Ellipsometric control of stress homogeneity in surface layer of amorphous metal alloy ribbons

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The level of internal strain changes in the subsurface layer of Fe-based ($Fe_{75}Ni_4Mo_3Si_2B_{16}$ and $Fe_{80}B_{20}$) amorphous metal alloys has been analyzed. Optical measurements were carried out for as-cast ribbons. . The noncircular form of the polar dependence of light incidence principal angle indicates a high sensitivity to internal stress variations arising during ribbon preparation.

Проанализирован уровень изменения внутренних напряжений в поверхностном слое лент аморфного металлического сплава на основе железа ($Fe_{75}Ni_4Mo_3Si_2B_{16}$ и $Fe_{80}B_{20}$). Оптические измерения проведены для свежеизготовленных лент. Некруговая форма полярной зависимости главного угла падения указывает на существование и характер изменения внутренних напряжений в плоскости ленты, возникших в процессе ее изготовления.

Amorphous metal alloys (AMA) on the basis of iron are intensively investigated in connection with their potential wide practical applications. Chemical, thermodynamic, magnetic, mechanical, and other properties of the AMA ribbons prepared from the melt by spinning method differ considerably from those of the ordered material. These properties are defined not only by structure features formed during manufacturing of such material but by the further heat treatment as well [1]. The former is main factor influencing the physical properties of such materials and nanostructures which depend strongly on the preparation conditions. That is why for these objects often not the physics of a phenomenon as such is studied but the physics of a specific sample is just taken into account, since so-called technological polymorphism [2] is manifested for the sample structure at the same composition of its chemical components due to nonidentical preparation conditions. As a consequence, the physical properties of such materials are difficult to predict, and their necessary precision as functional ones is not yet reached, so the fields of their usage are narrowed. Besides, the elastic stressed state within subsurface layer of the amorphous alloy ribbons may be formed [1].

Meanwhile, to identify correctly the amorphous state of a material, the existence of similar influence of two independent factors, namely thermal and deformational ones, on the structure of its nearest atomic arrangement is to be taken into account [3]. That is why the aim of this work is to discriminate the two mentioned constituent factors to provide a correct identification of initial amorphous state in a material induced by its preceding preparation procedure, and then to describe correctly the further structural modifications in such disordered material taking into account the existence of an elastic-stressed state within the subsurface layer.

Being made from the melt by means of rapid quenching on a running disk at a cooling rate of 10^6 K/s, a set of samples of amorphous metal alloys $Fe_{75}Ni_4Mo_3Si_2B_{16}$ (MG) and $Fe_{80}B_{20}$ was prepared as ribbons of 10 to 15 mm width and 20 to 40 μm thickness (d). The melt temperature was adjusted to 50–100 K above the melting point. Both contact side of the sample (i.e. that being in contact with cooler during preparation) and noncontact one (remaining free during preparation) were investigated. Optical properties were studied using a home-made reflectometer-goniometer of original design to test the freshly prepared ribbons of AMA $Fe_{75}Ni_4Mo_3Si_2B_{16}$ (MG) and $Fe_{80}B_{20}$. The angular ellipsometry has been proposed as an effective research method of structural ordering in subsurface layers of amorphous ribbons and their stress homogeneity [4]. The research of the reflected light polarization allows to obtain the most complete information on the dielectric function of the subsurface layer. Angular ellipsometric measurements for the samples of the mentioned alloys were fulfilled using a standard LEF-3M laser ellipsometer. As a light source, a He–Ne laser (632.8 nm wavelength) was used. According to the optical scheme of this measuring device, the phase shift Δ between *p*- and *s*-components of the polarization vector and the azimuth Ψ of the restored linear polarization were calculated.

We have studied both ribbon sides, namely, the freely formed (or noncontact) side and that was directly contacted the rotating disk in the course of manufacturing. Visually, the reflected light at noncontact surface presents a predominant mirror component as compared to that at the contact surface, which scatters light much more. This indicates that the root-mean-square roughness of the latter surface is several orders of magnitude higher than the corresponding value of opposite (noncontact) side of the ribbon [1]. The value of roughness and structural features of the contact surface is defined only by the interaction character of the melt of AMA $Fe_{75}Ni_4Mo_3Si_2B_{16}$ and $Fe_{80}B_{20}$ with the rotating disk surface, and thus by the disk surface roughness.

Optical measurements were carried out for samples of Fe-based amorphous metal alloys at 12 orientations of the ribbon longitudinal axis relatively to the plane of light incidence. Having determined the deviations of ellipsometrical parameters for the light reflected from the ribbon surface as a function of its orientation in the ribbon plane, the level of strain changes in the subsurface layer of amorphous ribbons fabricated by rapid melt quenching can be estimated. The existence of elastic-stressed state within subsurface layer for each ribbon side and the difference of the state characteristics in as-cast ribbons of amorphous alloys on both sides can be also revealed. To that end, both sides of an amorphous alloys ribbon were probed by light using experimental methods based on angular and azimuthal ellipsometric measurements on rotating samples [5]. By rotating a ribbon around a normal to its surface within 360°, one can acquire the polar diagrams (so-called indicatrices of ellipsometric parameters over the longitudinal axis azimuth with regard to the plane (*p*-plane) of light incidence) as a result of such orientation measurements.

The procedure of these ellipsometric experiments comprises adjusting of the longitudinal ribbon axis in its plane at 12 fixed azimuths such as $\theta_1 = 0°$ (the axis direction coincident with *p*-plane), and then at further set of azimuths at 15° steps up to $\theta = 180°$ (at $\theta_7 = 90°$, the direction of the mentioned axis coincides with *s*-plane of the sample reflecting light). The angular dependences of ellipsometric parameters Δ and Ψ for the noncontact and contact surfaces of as-cast ribbon sample at various azimuthal angles θ for different angles φ of light incidence were measured to determine the principal angle φ_0 of incidence at each azimuth θ of the longitudinal ribbon axis in its plane. Such angular dependences $\Delta(\varphi)$ for both sides of a $Fe_{75}Ni_4Mo_3Si_2B_{16}$ (MG) AMA ribbon are shown in Fig. 1 where straight lines are certain approximations of these dependences for several azimuths θ in the ribbon plane.

Experimental dependences $\varphi_0(\theta)$ in the polar and Cartesian coordinate systems are shown in Fig. 2, i.e. azimuthal distribution for φ_0 is shown in the former system by closed curves (indicatrices) in the ribbon plane. The azimuthal angles θ_i ($i = 1,...,12$) are measured from the direction of the ribbon longitudinal axis. Similar measurements of the $\Delta(\varphi)$ dependence for a $Fe_{80}B_{20}$ AMA ribbon (using 45° azimuthal rotation of the sample in its plane) have been carried out. The noncircular form of such indicatrices reveals an orientation effect. It is possible to allocate two characteristic directions of optical anisotropy (for φ_0 value) of such a surface layer for the ribbon contact side at angles $\theta = 60°$ and 135° relatively to the ribbon longitudinal axis. Hence, the polar dependence $\varphi_0(\theta)$ is very sensitive to a variation of the internal stresses arising in the course of the ribbon preparation. Meanwhile, because samples were not polished additionally the deviation of Ψ values is too large.

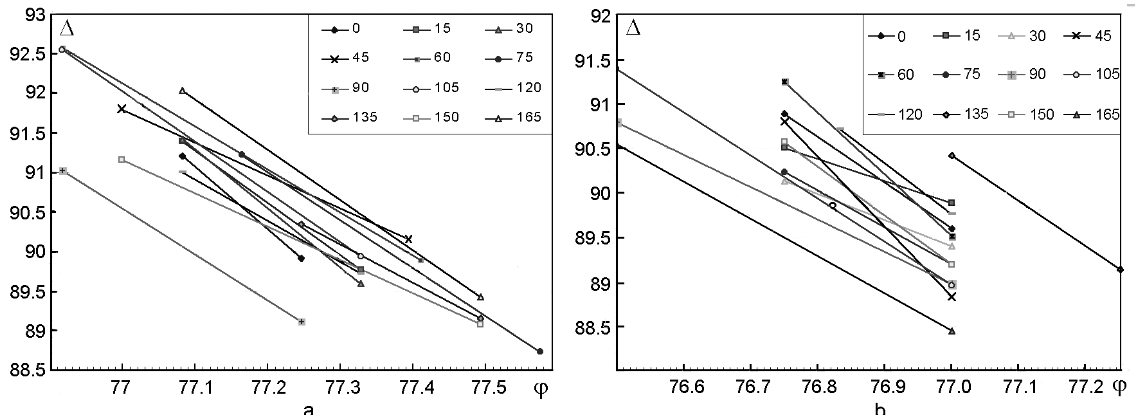

Fig. 1. Angular dependences $\Delta(\varphi)$ for contact (a) and noncontact (b) sides of a $Fe_{75}Ni_4Mo_3Si_2B_{16}$ AMA ribbon.

For noncontact ribbon surface, a change of the microrelief parameters may also occur as a result of appropriate thermodynamic processing under influence of internal mechanical stress fields in the ribbon. These changes are caused by diffusion processes at the surface layer and structural phase transformations in the superficial layer. Such processes are accompanied by migration of separate atoms on the surface as well as of their associations including hundreds and thousands atoms [5]. Thus, one of the feasible causes of such surface state as it is suggested up-to-date [6, 7] might be the diversity of atomic coordination due to structural stressed state in the nearest order topology at the same material composition. Therefore, any change of such state under external action may be registered experimentally. Specifically, a detailed study of radial distribution function for atoms in as-cast binary amorphous alloy consisting of a transition metal and a metalloid (e.g., nickel-phosphorus [7]) and its changes for such type of materials after annealing (T = 250°C for 30 min) proves a distinct difference between structural parameters in the nearest atomic arrangement. Only due to such thermal factor, the whole structure of this arrangement has already been rebuilt within the first 5 coordination spheres at corresponding interatomic distances r from $r = 2$ or 3 Å (the 1st coordination sphere) to 10–12 Å (the 5th coordination sphere).

In our opinion, the result obtained testifies the presence of structural microheterogeneity in the ribbons formed at their preparation and occurring as elementary structural units which can act like a cluster or microcrystalline form [4]. The $\varphi_0(\theta)$ dependence for the ribbon contact side correlates appreciably with $\varphi_0(\theta)$ dependence for noncontact one (Fig. 2). But for the latter, the optical anisotropy is much larger than in the former: the appropriate amplitude deviations in both curves confirm this statement. Such a relation testifies the presence of a stronger straining factor for the noncontact ribbon side caused by the preparation conditions. At the ribbon formation, owing to a of cooling speed gradient along its thickness, the internal stress fields arise in the surface layer and local microheterogeneities are formed in such amorphous structure. These stresses in the surface layer can be decreased by subjecting the AMA ribbons to additional heat treatment.

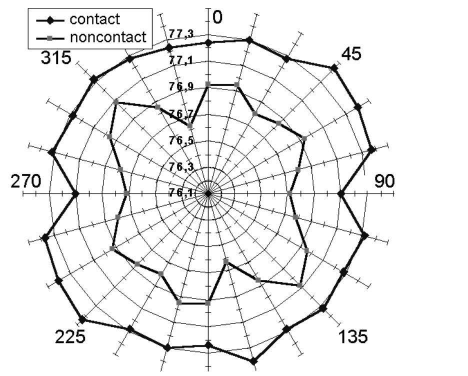

Fig. 2. Dependence $\varphi_0(\theta)$ for amorphous $Fe_{75}Ni_4Mo_3Si_2B_{16}$ alloy ribbon in polar coordinate systems.

Thus, the ellipsometric measurements prove that after manufacturing ribbons of AMA $Fe_{75}Ni_4Mo_3Si_2B_{16}$ (MG) and $Fe_{80}B_{20}$, their surfaces are in an intense stressed state and the level of such strains can be estimated from the parameters of polar dependence of the principal angle in the ribbon plane. Then, residual strains are formed in the ribbon due to rapid cooling of the melt, and spatial distribution of the internal stresses is determined by the ribbon preparation conditions but not by the directed roughness of the ribbon surface.

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Еліпсометричний контроль ступеня однорідності стрічок аморфних металічних розплавів

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Проаналізовано рівень змін внутрішніх напруг у поверхневому шарі стрічки аморфного металічного розплаву на основі заліза ($Fe_{75}Ni_4Mo_3Si_2B_{16}$ і $Fe_{80}B_{20}$). Оптичні виміри проведено для свіжевиготовлених стрічок. Неколова форма полярної залежності головного угла падіння світла вказує на існування і характер варіації внутрішніх напруг уплощині стрічки, зумовлених процесом виробництва.