The influence of deformation by equal-channel multiple-angle pressing and durable thermal treatment on phase composition and physical properties of NbTi alloy

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The influence of deformation by equal-channel multiple-angle pressing and of thermal treatment on phase composition, state of structure and current-carrying capacity of NbTi alloy superconductor have been studied. Deformation is shown to result in the decreasing of subgrain size (100...200 nm). In the temperature range to 450°C, the used structure was found thermally stable. This is explained by precipitation of disperse particles of the second α-phase at the subgrain boundaries. After the deformation there was a distinct increase in magnetization of the alloy due to magnetic flux fixing at boundaries of crystallites and at α-phase precipitations.

Исследовано влияние деформации равноканальным многоугловым прессованием и термообработки на фазовый состав, структурное состояние и токонесущую способность сверхпроводника из сплава NbTi. Установлено, что деформация приводит к уменьшению размеров субзерен (100...200 нм). Установлена термостабильность структуры, сформированной прессованием в диапазоне температур до 450°C, что объясняется выделением дисперсных частиц второй α-фазы по границам субзерен. После деформации наблюдается отчетливое повышение намагничивания сплава вследствие закрепления магнитного потока на границах кристаллитов и выделениях α-фазы.

The functional properties of superconductors based on NbTi alloy, are determined mostly their structure-phase state (the characteristics of structure and grains (subgrains) size, volume content, density and secondary phase percipitaions size), which, in its turn, is established by the preliminary multiple-route treatment (prehistory) of the material. We know a number of technological ways for creating favorable structure state and achieving with it a high current-carrying capacity of alloy wire articles: microdoping with α and β stabilizers and neutral elements on the metallurgy stage, mechanical-thermal treatment with changes in deformation direction of the ingots, doping and mechanical-thermal treatment (MTT) combination, hydraulic forging of the mountings, deformation modes of traditional treatment methods — pressing and wire-drawing optimization, deformation combined with durable intermediate thermal treatments (ITT) [1–5].

In this connection, we can expect that, development and use of special methods, such as equal-channel multiple-angle pressing, alternative to the known ones, can provide for the further progress in this sphere. Equal-channel multiple-angle pressing (ECMAP) without blank section change in fractional mode with unit intensity per cycle ($e < 1$), due to its technological properties of nonmonotonous alternating plastic strain, provides the forming of a special structure state, unachievable by traditional methods [6–7]. Such structure, virtually, with absent texturing, dislocations density gradient, regular alternation of low-size regions with high and low dislocations density, favors the precipitation of disperse secondary phases, which are the centers of magnetic flux quanta setting, on the subgrains boundaries. A homogenous disperse structure with low level of microstresses provides satisfactory technological plasticity, sufficient for making the blank into wire and so, supports the realization of the proposed way to rise the superconducting characteristics. On the other hand, we regard another important approach in this work is the use of durable thermal treatment on the stages of blank treatment with equal-channel multiple-angle pressing. The thermal treatment should be done in the temperature range, which corresponds to the two-phase region in the diagram of niobium-titanium alloy states. This is expected to provide forming of even more favorable structure-phase state for current-carrying capacity rise. This phase state is characterized by increased volume content as well as α-phase precipitation density. The aim of our work is studying the possibility to increase current characteristics for niobium-titanium alloy by complex exposure to ECMAP deformation in the fractional mode and durable thermal treatment. The main direction of our research is to find out the ECMAP deformation and durable thermal treatment influence upon the phase composition, structure state and, finally, current-carrying capacity of NbTi alloy superconductor.

In our experiments, the subjects of inquiry were bimetallic bars of niobium-titanium alloy (Nb — 60 at. % Ti) in the copper (MOб) matrix. The bars of 60T alloy 15 mm in diameter, obtained by hot pressing at 750°C, underwent equal-channel multiple-angle pressing without blank section change, according to four-channel deformation scheme with channels intersection angles $Q_1 = Q_3 = 80°$ and $Q_2 = 70°$, unit per cycle intensity of displacement deformation ($\Delta\Gamma$), equal to 1.42 and equivalent, respectively, to 0.82 deformation extent (e) [8]. For purposeful forming of fine-dyspersated random-oriented alloy structure, the blank was turned for 180° around its transverse axis and for 90° around its longitudinal axis in each further pressing cycle. The compacting pressure was 600...800 MPa. Deformation accumulation under the mentioned conditions was caused by repeating pressing cycles in the interval 4–8–12–16–20. Full accumulated deformation was $e = 16.4$.

The thermal stability of the structure formed in ECMAP alloy with accumulated $e = 16.4$ was studied at one hour exposure to temperatures in the range 300, 350, 400 and 450°C. The deformed samples were heated with exposure from 1 to 47 h at 400°C in the vacuum 10^{-5} mm of mercury. Microstructure, fine structure, physical and mechanic properties of the alloy after its deformation and thermal treatment were studied by optic microscopy, X-ray structure analysis and microindentation methods. The chemical content was studied electron-scan microscope JSM-T300 with c power dispersion device Link 860-500 for local analysis. The current-carrying capacity of initial and ECMAP deformed alloy was evaluated by scaling the magnetization curves for massive sample cut out from cubic blanks with sides 5 mm, taken at 4.2 K in the external magnetic field with induction up to 14 T and sample orientation in it on three main directions.

The results of optic microscopy and X-ray examination of fine structure show that samples deformed by equal-channel multiple-angle pressing in the fractional mode without section change have a more homogeneous and fine-dyspersated structure with a lower texturization level than in case of treatment by traditional hot pressing (Fig. 1). The characteristic size of structure elements (subgrains) are 100...200 nm unlike the corresponding characteristic ~1100 nm for hot-pressed samples and ~500 nm for samples, obtained after mechanical-thermal treatment (PMTO) with changes in deformation direction. Further heating with exposure to temperature 300...450°C does not cause any sufficient change in the characteristic size of structure elements (subgrains) for ECMAP deformed alloy. This can be explained by the fact that on the subgrains boundaries disperse particles of second α-phase, preventing the subgrains from growing. But at the same time, the observed narrowing of X-ray lines, microstresses level ($\Delta a/a$) testify to alloy poligonization activation under thermal influence in the mentioned temperature range along with dislocations density in the subgrains corpuses (Fig. 2).

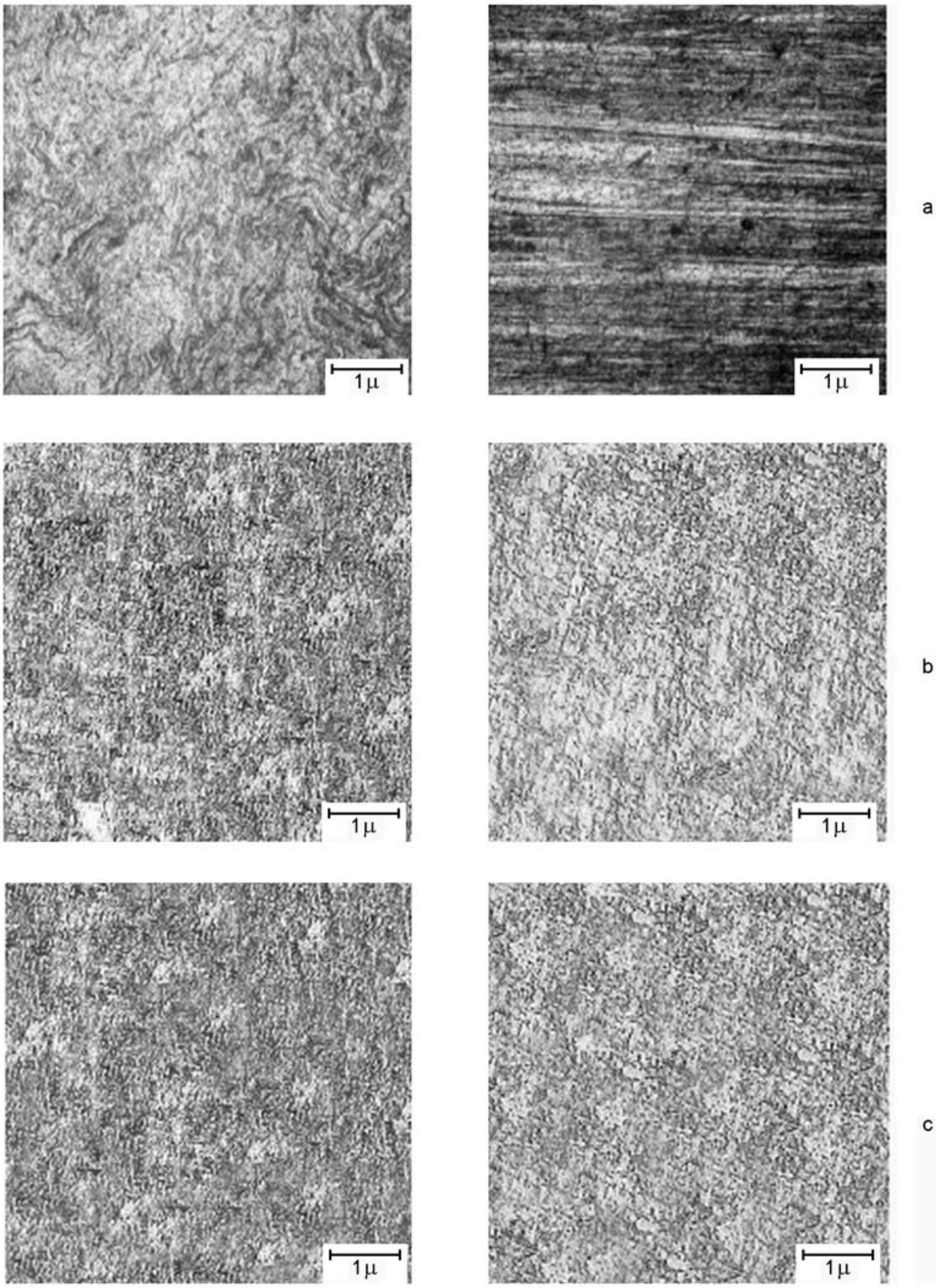

Fig. 1. The microstructure of 60T alloy (cross-section and longitudinal section): a — initial state, b — after cold ECMAP deformation (e = 16.4), c — after ECMAP and annealing at 400°C for 1 h.

The use of ECMAP in the intermediate stage of superconductor obtaining technology causes structure changes in alloy 60T content with additional quantity of α-phase precipitation. The X-ray studies show that preliminary ECMAP influence at full structure treatment of the blank along its section and height triggered secondary phases nucleation and the increase of α-phase volume content up to 3 %. But structure heterogeneities formed, subgrains boundaries, decorated with non-superconducting α-phase precipitation substantially determine the increased critical current level.

In Fig. 3 one can see the results of massive ECMAP-treated sample current-carrying capacity measuring depending on the magnetic field (e = 16.4) in comparison with sample, not treated by ECMAP. The figure implies that equal-channel multiple-

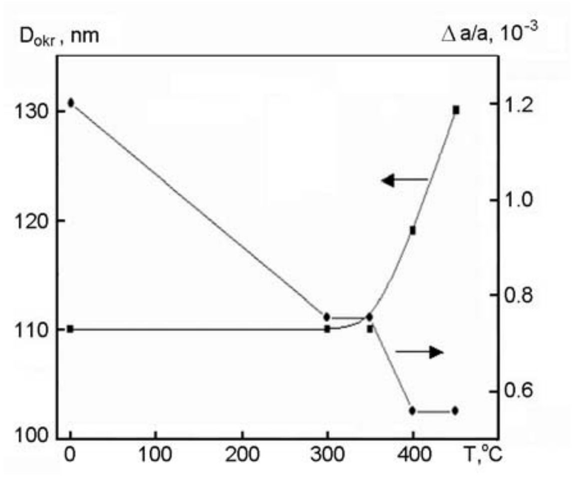

Fig. 2. The thermostability of alloy 60T thin structure, formed by ECMAP.

angle pressing treatment causes a higher current-carrying capacity level in all the magnetic fields range, than in case when ECMAP treatment was not used. After ECMAP deformation, a distinct increase of alloy magnetization due to magnetic flux fixing on the crystallites boundaries and α-phase precipitations. The analysis of magnetization curves for the initial and deformed samples of the alloy, taken from the external magnetic field with the induction up to 14 T at 4.2 K, the cubic sample oriented along the three main directions, showed the magnetic properties anisotropy presence in the initial hot-pressed as well as in the ECMAP deformed alloy with its special structure state.

We know that critical current increase is determined by α-phase particles presence, and the increase rate correlates with volume content, the particles size, their density and disposition. The corresponding structure with great density and fine-dyspersated α-phase particles content is formed during deformation and long repeated annealings. One should expect that the combination of ECMAP deformation with durable thermal treatments provides favorable structure-phase state and, finally, a higher critical current level. Fig. 4 presents the research results on the influence of ECMAP deformation followed by thermal treatment, including the durable one, upon the changes of alloys 60 T phase state. After durable (up to 47 h) thermal treatments of ECMAP deformed samples, α-phase volume content at homogeneous distribution increases from 3 to 9 %.

The dependence of α-phase amount with exposure time increase at temperature 400°C is nonmonotonous. There are two regions of α-phase amount increase, divided with a noticeable minimum, and this fact proposes and reflects the mechanisms of α-phase precipitation and increase. Initially, α-phase amount increases, probably, due to nuclei number increase. The nuclei are formed on the dislocations and almost do not grow. Then the mechanism becomes exhausted, due to the decrease of dislocation density in cell bodies as well as a more clear cellular structure forming. The minimum on the dependence of α-phase amount on annealing time in 3–5 h interval can be explained by the solution process of particles smaller than critical size, which after dislocations drift or annihilation were left in the cells volume and did not find themselves on the subboundaries, stable nuclei still having not grown yet. Subsequent substantial α-phase volume content increase takes place here due to the linear growth of the nuclei, fixed on the subboundaries. Summarizing the result of the alloy microanalysis, we can pay attention to the diffusion nature of $\beta \to \alpha$ phase transformations on samples, durably thermally treated. The redistribution of titanium and niobium is observed,

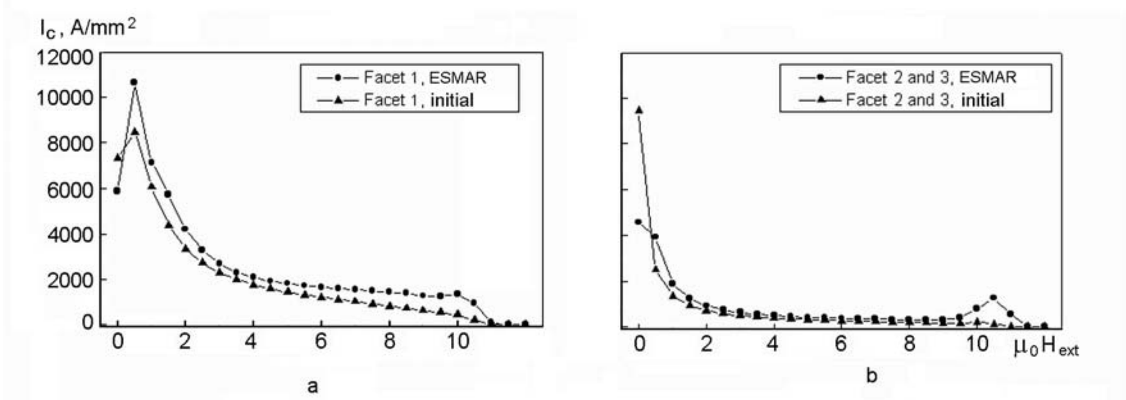

Fig. 3. The influence of ECMAP deformation on current-carrying capacity.

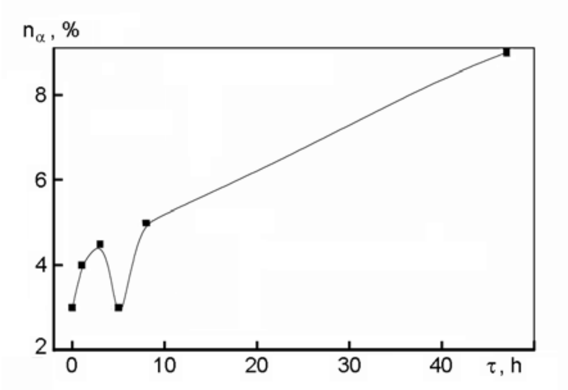

Fig. 4. The dependence of α-phase volume content upon the annealing time at 400°C.

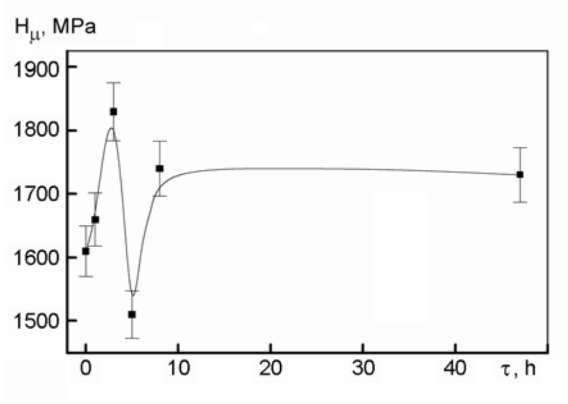

Fig. 5. The influence of thermal treatment duration on microhardness.

the alloy becomes heterogeneous by its content, the regions depleted of titanium and enriched by niobium are formed. Quantitative estimation of phase heterogeneity in 60T alloy in the annealed samples (400°C, 47 h) shiws the content change up to 7.5 %, which corresponds to secondary phases forming.

The disperse α-phase particles precipitation and matrix depleting of titanium cause physical properties to change. Fig. 5 shows the results of microhardness measuring for ECMAP deformed alloy depending on the exposure time at 400°C. The results correlate with the α-phase amount changes. Microhardness value H_μ does not essentially change along the section and height of the sample; microhardness irregularity (H_{max}/H_{av}) is 1.06, which confirms the fullness of structure treatment and structure state homogeneity. Analyzing the result of our work, we should note the following. The main influence factor for the alloy phase composition on the intermediate stage of making the ingot into wire is cold nonmonotonous, alternating-sign plastic ECMAP deformation without blank section change, because it predetermines changes in the phase composition. The physical base lies in the creation during ECMAP treatment a specific, more favorable, structure, which can lead to critical current increase by activating $\beta \rightarrow \alpha$ transformation process in 60T alloy and α-phase amount increase after durable thermal treatments later, other technological conditions being equal.

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Вплив деформації рівноканальним багатокутовим пресуванням і тривалої термообробки на фазовий склад і фізичні властивості сплаву NbTi

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Досліджено вплив деформації рівноканальним багатокутовим пресуванням та термообробки на фазовий склад, структурний стан і струмонесучу здатність надпровідника зі сплаву NbTi. Виявлено, що деформація спричиняє зменшення розмірів субзерен (100...200 нм). Встановлено термостабільність структури, сформованої пресуванням у діапазоні температур до 450°C, що пояснюється виділенням дисперсних частинок другої α-фази на межах субзерен. Після деформації спостерігається виразне підвищення намагнічування сплаву внаслідок закріплення магнітного потоку на межах кристалітів та виділеннях α-фази.