Processing of porous GaAs at low frequency sparking

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We report on the preparation of photoluminescent porous GaAs by the application of high voltage spark discharges at low repetition rates (20 Hz) in air and in argon atmospheres. The spark-processed porous (spp) samples were characterized by the observation of their visible photoluminescence (PL) when illuminated with UV monochromatic radiation. In contrast to previous work on spp-GaAs at high sparking frequencies we find that the PL of samples prepared at low sparking frequency is highly reproducible from sample to sample. Important differences are observed in the initial PL spectra of the spp-GaAs according to the atmosphere of preparation under similar conditions. After prolonged air exposure both the spp-GaAs prepared in air and in argon show two similar broad peaks at energy positions 2.5 and 3.1 eV. Raman results indicate that the PL might not be associated to any size dependent mechanism. We present evidence that oxygen compounds formed by the exposure to air of the samples play a role in the PL excited in the spp-GaAs. This is reinforced by x-ray photoelectronic spectroscopy measurements that indicate that the spp-GaAs is covered by an oxidized film. © *1999 American Vacuum Society.*
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I. INTRODUCTION

The spark-processing method is a useful alternative to prepare new photoluminescent semiconductor porous materials.[1] It has some potentially desirable features compared to the electrochemical etching method that has been extensively used in silicon,[2,3] such as (a) it is a non-wet approach that could be used to prepare optoelectronic devices, (b) the luminescent area affected can be chosen at will, (c) the emission can be tuned in some materials by changing the parameters of preparation (wafer-tip separation, substrate temperature, ambient gas, pressure, spark frequency, current, voltage, and exposed time, etc.), (d) the same equipment and procedures can be used for the preparation of any type of semiconductor material.

The list of materials spark processed, whose luminescence properties have been observed as reported in the literature are: Si, Ge, GaAs, Sb, Bi, Sn, As, and Te[4] and to this list recently, it was included the semiconductors CdTe, GaSb, InSb and InP.[5,6]

In this work, we report the preparation by spark processing of porous GaAs (spp-GaAs) at low frequency sparking and the characterization of its photoluminescence (PL) at room and low temperatures. In contrast to the previous work on spp-GaAs at high sparking frequencies, we find that the PL of samples prepared at low sparking frequency is highly reproducible from sample to sample. In order to clarify the effect of oxidation in the luminescent properties, we used two different atmospheres during the preparation: air and argon (Ar). To probe into quantum size effects as possible origin of the PL, we did a Raman study of three different luminescent regions of the sample. We present the result of a study of the chemical composition of the spp-GaAs surface using x-ray photoelectronic spectroscopy (XPS).

II. EXPERIMENT

Commercial *n*-type wafers GaAs(100) were used, in the spark processing method to prepare the porous material examined. A unipolar spark generator with a repetition rate of 20 Hz and voltages of 15 000 V was used for the spark processing of the samples studied. A tungsten tip separated 1 mm from the sample was used as anode. Nine samples of spp-GaAs were prepared. Four samples were prepared in air and the other five in argon atmosphere. The preparation times and other conditions of preparation of the spp-GaAs samples are given in Table I.

The PL spectra were excited using a 325 nm He–Cd UV LICONIX laser, and standard phase sensitive lock-in amplification of a photomultiplier detector signal that is examined after passage through a 50 cm Sciencetech monochromator. Parasitic plasma emission from the laser as well as scattered laser light were filtered by the use of appropriate interference and long-pass filters, respectively. Care was exercised to concentrate the radiation only to the circular eroded crater produced by the spark process. The temperature PL measurements were done placing the sample in the cold finger of a closed-cycle He refrigerator that allows setting it to almost any temperature between 10 and 300 K. For the XPS measurement, we used an electron analyzer MAC 2 (Riber) and a x-ray nonmonochromatic Mg $K\alpha$ x-ray source (1253.6 eV). The samples were fixed in an attachment of a copper substrate and introduced to the analyzer chamber working at a pressure of 1×10^{-8} Torr. The spectra were taken at room temperature, at high energy resolution steps of 0.5 eV, and in two ranges of energy: 0–1400 and 0–200 eV. The morphol-

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