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Microstructural investigation of alumina implanted with 30 keV nitrogen ions

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Abstract

Among ceramics, alumina is being widely used as biomaterials now these days. It is being used as hip joints, tooth roots etc. Ion implantation has been employed to modify its surface without changing it bulk properties. 30 keV nitrogen with varying ion dose ranging from 5×10^{15} ions/cm^2 to 5×10^{17} ions/cm^2 is implanted in alumina. Surface morphology has been studied with optical microscope and atomic force microscope (AFM). Improvement in brittleness has been observed with the increase in ion dose. Compound formation and changes in grain size have been studied using X-Ray diffraction (XRD). AlN compound formation is also observed by Fourier transform infrared spectroscopy (FTIR). The change in the grain size is related with the nanohardness and Hall–Petch relationship is verified.

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1. Introduction

It is a most important challenge to make a material which can replace the ill or damaged organs of the human body. Certain compositions of ceramics, glasses, glass ceramics and composites have shown bone like bonding. Not only as biomaterials, ceramics are being widely used as heat and electrical resistant materials, in integrated circuits, in reactor, as catalyst, automobiles, aerospace, and have enormous application in day to day life. Ceramic materials for medical applications have emerged as a new topic of research since few years. However alumina (Al_2O_3) and zirconia (ZrO_2) are two types known as inert, and that was the main reason why they began to be employed in implant manufacturing.

Ceramic materials have superior mechanical properties, heat resistance and chemical stability, compared to those of metals. Ceramic materials have been considered as the candidate for biomaterials used for tooth root, artificial bone etc. [1]. Metallic materials such as stainless steels, cobalt–chromium alloys and titanium and its alloys are being used for ortho-prostheses [2]. Ti–Al alloys are being used as orthopedic implant devices [3]. In this case, nitrogen implantation increases the corrosion resistance of Ti-alloys and thus enhancing the life of body-implants. [4]. Corrosion resistance has been increased by nitrogen ion implantation of as-cast Ti–6Al–7Nb alloys [5]. But metals and alloys are much heavier compared to the bone. This causes

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stress shielding, remodeling of bone and failure of total joint due to strain mismatch. Therefore ceramics may be the better candidate for biocompatibility. Looking onto the fact, Qing Zhao et al. [6] have modified the Al_2O_3 surface by NH_2^+ ion implantation and have found better biocompatibility with animal bone tissue than the plain ceramic surface. Nanohardness of polycrystalline α-alumina after Zr^+, Cr^+, Ti^+, Ni^+ ion implantation is investigated by Halitim et al. [7]. The samples were implanted to a dose of 10^{17} ions/cm^2. Radiation induced defects have been found to be responsible for the much hardening in Al_2O_3. We have chosen nitrogen ion because AlN compound formation is expected to form after nitrogen implantation. Very few people have studied the formation of AlN on the alumina surface by ion implantation. AlN has recently received a great deal of attention as a dielectric material combining some very interesting properties such as high thermal conductivity, thermal stability, high hardness, piezoelectricity, high ultrasonic velocity and oxidation resistance. AlN could also be a suitable insulator or passivation film for semiconductor components. Synthesis of AlN by chemical vapor deposition and its characterization using FTIR and XRD is described by Wang et al. [8]. The FTIR spectrum shows Al–N bonds at 670 cm^{-1}. The objective of the present work was to improve the surface properties of alumina without changing its bulk properties by nitrogen implantation. The biocompatibility behaviors will be published elsewhere. In this paper we will describe the change in microstructural and mechanical properties after nitrogen ion implantation.

2. Experimental

Commercially available optically polished polycrystalline α-alumina substrates were cut into 1 cm × 1 cm size and were cleaned at room temperature in acetone ultrasonic bath. The cleaned samples were implanted using 30 keV N^+ ions at various doses ranging from 5×10^{15} ions/cm^2, 5×10^{17} ions/cm^2. The nitrogen ion range, ionization due to ion collision and vacancies produced were theoretically investigated with the software stopping and range of ions in matter (SRIM) 2003.

The ion implanter used in this work is an electromagnetic isotope separator modified suitably as an ion implanter and is located at the Department of Physics, Mumbai University. It has pre-analysis configuration with a single step acceleration of ions at 30 kV before mass analysis. A hot cathode arc-discharge ion source with a fine rectangular slit is used to produce the ions. The mass dispersion is about 1 cm at mass = 50 and $\Delta m = 1$ amu. For the measurement of the ion dose, alumina was covered with thin aluminum foil from all sides so that part of the beam current also falls on the foil. To avoid heating of the sample, the beam current was kept below 2 μA so that temperature does not exceed 40 °C during implantation. The vacuum during the ion implantation was maintained below 1×10^{-7} mbar using a turbo molecular pump. Microstructural analysis is studied using AFM of ND-MDT SOLVER PRO and optical microscope (Leica, Germany) located at Birla Institute of Technology (BIT), Mesra, Ranchi, India. For all the samples, AFM was operated in semi contact mode. The feedback mechanism is employed to adjust the tip-to-sample distance to keep the force between the tip and the sample constant. SPM-controller PCI interface Window compatible software is used for the AFM data analysis. Nanohardness was measured using CSM instruments nanohardness tester. Nanohardness measurement is carried out at Material Science Division, Indira Gandhi Centre for Atomic Research (IGCAR) Kalpakkam, India. The maximum load of 10mN was applied and loading rate was also kept same for all samples. For the determination of compound formation and estimation of grain size, we have used X-ray diffractometer (Rigaku, Japan). XRD is carried out at Department of Polymer Engineering BIT Mesra. The source was Cu Kα radiation having wavelength 1.54060 Å generated at 60 kV and 30 mA was employed for this purpose. The scan speed was maintained at 0.05°/min. The grain size (D) was calculated using Scherrer's formula [9],

$$D = 0.9\lambda/B\cos\theta_B,$$

where θ_B is the Bragg's angle, λ is the X-ray wavelength (1.54 Å) and B is the calculated full width half maxima (FWHM) in radian. Overall error in the estimation of grain size is 15%. Compound formation is also studied using FTIR (IR-Prestige 21, SHIMADZU, Japan) spectroscopy located at BIT Mesra Ranchi, India.

3. Results and discussion

Accelerated ions in material loses energy by two processes, "nuclear loss" and "electronic loss". Nuclear loss is due to elastic collisions whereas electronic loss is due to inelastic collision. Atomic displacement is due to nuclear collision and in general electronic loss is spent in ionization of the parent atoms. Although electronic loss is not expected to cause displacement of the lattice atoms in metals, recent years have seen many effects involving atomic displacement such as damage creation, anisotropic growth in metal glasses, annealing, phase transformation, amorphization and ion beam mixing [10]. As alumina is an insulator, nuclear loss as well as electronic-loss induced phenomena may be expected in our studies. For 30 keV nitrogen ions, the maximum ion depth in alumina is about 100 nm whereas the maximum ion profile concentration is around 48.7 nm as measured using SRIM. The electronic loss (27.9 eV/Å) is more than nuclear loss (20.3 eV/Å. The number of vacancies produced per ion per angstrom is 0.30 and the total ionization due to ion–atom collision at the surface is 28 eV/Å). We are interested in modifying its surface properties. Surface morphology and mechanical properties have been studied in details.

All the implanted samples and unimplanted sample were studied by AFM and Optical Microscope (1000×). Fig. 1