation. The particle size ranges from 100 nm to 2 μ m with maximum density at 800 nm. The bar graph of W particles shows that there is maximum concentration of particles in range of 0.7–0.9 μ m. Figure 2d shows the SEM micrograph and respective bar graph of Mo particles deposited on the silicon surface. The particle size ranges from 75 nm to 2 μ m with maximum density at 1 μ m. There are only a few big particles of 2 μ m size. There is also some cluster formation due to agglomeration of particles. The bar graph of Mo-particles shows that maximum numbers of particles have range of 0.2 μ m. There are only a few numbers of particles above 1.2 μ m.

Figure 3a shows the SEM micrograph and respective bar graph of CdO particles embedded in silicon substrate. The SEM image reveals some circular and most visible particles. This Figure shows that particles range in size from 71 nm to 1.1 μ m with maximum density at 400 nm. The particles under 100 nm are also formed on silicon surface. Figure 3b shows that nano to micro-sized particles of Y₂O₃ and respective bar graph.

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The particle size ranges from 71 nm to 1.1 μ m with maximum density at 300 nm. Some spherical particles are also observed in this micrograph. The bar graph of yttrium oxide particles shows that there are maximum numbers of particles in range of 0.3 μ m-0.5 μ m. There are only a few numbers of particles above 0.5 μ m.



Figure 3. SEM images and respective bar graphs of CdO and Y_2O_3 particles formed on silicon substrate.

4. Discussion

When the laser light of suitable intensity is focused on target surface, material vaporization takes place and dense vapor plume is formed which moves away from the target surface. The vapor is comprised of ions, electron, atoms, molecules, clusters, and electromagnetic radiations. The expansion of vapor beyond the Knudsen layer is described by an adiabatically expanding gas. The ionic, atomic, and molecular species forms particles of size ≤ 100 nm by diffusion upon condensing on the substrate surface. Irregular shaped particles are formed due to laser induced thermal and mechanical stresses at the metallic target surface. The laser also induces thermal and mechanical stresses on and inside the material surface, which leads to exfoliation of the material. This exfoliation is responsible for submicron to micron sized cluster formation, which also reaches to the substrate surface. The appearance of circular shaped particles in case of metal oxides ablation is due to molten material ejected out from the melted surface of the target due to recoil pressure. Hydrodynamic instabilities are responsible for the formation of such molten globules on the substrate surface. The maximum size of metal oxide particles was found to be smaller (1.1 μ m) than metal particles (2 μ m). It is attributed to the fact that the oxides have higher absorption coefficient than metals. Therefore, the metal oxides absorb higher laser energy and can be converted into comparatively smaller particles than metals.

5. Conclusions

The particles from nanometers to micrometers range have been successfully fabricated by pulse laser ablation of metals and metal oxides on silicon surface under vacuum. The Al particle size ranges from 76 nm to 2 μ m with maximum density at 600 nm. The Platinum particles size ranges from 76 nm to 1.8 μ m with maximum density at 100 nm. The tungsten particle size varies from 100 nm to 2 μ m with maximum density at 800 nm whereas Molybdenum particles size ranges from 75 nm to 2 μ m with maximum density at 1 μ m. The Cadmium Oxide particle size ranges from 71 nm to 1.1 μ m with maximum density at 400 nm whereas yttrium oxide particles size ranges also from 71 nm to 1.1 μ m but maximum density of particles at 300 nm. The formation and shape of the particles are described by the different phenomena during laser ablation.

Acknowledgement

Thanks to Prof. Dr. Shahzad Naseem, Director of Center for Solid State Physics, University of Punjab, Lahore, Pakistan for scanning electron microscopy analysis.

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