COMPOSITION ELEMENT PROFILING STUDY IN HIGH T_c-FILMS USING ACCELERATED LIGHT ION BEAMS

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Evaporation of high T_c-films on oxygen containing wafers is a well developed manufacturing technique. Still, due to strong interaction of elements being evaporated with a substrate some deviations in stoichiometric composition and crystal structure can occur and superconducting properties are not realized. Besides, some difference can exist in composition inside a film and in the thin layer near the surface. Hence, the study of a depth distribution of elements in a film is actual, especially, of oxygen content due to the largest mobility of these atoms in $YBa_2Cu_3O_{7-\delta}$ (123) superconductor lattice. Rutherford backscattering (RBS) and nuclear reactions (NR) are non-destructive methods supplying quantitative data for the elemental analysis [1–3].

Polished single crystal wafers of MgO were used as substrates for the manufacturing of thin films (123). The substrates of MgO with $\varepsilon = 10$ and $\tan\delta = 0.001$ are advantageous for

application in microwave electronics (as waveguides, coaxial microstrip connections in intergrated microcircuits etc.). The films (123) were manufactured by high frequency magnetron sputtering of the target with stoichiometric composition [4]. The film thickness was up to 10^4 Å; the superconductor transition temperature was 90° K.

For the measurements of the depth profile of heavy elements Rutherford backscattering of helium ions with the energy $E_\alpha = 3.07$ MeV was used. The experiments were carried out at the Van de Graaff accelerator EG-5 of JINR, Dubna. The diameter of the incident beam was $\simeq 1$ mm, the detection angle of backscattered ions was 170°.

The experimental spectrum of the backscattered helium ions and the results of its processing by a specialized program are shown in Fig.(a). The spectrum obtained by modelling fits the film composition $Y_{1.25}Ba_2Cu_{2.7}O_{7-\delta}$. However, homogeneity in the element distribution is disturbed in the near surface region as well as at the film-substrate interface. The fitting was carried out using the variations of the heavy elements profile at both outer and inner film boundaries. The resulting distribution of the elements for Y, Ba and Cu atoms is shown in Fig.(b).

The determination of the oxygen profile was carried out using the nuclear reaction $^{16}O(d,\alpha) \rightarrow^{14} N$ with the total energy transferred to the products of NR $Q_R = 3.111$ MeV [1]. The experiments were done at the electrostatic accelerator ESA-2 of PINP, Russian Academy of Sciences, Gatchina. The samples were irradiated with monoenergetic deuteron beam with the energy $E_d = 0.9$ MeV and the diameter in $\simeq 1.5$ mm. The resulting spectra of α-particles were measured by silicon high resolution planar detector set at 135° to the projectile. Monoenergetic α-particles are produced at various depth inside the film and due to the energy loss of helium ions the energy straggling occurs resulting in the spectrum measured by the detector. Thus, the energy and the yield in α-particle spectrum contain information about the oxygen distribution in the film. In the present experiment the maximum α-particle energy was 2.675 MeV.

The depth resolution Δx is limited by the energy resolution of silicon detector ($\Delta E =$ 8 keV at $E_\alpha = 2.675$ MeV), ionization energy loss of α-particles in the target and the experimental position design [1] and equals 150 Å for (123). One should emphasize that this resolution is achieved only for the film surface and grows up to 500Å for the total film thickness in 10^4 Å due to the energy straggling [5].

In Fig.(c) the experimental spectrum of α-particles produced in NR $^{16}O(d,\alpha) \rightarrow^{14} N$ is shown measured at the same point of the ion beam incidence on the film as in RBS spectrum measurements (Fig.(a)). The oxygen depth distribution (Fig.(d)) was extrapolated from the experimental α-particle spectrum (Fig.(a)) using the data in [1]. The oxygen distribution in the deeper layers of the film was homogeneous with a content equal to 53.8 at.% whereas the surface layer in 2000 Å was enriched with oxygen up to (63 ± 2) at.%. The relative accuracy determined by the statistical error is 2.5%.

On the contrary, RBS data show the deficiency of the heavy element atoms in the surface layer. Previously the anomalies in barium sublattice in the surface layer in 1000 Å were detected by ion-channeling measurements made in the same place of the specimen [6]. The correlation of the Ba-sublattice defect region thickness and that of increased oxygen content show their interdependency. The reasonable explanation can be high chemical activity of barium and its possible oxidation.

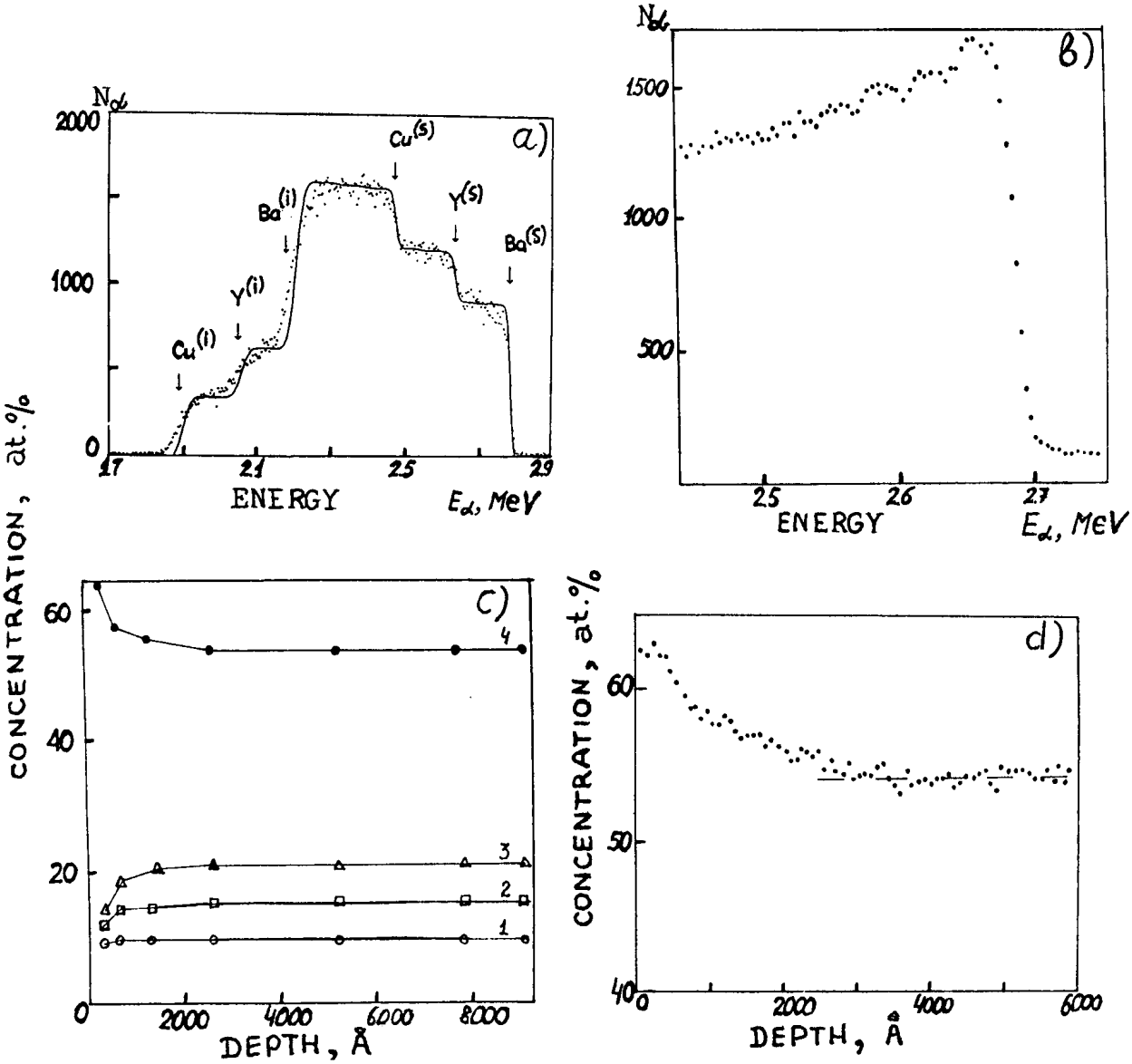

Fig. Experimental spectra of helium ions and the results of their processing:
a) backscattering spectrum of $^4He^+$ with starting energy $E_\alpha = 3.07$ MeV from high T_c (123) film on MgO substrate (points) and result of its modelling for $Y_{1.25}Ba_2Cu_{2.7}O_{7-\delta}$ composition (solid line). Edges of steps correspond to ion scattering from outer and inner boundaries of film;
b) element concentration distribution of: Y (curve 1, o — open circles), Ba (curve 2, □ — squares), Cu (curve 3, △ — triangles) and O (curve 4, • — full circles) obtained by detailed modelling of spectrum in Fig.(a);
c) spectrum of helium ions resulting from NR in the same film, deuteron energy is 0.9 MeV;
d) oxygen concentration depth profile for (123) film obtained from Fig.(c). Depth scale in Å corresponds to film density in 6.35 g/cm^3

The results of RBS spectra processing enable to make a conclusion (though indirect) about the increased content of the light element (presumably oxygen) in the surface layer of high T_c-film. Its distribution to be supposed is represented in Fig.(b), curve 4 (full

circles). The experimental depth profile of the oxygen measured by NR (Fig.(d)) shows close agreement with that obtained by modelling (Fig.(d), curve 4) in both depth scale and element content.

Thus, combination of RBS and NR measurement modes provides complex reliable analysis of high T_c films.

REFERENCES

1. *Verbitskaya E.M. et.al.* Preprint LNPI–1755. Leningrad, 1991. 16 p.
2. *Andrienko V.B. et.al.* Preprint LNPI–1442. Leningrad, 1988. 26 p.
3. *Dyumin A.N. et.al.* Preprint PNPI–1856. Gatchina, 1993. 14 p.
4. *Boguslavsky et.al.* // Superconductivity: Phys.Chem.Tech. (Russian), 1991. V.4. N°9. P.1790–1795.
5. *Mayer J.M., Rimini E.* Ion Beam Handbook for Material Analysis. New-York. Academic Press, 1977. P.1–20.
6. *Verbitskaya E.M.et.al.* // J.Tech.Phys., 1993. V.63. N°5. P.111–116.