

Retraction Notice

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History

Expression of Concern:

☐ yes, date: yyyy-mm-dd**X** no

Correction:

☐ yes, date: yyyy-mm-dd**X** no**Comment:**

The substantial portions of the text came from T. Theivasanthi et al., "Electrolytic Synthesis and Characterization of Silver Nanopowder".

This article has been retracted to straighten the academic record. In making this decision the Editorial Board follows [COPE's Retraction Guidelines](#). Aim is to promote the circulation of scientific research by offering an ideal research publication platform with due consideration of internationally accepted standards on publication ethics. The Editorial Board would like to extend its sincere apologies for any inconvenience this retraction may have caused.

Guiding this retraction: The ANP Editorial Office

Extensive Studies on X-Ray Diffraction of Green Synthesized Silver Nanoparticles

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Abstract

Silver nanoparticle preparation and X-ray diffraction studies are reported in this paper. Metallic nanoparticles are traditionally synthesized by wet chemical techniques, where the chemicals used are quite often toxic and flammable. This paper deals with a cost effective and environment friendly technique for green synthesis of silver nanoparticles from silver nitrate solution by co-precipitation using the leaf extract of different species of Ocimum which acts as reducing and capping agent. The important ingredients responsible for the formation of silver nanoparticles present in the leaf extract are triterpenes, flavonoids and eugenol. Wide range of experimental conditions has been adopted in this process and its X-ray diffraction characterizations have been studied. The average crystalline size was 12 nm. The particle size and strain which were calculated using Williamson-Hall equation were 12.3 and 0.3688 respectively. The dislocation density was $7.9 \times 10^{14} \text{ m}^{-2}$.

Keywords

Silver Nanoparticles, Green Synthesis, Ocimum, X-Ray Diffraction

1. Introduction

In the recent research preparation of nanoparticles and the study of it are given much importance [1]-[3]. It is their physical and chemical properties that are attracting the present science field when compared with the bulk Materials [4]. Optical, electronic, magnetic and catalytic characters of metal nanoparticles are depending on their

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size, shape and chemical environs [2] [3]. The control particle size, particle shape and morphology are very much important in nanoparticle preparation. The most important tool to study the nano materials is X-ray diffraction. In the present study we discuss about the simple and low cost preparation of silver nanoparticles and XRD studies. Silver nanoparticles with particle size 25 nm are prepared in room temperature and the results are confirmed by XRD. A variety of methods have been adopted to make nanoparticles on solid surfaces, including diverse lithographic techniques, deposition of a metal colloid, controlled nanoparticles growth by diffusion, vacuum deposition of metal, electrophoretic chemical and electrochemical deposition of metal nanoparticle, etc. [5]-[8]. For uniform size, shape and spacing of nanoparticles, there are various techniques like Lithographic and vacuum deposition of metal, but expensive techniques. One of the suitable, simplest and low-cost methods is co-precipitation method which can be used in wide range of materials.

In this present paper we are explaining the formation of silver nanoparticles through green method. We are also investigating various parameters like stress, strain, peak indexing, d-spacing, instrumental broadening, specific surface area, crystallinity index and dislocation density from XRD data. We are also trying to show that our XRD results are correlated with TEM results.

2. Experimental Details

2.1. Silver Nanoparticle Preparation

10 ml of *Ocimum sanctum* leaf extract was taken in a burette and 100 ml of aqueous solution of 10 mM silver nitrate was taken in a beaker. Beaker was placed on a magnetic stirrer and the titration was carried out by drop wise addition of leaf extract to silver nitrate (AgNO_3) solution for reduction into Ag^+ ions. The mixture is thoroughly mixed for about 15 minutes and incubated at room temperature for 5 hours. Color changes were observed from watery to yellowish brown which represents the formation of silver.

2.2. Collection of Silver Nanoparticles

The silver nanoparticle obtained solution was purified by centrifugation at 4000 rpm for 30 min. The supernatant was transferred to a clean dry beaker for further settlement of particles and repeated centrifugation was carried to purify silver nanoparticles (AgNPs). The pellet so obtained was dried in an incubator. The particles obtained were stored for further characterizations.

2.3. X-Ray Diffraction Studies

In order to examine the physico-chemical make-up of unknown materials, the mineralogists and solid state chemists use primarily the Powder X-ray Diffraction techniques which are the most important characterization tools used in solid state chemistry and material science. The size, shape, lattice parameter determination and phase fraction analysis of the unit cell for any compound can be determined easily by XRD. The information of translational symmetry-size and shape of the unit cell are obtained from peak positions of Diffraction pattern.

3. Results and Discussions

3.1. Peak Indexing

From the peak positioning the unit cell dimensions are determined this process is called indexing which is the primary step in diffraction pattern analysis. Miller indices (hkl) are necessary to be assigned for each peak to index. It is not mere simple reverse of calculating peak positions from the unit cell dimensions and wavelength [9]. XRD analysis of the prepared sample of silver nanoparticles was done by a Bruker D8 advanced X-ray diffractometer using $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$), under 40 kV/30Ma-X-ray, $2\theta/\theta$ Scanning mode, Fixed Monochromator). Data was taken for the 2θ range of 30 to 80 degrees with a step of 0.02 degree. Data for some 2θ range has each peak was assigned in first step. Diffractogram of the entire data is in Figure 1.

Indexing has been done in two different methods and data are in Table 1 and Table 2. In Table 1, one need to find a dividing constant and values in the 3rd column becomes integers (approximately). Here, the constant is 35 ($140 - 105 = 35$). Moreover, the high intense peak for cubic materials is generally (111) reflection, which is observed in the sample. Four peaks at 2θ values of 38.048, 44.133, 64.303, and 77.326 deg corresponding to (111),

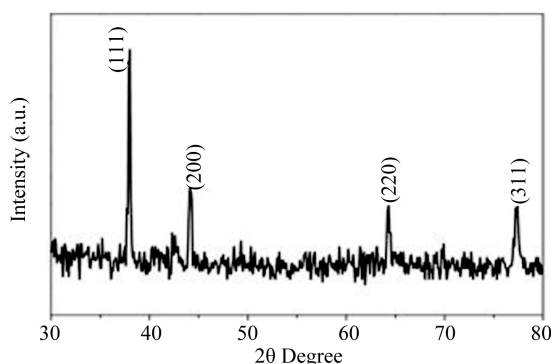


Figure 1. X-Ray diffraction patterns of silver nanoparticles.

Table 1. Simple peak indexing.

Peak position 2θ	$1000 \times \sin^2\theta$	$1000 \times \sin^2\theta/35$	Reflection	Remarks
38.0	105	3	(111)	$1^2 + 1^2 + 1^2 = 3$
44.1	140	4	(200)	$2^2 + 0^2 + 0^2 = 4$
64.3	282	8	(220)	$2^2 + 2^2 + 0^2 = 8$
77.3	389	11	(311)	$3^2 + 1^2 + 1^2 = 11$

Table 2. Peak indexing from d-spacing.

2θ	d	$1000 \times d^2$	$1000 \times d^2/59.69$	hkl
38.04	2.3631	179.07	3	(111)
44.13	2.0465	238.76	4	(200)
64.30	1.4474	477.33	8	(220)
77.32	1.2333	657.44	11	(311)

(200), (220) and (311) plane of silver were observed and compared with the standard powder diffraction card of JCPDS, silver file No. 04-0783. The XRD study confirms that the resultant particles are (FCC) silver nanoparticles [10]. Table 3 shows the experimentally obtained X-ray diffraction angle (2θ) and the standard diffraction angle (2θ) of silver particles are in agreement [11]. The ratio between the intensities of the (200) and (111) diffraction peaks and (220) and (111) peaks enumerated in Table 4 is also slightly higher than the conventional value (0.41 versus 0.31) and (0.34 versus 0.22) [12].

3.2. Particle Size Calculation

From this study, considering the peak at degree, average particle size has been estimated by using Debye-Scherrer formula [13]-[15].

$$D = 0.9\lambda / \beta \cos\theta \quad (1)$$

where " λ " is wave length of X-ray (0.1541 nm), " β " is FWHM (full width at half maximum), " θ " is the diffraction angle and " D " is particle diameter size.

$$1) 2\theta = 38.04$$

$$\beta = (38.303 - 37.804) \times 3.14 / 180 = 0.0087 \text{ radians}$$

$$D = 0.9 \times 0.1541 / 0.0087 \times \cos 19.02$$

$$D = 16.8 \text{ nm}$$

$$2) 2\theta = 44.13$$

$$\beta = (44.401 - 43.792) \times 3.14 / 180 = 0.0106 \text{ radians}$$

$$D = 0.9 \times 0.1541 / 0.0106 \times \cos 22.06$$

$$D = 14 \text{ nm}$$

Table 3. Experimental and standard diffraction angles of AgNPs.

Experimental diffraction angle (2θ in degrees)	Standard diffraction angle (2θ in degrees) JCPDS silver: 04-0783
38.048	38.116
44.133	44.277
64.303	64.426
77.326	77.472

Table 4. Ration between the intensity of the diffraction peaks.

Diffraction peaks	Sample value	Conventional value
(200) and (111)	0.41	0.31
(220) and (111)	0.34	0.22

3) $2\theta = 46.30$

$$\beta = (64.403 - 64.001) \times 3.14 / 180 = 0.0070 \text{ radians}$$

$$D = 0.9 \times 0.1541 / 0.0070 \times \cos 32.15$$

$$D = 23.35 \text{ nm}$$

4) $2\theta = 77.32$

$$\beta = (78.000 - 77.103) \times 3.14 / 180 = 0.0156 \text{ radians}$$

$$D = 0.9 \times 0.1541 / 0.0156 \times \cos 38.65$$

$$D = 11.28 \text{ nm}$$

The particle size is less than 30 nm and the details are in **Table 5**. Williamson-Hall equation is another method to calculate particle size and strain. The Williamson-Hall equation is expressed as follows

$$\beta \cos \theta = (k\lambda/D) + 2\varepsilon \sin \theta \quad (2)$$

where β is the full width at half maximum (FWHM) peak, k is Scherrer constant, λ the wave length of the X-ray, D is the crystalline size, ε the lattice strain and θ the Bragg angle. $\beta \cos \theta$ is plotted against $2 \sin \theta$ using a linear extrapolation to this plot where the intercept gives the particle size ($k\lambda/t$) and slope gives the strain (ε).

3.3. Calculation of d-Spacing

The value of d (the interplanar spacing between the atoms) is calculated using Bragg's Law

$$2d \sin \theta = n\lambda \quad (3)$$

$$d = \lambda / 2 \sin \theta \quad (n = 1)$$

Wavelength $\lambda = 1.5148 \text{ \AA}$ for $\text{CuK}\alpha$

1) $2\theta = 38.04$

$$2\theta = 19.02$$

$$d = 0.1541 / 2 \times \sin 19.02$$

$$d = 0.2363 \text{ nm (2.3638 \AA)}$$

2) $2\theta = 44.13$

$$2\theta = 22.06$$

$$d = 0.1541 / 2 \times \sin 22.06$$

$$d = 0.20509 \text{ nm (2.0509 \AA)}$$

3) $2\theta = 64.30$

$$2\theta = 32.15$$

$$d = 0.1541 / 2 \times \sin 32.15$$

$$d = 0.14478 \text{ nm (1.4478 \AA)}$$

4) $2\theta = 77.32$

$$2\theta = 38.66$$

$$d = 0.1541 / 2 \times \sin 38.66$$

$$d = 0.12333 \text{ nm (1.2333 \AA)}$$

Table 5. The grain size of silver nanoparticle.

2 θ of the intense peak (deg)	hkl	θ of the intense peak (deg)	FWHM of intense peak (β) radians	Size of the particle (D) nm	d-spacing nm
38.04	(111)	19.02	0.0087	16.8	0.2363
44.13	(200)	22.06	0.0106	14	0.2050
64.30	(220)	32.15	0.0070	23.3	0.1447
77.32	(311)	38.66	0.0156	11.2	0.1233

The calculated d-spacing details are in [Table 5](#).

3.4. Calculation for Expected 2 θ Positions

The FCC crystal structure of silver has unit cell edge “a” = 4.0857 Å and this value is Calculated theoretically by using formula,

$$a = \frac{4}{\sqrt{2}} \times r \quad (4)$$

For silver $r = 144$ pm. Following formulas are used in the calculation of the expected 2 θ positions of the first four peaks in the diffraction pattern and the inter planar spacing d for each peak.

$$\frac{1}{d^2} = \frac{(h^2 + k^2 + l^2)}{a^2} \quad (5)$$

Bragg's Law is used to determine the 2 θ value: $\lambda = 2d_{hkl} \sin \theta_{hkl}$

1) hkl = 111

$$1/d^2 = (1^2 + 1^2 + 1^2) / (4.0857 \text{ Å})^2$$

$$d = 2.3588 \text{ Å}$$

$$\sin \theta_{hkl} = 1.54 \text{ Å} / \{2(2.3588 \text{ Å})\} \rightarrow \theta = 19.2^\circ \quad (2\theta = 38.0^\circ)$$

2) hkl = 200

$$1/d^2 = (2^2 + 0^2 + 0^2) / (4.0857 \text{ Å})^2$$

$$d = 2.0425 \text{ Å}$$

$$\sin \theta_{hkl} = 1.54 \text{ Å} / \{2(2.0425 \text{ Å})\} \rightarrow \theta = 22.05^\circ \quad (2\theta = 44.1^\circ)$$

3) hkl = 220

$$1/d^2 = (2^2 + 2^2 + 0^2) / (4.0857 \text{ Å})^2$$

$$d = 1.4444 \text{ Å}$$

$$\sin \theta_{hkl} = 1.54 \text{ Å} / \{2(1.4444 \text{ Å})\} \rightarrow \theta = 32.1^\circ \quad (2\theta = 64.2^\circ)$$

4) hkl = 311

$$1/d^2 = (3^2 + 1^2 + 1^2) / (4.0857 \text{ Å})^2$$

$$d = 1.2318 \text{ Å}$$

$$\sin \theta_{hkl} = 1.54 \text{ Å} / \{2(1.2318 \text{ Å})\} \rightarrow \theta = 38.6^\circ \quad (2\theta = 77.3^\circ)$$

The expected 2 θ values are very close with the experimental 2 θ values mentioned in [Table 5](#).

3.5. XRD-Instrumental Broadening

A considerable broadening in X-ray diffraction lines will occur when particle size is less than 100 nm. The broadening is due to the particle size and strain from diffraction pattern. This broadening is used to calculate the average particle size. The sample and the instrument lead to the total broadening of the diffraction peak. The sample broadening is described by

$$FW(S) \times \cos \theta = k\lambda / \text{size} + 4 \times \text{Strain} \times 2\varepsilon \sin \theta \quad (6)$$

The total broadening β_t is given by the equation

$$\beta_t^2 \approx \left\{ \frac{0.9\lambda}{D \cos \theta} \right\}^2 + \{4\varepsilon \tan \theta\}^2 + \beta_0^2 \quad (7)$$

where ε and β_0 are the strain and instrumental broadening respectively. Using least squares method average particle size D and the strain of the experimentally observed broadening of the peaks are calculated. The instrumental broadening is presented in **Figure 2**. A method for deconvoluting size and strain broadening was proposed by Williamson and Hall by looking at the peak width as a function of 2θ . $\sin \theta$ on the x-axis and $\cos \theta$ on the y-axis (in radians) a Williamson-Hall plot is plotted. A linear fit is drawn to get the data. From y-intercept and slope particle size and strain are extracted respectively. The particle size is 12.3 nm and strain is 0.3688. **Figure 3** shows Williamson Hall Plot.

Line broadening analysis is one of the most accurate methods as the broadening affects the particle size at least twice the contribution due to instrumental broadening. Therefore the size range is calculated with this technique will lead to most accurate results.

3.6. Specific Surface Area

The important parameters such as particle size, shape and density are related to the specific surface area measurements ($\text{m}^2 \cdot \text{g}^{-1}$). Using Brumauer Emmete Teller (BET) equation the specific surface area of silver nanoparticle are measured [13]-[16].

$$S = \frac{6 \times 10^3}{D_p \cdot \rho} \quad (8)$$

where D_p is the size of the particles, S is the specific surface area, and ρ is the density of silver 10.5 g/cm^3 [17]. Using these formulas SSA is calculated and for the prepared silver nanoparticle is $46.457 \text{ m}^2/\text{gm}$. The particle size is comparable with the crystalline size which is calculated in Debye-Scherrer and Williamson-Hall plot methods. These values are given in **Table 6**.

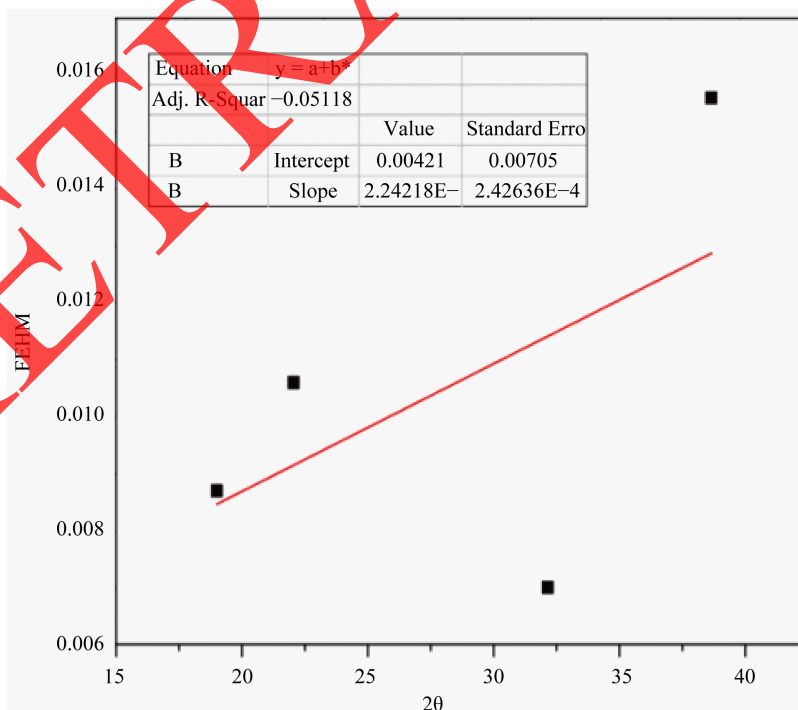


Figure 2. Typical Instrumental Broadening. $y = -0.0005696x + 0.00204$.

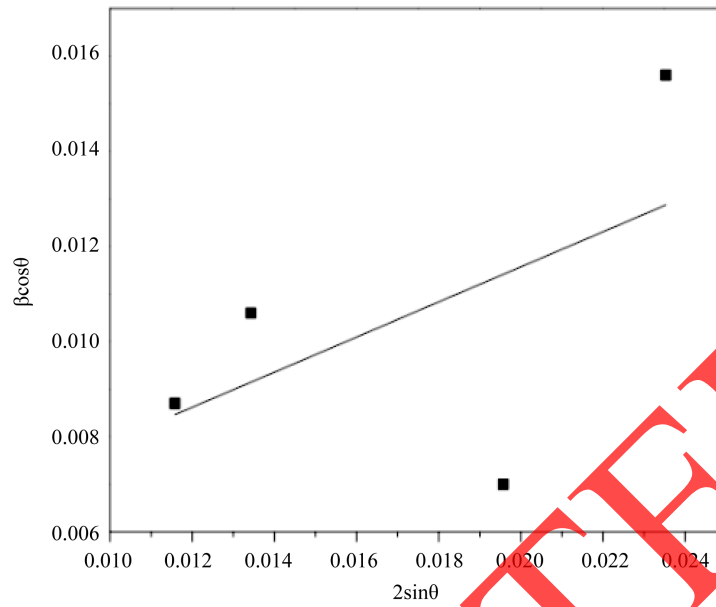


Figure 3. Williamson hall plot is indicating line broadening value due to equipment.

Table 6. Crystalline size calculated from XRD, Particle size calculated from specific surface area.

Average crystalline size calculated from XRD (nm)	Particle size calculated from Williamson-Hall plot (nm)	Particle size calculated from specific surface area (nm)
12.8	12.3	12.2

3.7. Crystallinity Index

The peak breadth of a specific phase of material is directly proportional to the mean crystallite size of that material. Crystallite materials are typically indicated by the sharper XRD peaks are. In XRD data of silver nanoparticles a peak broadening was observed. Using the Scherrer equation the average particle size calculated was 12.8 nm. By comparing the crystallite size as ascertained by SEM particle size crystallinity of the sample is evaluated. Crystallinity index Equation was presented below:

$$I_{cry} = \frac{D_p(TEM, SEM)}{D_{cry}(XRD)} (I_{cry} \geq 1.00) \quad (9)$$

where I_{cry} is the crystallinity index, D_p is the particle size (obtained from either TEM or SEM morphological analysis, which is shown in Figure 4 and Figure 5), D_{cry} is the particle size (calculated from the Scherrer equation).

3.8. Dislocation Density

A dislocation within a crystal structure could be a crystallographic defect, or irregularity. The properties of materials can be influenced by the presence of dislocations which are a type of topological defects mathematically. The intrinsic stress and dislocation density of silver nanoparticles are determined by the X-ray line profile analysis. It is found that intrinsic stress and dislocation density of silver nanoparticles are 0.275 GPa and $7.0 \times 10^{14} \text{ m}^{-2}$ respectively [18]. The dislocation density (δ) in the sample has been determined using expression [19].

$$\delta = \frac{15\beta\cos\theta}{4aD} \quad (10)$$

where δ is dislocation density. It is calculated from broadening of diffraction line measured at half of its maximum intensity (radian), θ Bragg's diffraction angle (degree), a lattice constant (nm) and D particle size (nm).

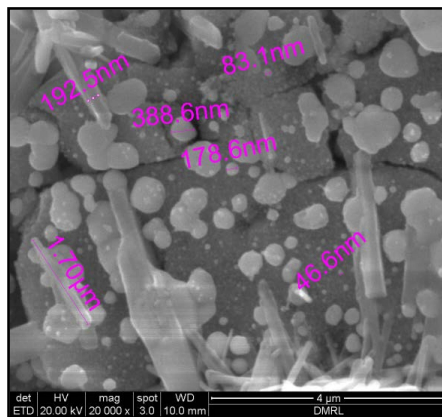


Figure 4. SEM image of silver nanoparticle.

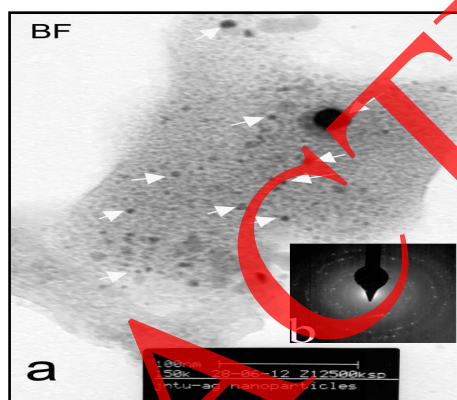


Figure 5. (a) TEM image of silver nanoparticle; (b) SAED Pattern of silver nanoparticle.

The dislocation density of sample silver nanoparticles is $7.9 \times 10^{14} \text{ m}^{-2}$.

3.9. Unit Cell Parameters

The calculated Unit cell parameters from XRD are given in **Table 7**.

Silver nanoparticles have already been studied extensively due to their potential technological applications in various fields like catalysis, lubricants, electronics etc. X-ray diffraction is one the most important characterization tool used in nano material research field. Silver nanoparticles have been successfully prepared by Co-precipitation method in normal room temperature using Ocimum leaf extract and their structural characterizations have been studied by X-ray diffraction. The resulted silver nanoparticles are less than 20 nm. Four peaks at 2θ values of 38.04, 44.13, 64.30 and 77.32 degrees corresponding to (111), (200), (220) and (311) planes of silver have been observed. These values are compared with the JCPDS, silver file No. 04-0783. The XRD study confirm that the resultant particles are (FCC) silver nanoparticles.

4. Conclusion

The reduction of metal ions occurs due to the presence of leaf extract, thus it leads to the formation of silver nanoparticles with well-defined dimensions. The synthesized silver nanoparticles have been characterized and confirmed by XRD. Different parameters like stress, strain, peak indexing, d-spacing, instrumental broadening, specific surface area, crystallinity index, dislocation density and all other unit cell parameters were studied using XRD. The average crystalline size was calculated to be 12 nm and the crystalline structure had been confirmed to FCC. The obtained silver nanoparticles from leaf extract were well matching with theoretical and experimental 2θ positions and d-spacing values.

Table 7. XRD parameters of silver nanoparticle.

Parameters	Values
Structure	FCC
Space group	Fm-3m (space group number: 225)
Point group	m3m
Packing fraction	0.74
Symmetry of lattice	cubic close-packed
Particle size	12.8 nm
Bond angle	$\alpha = \beta = \gamma = 90^\circ$
Lattice parameters	$a = b = c = 4.0857 \text{ \AA}$
Vol. unit cell (V)	68.20 \AA^3
Radius of atom	144 pm
Density (ρ)	10.5 g/cm
Dislocation density	$7.9 \times 10^{14} \text{ m}^{-2}$
Mass	107.8682 amu

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