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Ion beam induced adhesion in Al/diamond bilayer

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Diamond anvil cells are often used to study phase transitions in materials under high pressure. To probe these transitions, electrodes with low resistance and good adhesion properties are desired. Such electrodes were obtained using ion beam mixing techniques. The diamond anvil was first irradiated with a 100 keV Ar⁺ beam with an ion dose of 10¹⁶ ions/cm². Resistance measurements of the irradiated area indicated that ion beam damage induced a low-resistance phase of carbon. A thin Al film (≈ 500 Å) was then deposited on the diamond anvil followed by Ar⁺ irradiation. The deposition and irradiation processes were repeated, followed by resistance measurements after each irradiation. The measurements indicate the formation of a low resistance layer ($R \approx 10$ Ω). A qualitative test of adhesion was performed by scratching the formed layer with the resistance probes. The resistance of the electrode showed an acceptably small increase as a function of scraping. These results can be understood on the basis of recoil mixing in the Al/diamond system.

1. Introduction

The study of high pressure induced phase transitions in materials is a challenging task, and electrical resistance measurements using the diamond anvil cell are an essential part of such studies [1–5]. To carry out these measurements, contact electrodes with good adhesion to the surface of the diamond anvil are required, especially when a measurement of small resistance is considered. Ion induced mixing is frequently proposed as a technique to enhance adhesion in various film/substrate systems [6,7]. This is accomplished by the irradiation of these systems with beams of a few hundred keV heavy ions using small accelerators. The irradiation of materials results in the implantation of the impinging particles and the displacement of a fraction of the atoms constituting the surface layers. Such processes lead to the formation of a wide range of compositions and thus can be utilized to obtain metastable phases of materials. A considerable amount of work has been devoted to the study of the various mixing mechanisms that are responsible for this phenomenon in different systems at different temperature regimes as discussed by many workers [8–11]. However, data on the formation of binary alloys in the Al–C system are scarce, and no work has been done on the adhesion of Al to diamond as far as the authors know. In this work, the mixing induced adherent low resistance Al–C layer was obtained by the irradiation of the Al-coated diamond. The

properties of this layer were identified by resistivity measurement of this layer as it was scraped.

2. Experimental

An ion beam of Ar⁺ with 100 keV energy was used to irradiate the (111) diamond surface. A circular mask was used so that an area of about 4.8 mm² was exposed to the ion beam. The anvil surface was irradiated with a dose of 10¹⁶ ions/cm². A case was designed to hold the diamond anvil and to facilitate both irradiation and electrical measurement. A thin film of aluminum with a thickness of 450 Å was then deposited on the anvil surface where the same mask mentioned earlier was used during evaporation. The film thickness was chosen to be less than the projected range of 100 keV Ar⁺ ions in Al which is 950 ± 323 Å as calculated by TRIM computer code. A slow deposition rate of 1–2 Å/s was used to obtain a uniform layer of Al, and a pressure of 10^{−6} Torr was maintained during evaporation. At this pressure residual gas impurities might contribute to the adhesion of the evaporated layer. However, these films exhibited poor adhesion prior to ion beam mixing. The bilayer was then irradiated with a 100 keV Ar⁺ ion beam with a dose of 3.0×10^{16} ions/cm². The four-point probe method was used to measure the sheet resistivity of the mixing-induced layer. Four thin tungsten wires (0.020 in. diameter) were fastened through a piece of Plexiglas that fits the mask used for irradiation. The four wires were located in a square array 2.2 mm on each side. These probes were then used to carry out the current and voltage measurements to determine the sheet resistivity of the layer. To examine the adhesion of

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the mixed layer to the diamond anvil, wire probes were used to scrape the surface and the resistivity measurement was repeated. The experiment was carried out with a few variations on this technique. In one variation, a second thin film of Al was deposited after the mixing of the first layer and was subsequently irradiated with the Ar^+ ion beam. This was done to enhance the concentration of Al in the mixed layer, leading to higher conductivity.

3. Results and discussion

Resistance measurements after the irradiation of the diamond anvil with no Al layer indicated that the conductivity of the surface had a finite value since an electrical resistance of a few $\text{M}\Omega$ was measured. This can be understood on the basis of the amorphization of the diamond anvil surface. As the incident energetic Ar^+ ion impinges on the surface it initiates cascades of recoil carbon atoms in the near-surface region as the ions slow down primarily due to nuclear collisions. This process results in damage of the diamond lattice in the near-surface region and probably forms graphite which is known for its better electrical conductivity as compared to diamond. This explains the increase in the conductivity of the diamond anvil surface. The four-point probe resistivity measurements of the Ar^+ irradiated Al–C bilayer system showed two basic useful features. First, these measurements indicated a resistivity of $3.5 \times 10^5 \mu\Omega\text{cm}$ which is a relatively small value. Second, this value for the resistivity showed an acceptable small increase as the surface layer was scraped ($3.7 \times 10^5 \mu\Omega\text{cm}$) which indicates good adhesion of the mixed surface layer to the diamond substrate. In this calculation of resistivity a layer of $450 \text{ \AA}$ was assumed. The experiment was repeated with some slight variations and the general characteristics of the mixed layer were reproducible. These results should be compared with those obtained from nonirradiated films where scraping lead to the removal of deposited Al layer. In this work the mixing-induced phases were not identified.

At least one equilibrium phase in the Al–C binary system has been reported in the literature, namely Al_4C_3 [12]. Also no phase diagram was found by others.

In conclusion, ion beam mixing was shown to be useful for producing surface layers of aluminum rich carbon with low electrical resistivity and good adhesion to the diamond substrate. The induced mixed layer was reasonably hard as it was scraped with the thin tungsten probes. The results should lead to very useful applications in the study of phase transitions in materials under high pressure.

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