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مشخصه یابی نانو ذرات تانتالوم تولید شده توسط فرایند کند و سوز لیزری با پهنای پالس نانو ثانیه در محلول اتیلن گلیکول

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چکیده – نانو ذرات تانتالوم طی کند و سوز با لیزر *Nd:YAG* به طول موج *nm* ۱۰۶۴ و فرکانس *Hz* ۱۰ ، در محلول اتیلن گلیکول تولید شدند. سپس خصوصیات آنها توسط آنالیزهای طیف سنجی فوق بنفش- مرئی، میکروسکوپ الکترونی عبوری، میکروسکوپ الکترونی روبشی، پراش اشعه ایکس و طیف سنجی رامان تعیین شد. متوسط اندازه نانو ذرات حدود *nm* ۱۴ برآورد شد. طبق مطالعات طیف سنجی فرابنفش - مرئی، مکان پیک نانو ذرات تانتالوم در محدوده طول موج *nm* ۲۱۵ مشاهده شد. خصوصیات کریستالی و ساختار فازی این ذرات نیز توسط پراش اشعه ایکس بررسی گردید. بعلاوه، به منظور شناسایی گونه های مولکولی جذب شده روی سطح نانو ذرات، طیف سنجی رامان انجام شد.

کلید واژه- تشدید پلاسمون های سطحی، طیف سنجی رامان، کند و سوز لیزری، نانو ذرات تانتالوم

Generation and characterization of Ta nanoparticles by Nanosecond laser ablation in Ethylene glycol

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Abstract- Tantalum nanoparticles (Ta NPs) were prepared in an ethylene glycol (EG) solution by ablation with a 1064 nm Nd:YAG operating at 10 Hz. Fabricated NPs were characterized by UV-visible spectroscopy, Transmission electron microscopy, Scanning electron microscopy, X-ray diffraction, and Raman spectroscopy. The average sizes of the NPs were estimated to be in the range of 14 nm. From the UV-visible studies, the plasmon peak position of the Ta NPs was observed in the spectral range of 215 nm. The crystalline nature and phase structure of the Ta NPs were investigated using X-ray diffraction. Furthermore, for the detection and identification of molecular species absorbed at NPs surface, Raman spectroscopy analysis was performed.

Keywords: Laser Ablation, Raman spectroscopy, Surface plasmon resonance, Tantalum nanoparticles

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Generation and characterization of Ta nanoparticles by Nanosecond laser ablation in Ethylene glycol

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1 Introduction

Metal nanoparticles (NPs) are very interesting since they possess characteristics, which is a connection between the bulk material and atomic structures. They have the size and shape dependent properties, which makes them totally favorable for many applications [1]. Different synthesis techniques for metallic colloids have been reported. Among them, Liquid Phase-Pulsed Laser Ablation (LP-PLA) is a new and highly promising technique for preparing nano-structured material. This technique is based on the generation of a plasma-plume due to irradiation of the solid surface immersed in a liquid with a highintensity laser beam. The plasma-plume contains neutral atoms, ions, and electrons from the target solid [2].
The aim of this work is the synthesis of Ta NPs by LP-PLA of Ta in EG and that the products have potential applications in regenerative medicine, dynamic random-access memory devices, capacitor, gas sensors, etc. Therefore, it is important to find methods to produce Ta NPs with improved properties.

2 Experimental Section

A rectangular Ta metal plate (1mm thickness, 99.9% in purity) was placed into a Pyrex container, which was then filled with 25 ml of ethylene glycol (EG) ($C_2H_6O_2$, MerckGermany) solution, as a solvent. The liquid depth was selected 7 mm, above the metal plate. The laser pulse was perpendicularly aligned to the tantalum plate surface, and it was ablated by Neodymium: Ytterbium Aluminum Garnet (Nd:YAG) nanosecond laser source (Q-PLUS model from Spectrum. A. T. N. Ltd). The first (1064 nm) harmonic wavelength, pulse width of 7 ns, repetition rates of 10 Hz, and laser shots number of 6000 pulses was utilized to ablate the Ta plate. A plano-convex lens with an 80 mm focal length was applied to focus the beam to a point approximately 2 mm in diameter on the Ta plate. In addition, the spot size of the laser beam on tantalum target surface was calculated to be 40 μm. Furthermore, the ablation process was typically carried out for 10 minutes at room temperature. The samples were prepared with laser fluence (LF) of 1.2 J/cm^2. The absorption spectra of produced Ta NPs were immediately characterized by a UV–Vis-NIR absorption spectrophotometer (Varian cary 500 scan) operated in the range of 175-3300 nm. All spectra were measured at room-temperature in a quartz cell with a 10 mm optical path. Phase recognition was performed using an STOE X-ray diffractometer with Cu Kα radiation (λ = 1.54060 Å). The suspensions were dropped onto Si substrates and dried for X-ray diffraction (XRD) measurement. The shape and size distribution of NPs were studied by Transmission electron microscope (TEM, Philips-EM 208S) operating at 100 kV. Scanning electron microscopy (SEM) investigations were performed with a KYKY-EM 3200 microscope. The Raman spectra were obtained using an FT-Raman system (Thermo Nicolet 960 USA). The 633 nm line from a 1 mW HeNe laser was used as the excitation source.

3 Results and Discussion

Figure 1 shows the experimental ultra-violet visible (UV–vis) absorption spectrum of colloidal solution of Ta NPs. It is significant to note that the peak position and shape of these spectra can be related to the particle size, shape, solvent type, etc.
The spectrum shows two distinct peaks in the UV region, one centered at about 215 nm and the other at 359 nm. We suggest that the first absorption peak corresponds to the surface

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plasmon absorption of Ta NPs, which is the characteristic absorption of metals. The second peak (the weak peak) at 359 nm is more difficult to assign.

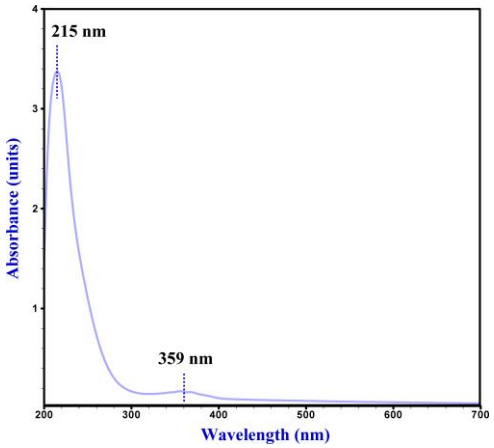


Figure 1: UV-vis absorption spectra of Ta NPs.

The polycrystalline structures of the PLAL-derived NPs are revealed by XRD patterns, as presented in Fig. 2.

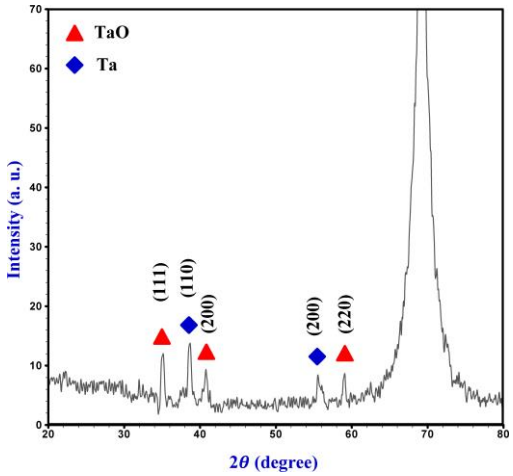


Figure 2: X-ray diffraction pattern of the laser fabricated Ta NPs in EG.

By comparing diffraction peaks with the standard cards (JCPDS file No. 02–1104 and No. 65–2903), the phase structures of Ta and TaO (Tantalum Oxide) can be determined, respectively. The peaks located at 35°, 41.1°, and 59.3° represent the characteristic reflections (111), (200), and (220) of TaO, respectively. In addition, the diffraction peaks at 38.6°, and 55.6° correspond to the (110), and (200) lattice planes of cubic structured Ta, respectively. It should be noted that an intense peak at about 70° corresponds to the (400) lattice plane of the silicon substrate. Figure 3 presents the TEM image and the corresponding size distribution histogram of Ta NPs synthesized in EG. It should be noted that the most of NPs are solid spheres. The histogram of NPs is measured by counting 400 particles in TEM images. The average size of them in the distribution is about 14 nm.

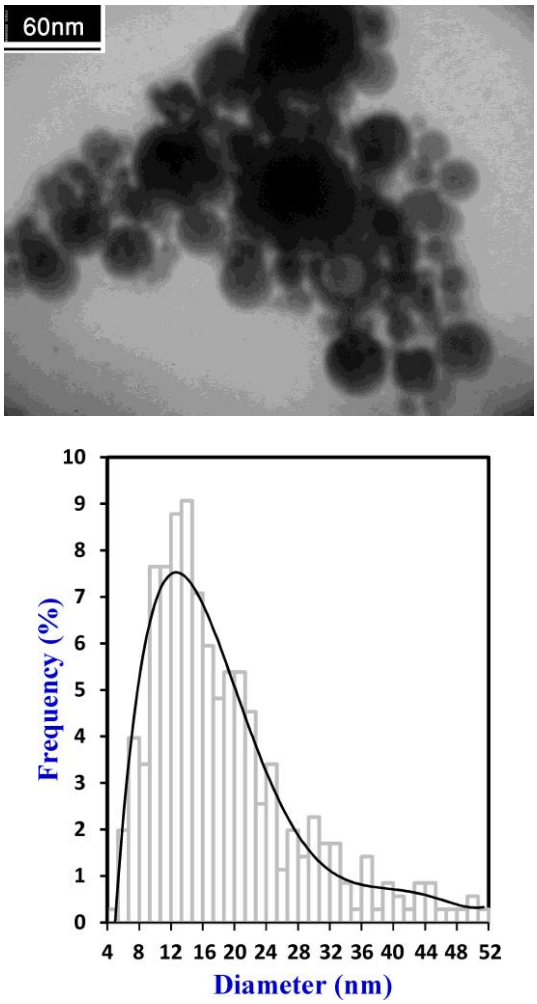


Figure 3: TEM image of Ta NPs with the corresponding particle size distribution.

Figure 4 depicts the typical SEM images of the products. A large portion of sphere-like NPs have diameters less than 50 nm. We also observed particles with sizes larger than 50 nm, which seems to be aggregated from small NPs (in the region indicated by the arrow). Only particles that were non-agglomerated were taken into consideration and the sizes of at least 400 particles were measured by microstructure measurement software. Hence, the aggregated NPs are not included in the size distribution histogram. The mean size of these particles are in agreement with TEM results.

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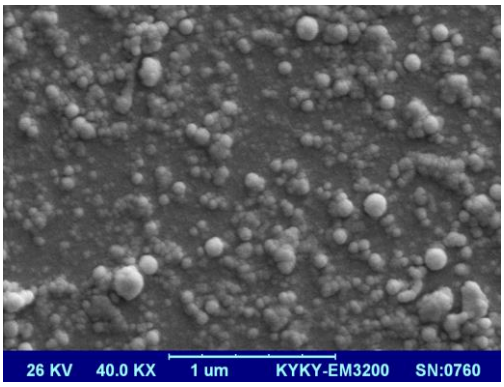


Figure 4: SEM image of synthesized Ta NPs.

Figure 5 shows a typical Raman spectrum of EG chemisorption on Ta NPs surface in the region of 3700–400 cm^{-1}. According to the figure, there are various bands, correlated with different species adsorbed at tantalum-EG solution interface. The intense band at 3346.4 cm^{-1} is generally assigned to the O-H (hydroxyl) stretching vibration [υ(O-H)] of EG molecule assembled on the Ta NPs surface [3]. It is found that the peak around 2921.4 cm^{-1} could be attributed to the C-H asymmetric vibration [υ_{as}(C-H)] adsorbed onto Ta NPs [3, 4]. It is noteworthy that the peak located at 2502.8 cm^{-1} belong to the combination of CH_2 bending and C-O stretching modes [$\delta(CH_2)+\upsilon$(C-O)].

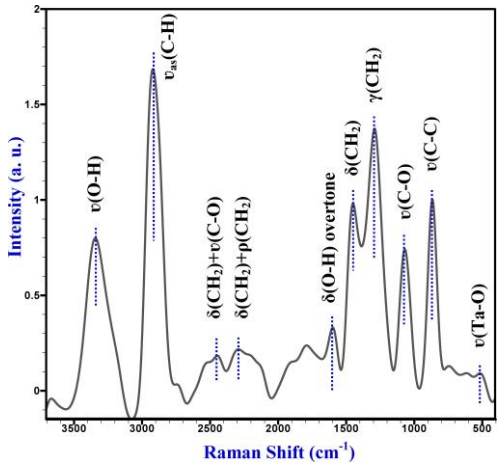


Figure 5: Raman spectrum of Ta NPs in EG.

The band around 2300.1 and 1600.3 cm^{-1}, are due to the combination of CH_2 bending + CH_2 rocking [$\delta(CH_2)+\rho(CH_2)$], and O-H bending modes, respectively [3, 5]. It is of interest to notice that the expected band for the CH_2 bending vibration [$\delta(CH_2)$] is observed at 1449.9 cm^{-1}, while the peak at 1292.9 cm^{-1} give information about the CH_2 wagging vibrations [$\gamma(CH_2)$] of the alcohol groups bound to Ta. The Raman signal at 1070.6 cm^{-1} can be assigned to the stretching mode of C–O bond [υ(C-O)], whereas that at 867.8 cm^{-1} corresponds to the stretch of C-C [υ(C-C)] adsorbed onto Ta NPs surface [3]. In the particular case of the metal-oxygen bond, the band in the low wavenumber spectral ranges of 521.2 cm^{-1} is consistent with the stretching vibration of Ta-O [υ(Ta-O)], which is evidence that the tantalum oxide NPs are formed [4].

4 Conclusions

Tantalum NPs with a size of less than 60 nm were produced by the ablation of a tantalum target in EG at LF value of 1.2 J/cm^2. We characterized the NPs using optical absorption spectroscopy, electron microscopy, X-ray diffraction, and Raman spectroscopy. The produced NPs represented a plasmon resonance peak in the UV spectral range of about 215 nm. The samples exhibited to be crystalline NPs with cubic structures. By Raman spectroscopy measurements, the different atom groups on the NPs surface were determined. We believe that pulse laser ablation in liquid media is a good approach for synthesizing specialized NPs, which is difficult to fabricate by conventional methods.

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