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NEW CROSS-SECTION METHOD FOR INVESTIGATION OF HEAVY ION IRRADIATION EFFECTS

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A new method of sample preparation of metals irradiated with heavy ions which has been developed in the Laboratory of Nuclear Reactions of JINR is described. It permits the study of the depth distribution of radiation damage not worse than the traditional cross-section technique. However it is a simpler method with a very high yield of specimens suitable for TEM-investigation. An example of using this method is presented.

The investigation has been performed at the Laboratory of Nuclear Reactions, JINR.

Новая методика "Cross-Section" для исследования воздействия тяжелых ионов

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Приведено описание методики препарирования металлических образцов, облученных тяжелыми ионами. Она позволяет изучать пространственное распределение радиационных дефектов не хуже, чем традиционный метод "cross-section". При этом она значительно проще и обеспечивает очень высокий выход пригодных для ПЭМ-исследований образцов. Представлен пример практического использования методики.

Работа выполнена в Лаборатории ядерных реакций ОИЯИ.

Introduction

Transmission electron microscopy (TEM) is at present the main instrument for direct analysis of radiation changes in various materials. It is known that the cross-section technique is the most informative method in TEM-studies of metals irradiated with heavy ions. However, its application has been limited by tedious preparation of the samples, described elsewhere[1-3]. Thus, a prolonged electroplating is required to

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obtain the necessary deposit thickness of about 1.5 mm. Later a method which introduces no damage must be used to slice these specimens perpendicular to the original irradiated surface and to cut off 3 mm diameter discs from these slices. The thinning of discs is also carried out perpendicular to the direction of irradiation and electroplating. Therefore, it is necessary to assure a good boundary between the original and the deposited layer, which is difficult at large thicknesses of the deposition.

An original method for creating an "artificial" damage profile on the plane parallel to the sample surface has been proposed by Reutov and Vagin (iy is briefly described in ref./4/). Charged particles pass through a mask of a special form, and as a result, their energy changes along the surface. However this technique cannot be used directly for ion energies less than 3-5 MeV/a.m.u.

This report deals with a new method of sample preparation for radiation damage depth distribution investigations in metals, irradiated with energetic heavy ions.

Experiment and Results

As usual in electron microscopy, samples with 3 mm diameter and thickness of 50-300 μm have been prepared. The preparation technique includes the following operations. A periodic microrelief characterized by depressions with well defined geometrical parameters is created on the sample surface (see figure 1).

Following the heat treatment the sample is irradiated with heavy ions with projective range $R_p < h$, where h is the depression depth. After the irradiation, the front surface is electroplated with the same material.

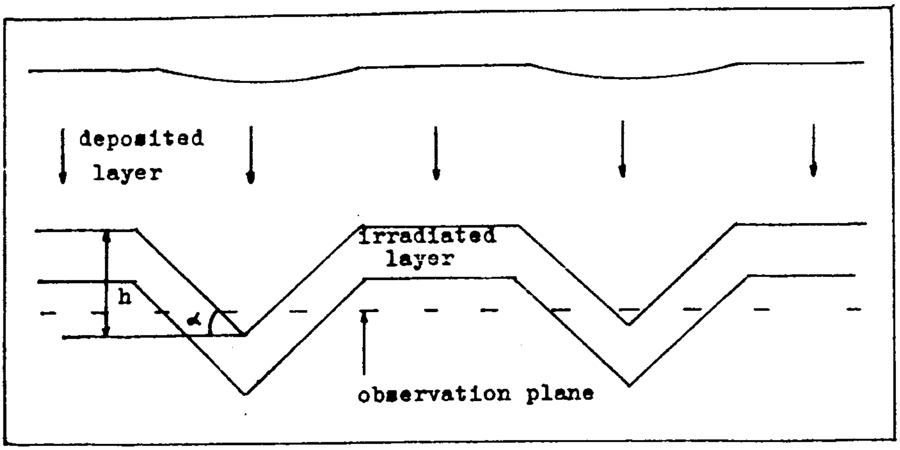

Fig.1. General scheme of sample preparation.

The thickness of the deposited layer is no more than (5-6)·h and equals in practice 30÷60 μm. The final operation includes two-side sample thinning to the plane of observation in a microscope located at a depth $R_p < x < h$ from the initial surface. Notice that if α is not equal to 90° the real spatial damage profile coordinates are obtained using the following simple expression: $x = x/\tan(\alpha)$.

No structural defects introduced during preparation were found of the unirradiated materials at the control experiments.

Thus, the presented method offers a possibility of studying the energetic dependence of heavy ion irradiation effects as good as mentioned above traditional cross-section technique. It is significantly simpler and does not require complicated and unreliable operations. All the necessary operations deal with standard microscope three millimeter discs. This method provides a very high (practically 100%) yield of specimens suitable for TEM.

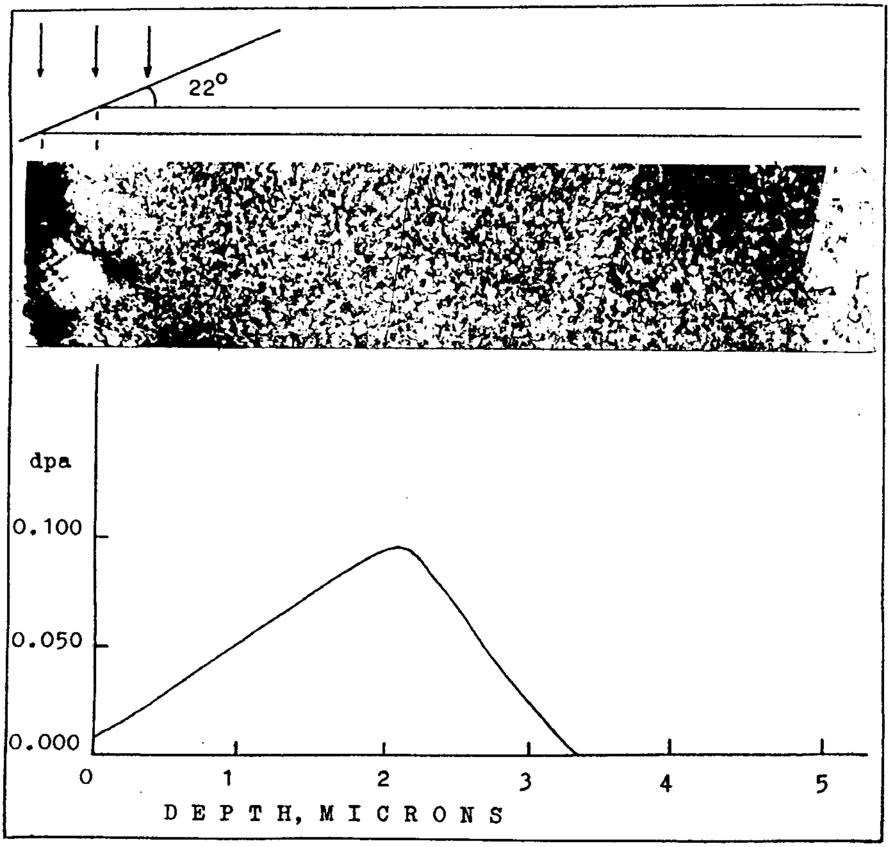

Fig.2. TEM micrograph of copper and damage profile for 46.3 MeV Ar ions irradiation through 10 μm Al foil.

Fig.3. TEM micrograph of the depth area near the end of the ion range.

An example of an application of the method is given below. The material used in this study was 99.99% copper previously annealled in a vacuum of 10^{-3} Pa at 700°C for 1 hour. The irradiation was performed to dose 10^{18} ion·m^{-2} with 46.3 MeV Ar ions through a 10 μm Al foil at room temperature at the IC-100 cyclotron. The aluminium shielding was done to obtain a suitable range of Ar ions in copper.

A TEM micrigraph of the irradiated copper is presented in figure 2. Also, the damage profile calculated by using the TRIM code is given in the same depth scale. It can be seen that the defects induced by irradiation extend to a calculated depth of about 2 micrometers. Similar results were obtained in some earlier works using the cross-section technique[2,5]. The discrepancy between the observed and calculated damage profiles is usually explained by stopping power errors.

Figure 3 shows an example of a TEM micrograph of the region near the end of the ion range. According to Narajan[2], the dislocations seen in this region are generated due to a stress build-up as a result of the volume change.

At present, the technique described above is being used to investigate the structure of metals irradiated with about 1 MeV/a.m.u. heavy ions. This programme is based on the IC-100 cyclotron experimental facility.

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