

Production of textured ribbons based on Ni–W paramagnetic alloys

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Received October 17, 2012

The objective of the work is searching for ways to produce substrates for second generation HTSC-covered conductors based on high tungsten content Ni–W paramagnetic alloys having a cubic texture of {001}<100> type. The work is based on the following hypothesis: increasing the stacking fault energy E_{sf} , and, as a consequence, cubic structure formation is possible due to rather strong compressive stress in the ribbon. On the conceptions developed, two new ways for production of a cubic texture in the ribbon were realized: (i) TiN thin layer plasma deposition, and (ii) Ni–W alloy rolling under conditions providing dislocation high density ("extreme rolling"). Optimal schemes for mechanical and thermal treatment of paramagnetic Ni–9.5 at. % W alloy based ribbons were developed including choice of synthesis method, rolling regimes parameters, ribbon thickness, annealing temperature and time, etc.

Целью работы является поиск путей получения подложек для ВТСП-покрытых проводников второго поколения на основе парамагнитных сплавов Ni–W с высоким содержанием вольфрама, обладающих кубической текстурой типа {001}<100>. В основу работы положена следующая рабочая гипотеза: повышение энергии дефектов упаковки E_{sf} и, как следствие, формирование кубической текстуры возможно в результате создания достаточно сильных сжимающих напряжений в ленте. На основе развитых представлений реализованы два новых пути получения кубической текстуры ленты: в результате плазменного осаждения тонких слоев TiN ($a_{TiN} \ll a_{Ni-W}$) и в результате проведения прокатки сплава Ni–W в условиях, обеспечивающих создание высокой плотности дислокаций ("экстремальная прокатка"). Разработаны оптимальные схемы механической и термической обработки ленты на основе парамагнитного сплава Ni–9.5 at. % W, включающие выбор метода синтеза, параметров режимов прокатки, толщины ленты, температуры и время отжига и т.п.

1. Introduction

As it is known (see, for example, [1–3]), the architecture of the 2nd generation HTSC-coated conductors (2G HTS) with current critical density $j_c \sim 10^6$ A/cm² at $T = 77.4$ K is rather complicated. Practically always, the following three main components are in the construction of HTSC-coated conductors:

1. A substrate; as a rule, it is a thin ribbon made from Ni–W alloys of different compositions.

2. A non-superconducting buffer layer (oxides, nitrides, etc.).

3. A superconducting film coating (for 2G HTS, it is $YBa_2Cu_3O_{7-\delta}$).

The application efficiency of the Ni–W ribbons is substantially influenced by both the alloys magnetic structure (a collinear ferromagnetic one — under low W content,

or paramagnetic one — under high W content), (see, for example, [4, 5]), and the crystallographic character of the ribbon texture. The ribbon texture is formed during mechanical and thermal treatment [6–10]. The paramagnetism of the ribbon-substrate results in decreasing the level of ferromagnetic losses during passing the alternating current over the superconducting film, i.e., in increasing the critical current effective density.

The presence of $\{100\}\langle 001\rangle$ cubic texture in the ribbon provides a possibility of epitaxial growth for $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductor film on its surface, when the charge transfer plane CuO_2 $\{001\}$ in the $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ lattice is positioned parallel to FCC cubic plane of Ni–W alloy [11]. Naturally, with such substrate and superconductor mutual orientation the optimal conditions are provided for superconducting current passing.

Seemingly, the preparation of substrates for 2G HTS with high current-carrying ability is quite simple problem. It is necessary to synthesize the Ni–W paramagnetic alloy with high W content, and then, from the alloy obtained, to prepare a ribbon with cubic texture by using mechanical and thermal treatments. However, numerous attempts to obtain a cubic texture in the Ni–W alloy ribbon with higher than 5 at.% W were unsuccessful (see, for example, [9, 12–15]). The serious obstacle for production of ribbons with such combination of texture and paramagnetic properties is the fact that paramagnetic Ni–W alloys with high W content have low value of stacking fault energy E_{sf} that prevent from cubic texture formation after high-temperature annealing [16].

In this connection, the objective of the work is searching for the ways to produce substrates based on Ni–W paramagnetic alloys with high W content for application in the construction of 2nd generation HTSC-coated conductors. In fact, the goal achievement is reduced to developing a strategy for preparation of the Ni–9.5 at. % W paramagnetic alloy ribbon with developed cubic $\{001\}\langle 100\rangle$ texture. The works on preparation of the ribbon-substrate with such combination of texture and paramagnetic properties are based on the following operational hypothesis: increasing the stacking fault energy, E_{sf} , and as a consequence, cubic texture formation is possible due to developing strong compressive stress in the ribbon. This hypothesis is founded on the result we obtained earlier: after plasma deposition

[17–19] of NaCl-structure TiN coating onto the Ni–9.5 at. % W alloy ribbon provided a certain $d_{\text{Ni-W}}/d_{\text{TiN}}$ ratio of the ribbon and the coating thicknesses, substantial increase of the cubic texture degree was found in comparison with an initial ribbon after the same thermal treatment [20]. The source of the compressive stress in the TiN coated ribbon is evidently large difference between Ni–9.5 at. % W alloy ribbon and TiN coating FCC lattice parameters: $a_{\text{Ni-W}} \sim 3.56 \text{ \AA}$, $a_{\text{TiN}} \sim 4.27 \text{ \AA}$.

In the present work an attempt was made to obtain the cubic texture in the Ni–9.5 at. % W paramagnetic alloy ribbon due to dislocation high density, hence, high degree of stress generated in the ribbon as a result of substantial change of mechanical and thermal treatment regimes.

The "base technology" developed earlier [20] for production of ribbons from Ni–W alloys consisted of the following main steps (operations):

1. Deep purification of nickel and tungsten powders for removing gaseous contaminations using high temperature treatment in vacuum (Ni) or in recovering gaseous mixture Ar + 4 % H₂ (W).

2. Preparation of Ni–W alloys with different compositions (0–9.5 at. % W) using the following methods:

- a) arc melting in inert atmosphere with consequent quenching;
- b) vacuum melting;
- c) powder metallurgy.

3. Rolling the Ni–W ingots at room temperature with intermediate annealing treatments at $\sim 600^\circ\text{C}$ in Ar + 4 % H₂ and the final annealing at $\sim 1000\text{--}1300^\circ\text{C}$.

4. Surface structure modification of the Ni–W alloy ribbon by deposition of non-superconducting TiN coating using a plasma-arc method. The TiN coating may serve simultaneously as a buffer layer.

After development and practical realization of the "base technology" principles for preparation of ribbons from Ni–W alloys with high W content, the main direction of the investigations and developments was optimization of the main steps of the technology process. Namely, the following problems were under consideration:

1. The choice of the optimal technology for Ni–W alloys synthesis.

2. The choice of the optimal technology for remaking the Ni–W ingots into ribbons.

Two types of cold rolling were analyzed:

- a) "traditional rolling", i.e. alternating the relatively low number of cycles "defor-

mation-annealing" at intermediate temperatures;

b) "extreme rolling", i.e. realizing the large number of rolling acts with low deformation without annealing.

3. Optimization of the final annealing regimes for Ni–W alloy ribbons in the sufficiently wide temperature and time ranges in vacuum and inert medium.

4. Optimization of conditions for deposition of TiN coating and studying the conditions effect on the texture and properties of Ni–W alloy ribbons.

At once it is worth to be mentioned, that the problems of optimization for "traditional rolling" (2a) and TiN deposition conditions (4) were solved in general in our earlier work [20].

So, the present work is devoted to studying the possibility of preparation of textured ribbons from paramagnetic Ni–W alloys using the "extreme rolling" (2b).

In Fig. 1 the general scheme is shown for investigations and developments on the optimization of the technology process for preparation of the textured ribbon based on Ni–9.5 at. % W paramagnetic alloys. Two different study directions are considered: ones based on the abovementioned "traditional rolling" followed by TiN layer deposition ("right" branch), and ones based on "extreme rolling" ("left" branch).

The questions connected with cubic texture formation in the paramagnetic Ni–9.5 at. % W alloy ribbons as a result of optimizing the TiN layer plasma deposition regime were discussed earlier [20]. In the present work we consider a possibility to form the cubic texture under cardinaly changed conditions of mechanical and thermal treatment of the ribbons.

2. Samples and investigation technique

As to methodical aspects of the work, the following important reason is worth to be noted: in all the optimization stages of the ribbon production process (alloy synthesis, deformation and thermal treatment), the choice in favor of any technology process is possible only on the basis of studying structure and properties of the final product — the annealed ribbon.

To choose the optimal method of work-piece preparation for the following treatment, the ribbons of thickness $d_{\text{Ni-W}} \sim 100\text{--}150 \mu\text{m}$ were obtained from Ni–9.5 at. % W paramagnetic alloy using cold rolling with

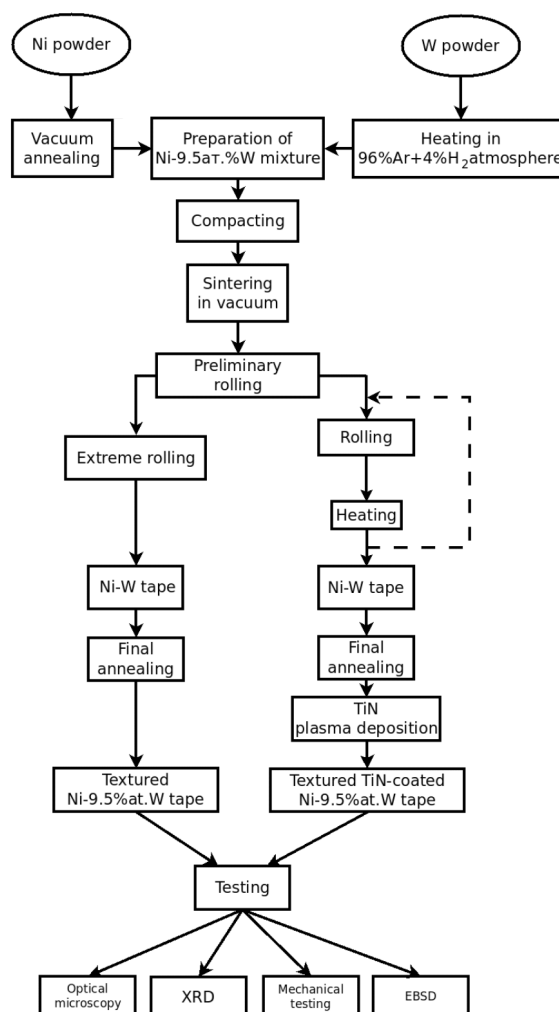


Fig. 1. A possible technology line-up for obtaining the high-tungsten Ni–W alloy based ribbons with cubic texture. (XRD means X-ray diffraction, EBSD means electron back scattering diffraction).

intermediate vacuum annealing steps ("traditional rolling", see Fig. 1).

Evidently, the data below concerning "optimal" regimes of thermal treatment are of preliminary character.

After the rolling, the ribbons were annealed in vacuum or in inert gas atmosphere in the wide temperature range (950–1150°C) during 1–4 h. The main investigation method was X-ray diffraction (XRD). Based on structure X-ray studying, the temperature and time dependences of the crystalline lattice parameter $a_{\text{Ni-W}}$, as well as the "texture parameter", the ratio I_{200}/I_{220} [20] were determined, where I_{hkl} — diffraction line intensity. The typical temperature dependences $a_{\text{Ni-W}}(T)$ and $I_{200}/I_{220}(T)$ for

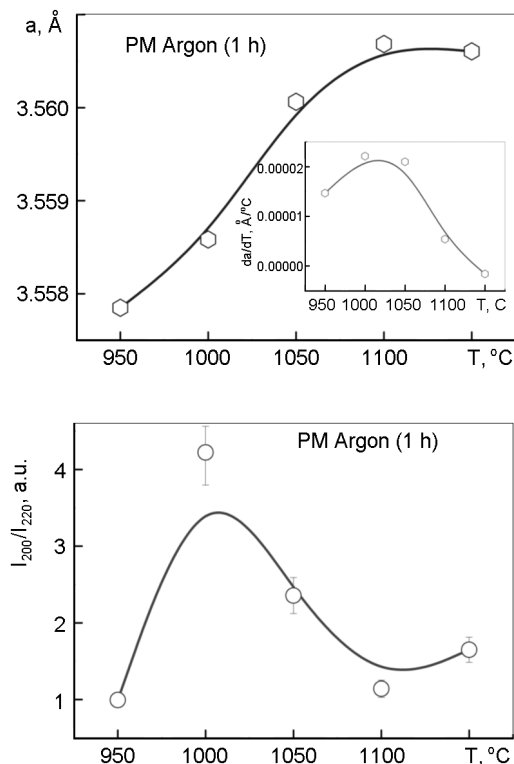


Fig. 2. Annealing temperature effect on the crystalline lattice parameter and I_{200}/I_{220} value for Ni-9.5 at. % W ribbons. Annealing time is 1 h.

the ribbon obtained by powder metallurgy method (PM) are shown in Fig. 2.

Variations of the dependences $a_{\text{Ni-W}}(T)$ and $I_{200}/I_{220}(T)$ at $T \sim 1000^\circ\text{C}$ are observed for the ribbons prepared by powder metallurgy, vacuum, and arc melting methods. These peculiarities indicate an intense recrystallization process and allow considering the temperature $T \sim 1000^\circ\text{C}$ as optimal for the final annealing.

The time dependences $a_{\text{Ni-W}}(\tau)$ and $I_{200}/I_{220}(\tau)$ for the ribbons made from Ni-9.5 at. % W alloy obtained by different methods are shown in Fig. 3.

The maximum on the plot $I_{200}/I_{220}(\tau)$ revealed for the minimum time interval $\tau = 1$ h, and extremely strong behavior of the plot $a_{\text{Ni-W}}(\tau)$ for the ribbon prepared from Ni-9.5 at. % W alloy obtained by the powder method are, obviously, connected with relatively high porosity of the initial workpiece.

These facts give all the reasons to consider that the optimal conditions for production of the textured ribbons from paramagnetic Ni-9.5 at. % W alloy are the follow-

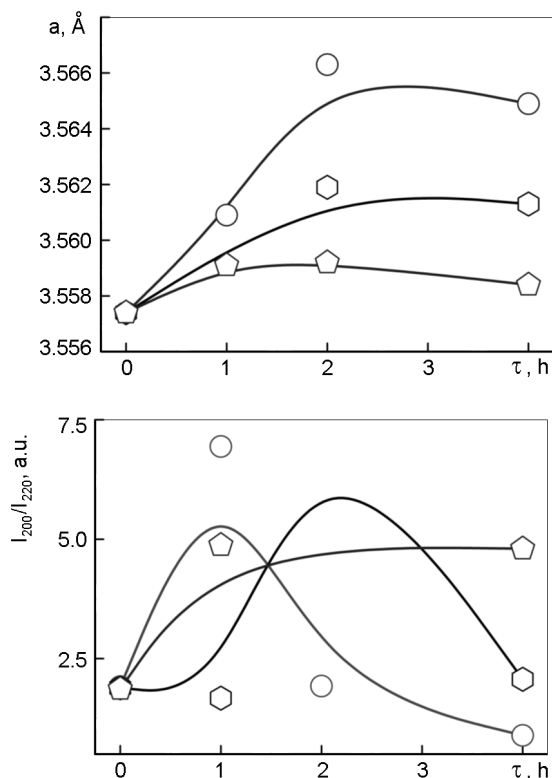


Fig. 3. Annealing time (in argon atmosphere at 1000°C) effect on the crystalline lattice parameter and I_{200}/I_{220} value for Ni-9.5 at. % W ribbons. Circle — powder metallurgy; pentagon — arc melting; hexagon — vacuum melting.

ing: the powder metallurgy as the alloy synthesis method; the final annealing temperature $T \sim 1000^\circ\text{C}$; the annealing time ~ 1 h.

3. Results and discussion

3.1. Choice of the optimal technology for remaking the Ni-W ingots into ribbons

As it was mentioned above, the idea of searching for new ways to obtain cubic textured ribbons from paramagnetic Ni-W alloys with high W content is based on an assumption about a possibility to increase the stacking fault energy E_{sf} under mechanical compressive stress. Probably, the necessary stress level can be achieved by optimization of deformation and thermal treatment of the ribbon. The scheme developed for preparation of ribbons with cubic texture from Ni-9.5 at. % W paramagnetic alloy is shown in Fig. 4.

For "extreme rolling" processing, two workpieces were made by the powder metallurgy method: a "little" ($2 \times 10 \times 50 \text{ mm}^3$),

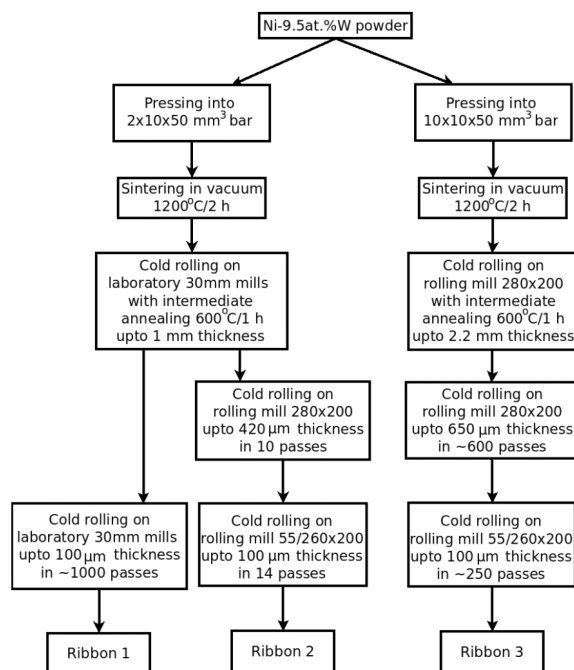


Fig. 4. The scheme of investigations on "extreme rolling" of Ni-9.5 at. % W alloys.

and a "big" ($10 \times 10 \times 50 \text{ mm}^3$). The deformation ways ("routes") of both workpieces are shown in Fig. 4 on left and right sides, respectively. The preliminary deformation was over at thickness 1 mm for the "little" and 2.2 mm for "big" workpiece. After an intermediate annealing ($600^\circ\text{C}/1 \text{ h}$), the cold rolling was carried out by different routes (Fig. 5):

1. Deformation of the "little" workpiece on the laboratory rolling mill with bowl diameter 30 mm, ~ 1000 passages; the ribbon final thickness $100 \mu\text{m}$ (*ribbon 1*).

2. Deformation of the "little" workpiece on the commercial rolling mill, 280×200 , 10 passages, ribbon thickness $420 \mu\text{m}$; rolling on the mill $55/260 \times 200$, 14 passages, ribbon thickness $100 \mu\text{m}$ (*ribbon 2*).

3. Cold rolling of the "big" workpiece consisted of deformation on the mill 280×200 , ~ 600 passages, and ribbon thickness $650 \mu\text{m}$; rolling on the mill $55/260 \times 200$, ~ 250 passages, and ribbon thickness $100 \mu\text{m}$ (*ribbon 3*). All the deformation diagrams are qualitatively similar: in the beginning part, the ribbon thickness decreased abruptly with increasing passage number N , then, the "deformation velocity" $\delta d/\delta N$ drops significantly (see the inset in Fig. 5). Significant quantitative differences are revealed in the plots curvature,

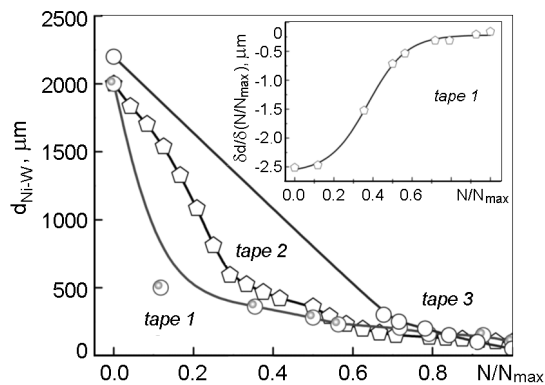


Fig. 5. The deformation diagram for workpieces of Ni-9.5 at. % W alloys. N is the number of passages, N_{max} is the maximum number of passages.

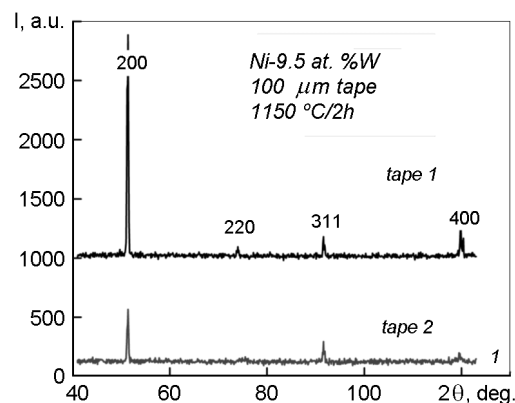


Fig. 6. X-ray diffraction patterns for Ni-9.5 at. % W ribbons obtained by two different technology "routes". The ribbon thicknesses and annealing conditions are the same.

and in the parts boundaries. Evidently, it is impossible to choose the certain deformation regime unambiguously only on the base of obtained data on the deformation dynamics of Ni-9.5 at. % W alloy under different rolling regimes. Such choice can be done only based on studying the ribbon texture variations as a result of the final high-temperature annealing (see also item 2.5).

3.2. Optimization of final annealing regimes for Ni-W ribbon

Obviously, for solving the multi-factor optimization problem of preparing the ribbon from paramagnetic Ni 9.5 at. % W alloy it is necessary to consider the effects of the following factors on the texture formation process:

1. Rolling "route" (see Fig. 4).
2. Final annealing temperature.

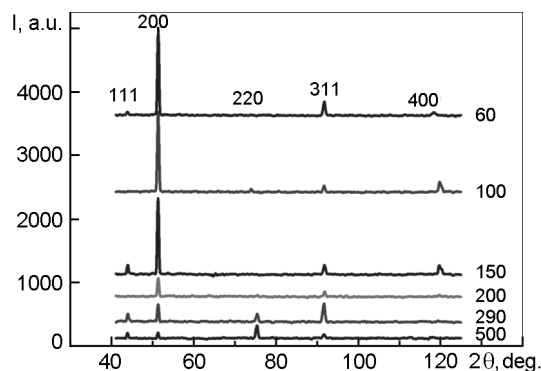


Fig. 7. X-ray diffraction patterns for Ni-9.5 at. % W ribbons prepared by the technology "route" No.1. Annealing conditions: 1150°C/1 h. Numerals near each X-ray diffraction pattern indicate the ribbon thicknesses in μm .

3. Time interval of the final annealing.

4. Environment in which the final annealing is carried out (vacuum, inert gas (argon) atmosphere).

To illustrate the strategy of searching for Ni-9.5 at. % W ribbon treatment optimal regimes, in Fig. 6 the X-ray diffraction patterns are shown for the ribbons obtained by deformation with the laboratory mill with roll diameter 30 mm, after about 1000 passages (*ribbon 1*), and with the commercial mill 280×200, after 10 passages, ribbon thickness 420 μm ; rolling with the mill 55/260×200, 14 passages (*ribbon 2*). In both cases the ribbon final thickness was $\sim 100 \mu\text{m}$. The (200) diffraction maxima intensities show that under other equal conditions (ribbon thickness, annealing temperature and time) the "route" No.1 provides the maximum possibility for formation of a perfect cubic texture.

The next problem was connected with the ribbon thickness optimization. In Fig. 7a number of X-ray diffraction patterns for Ni-9.5 at. % W ribbon are shown (the annealing conditions are the same as in Fig. 6): $60 \leq d \leq 500 \mu\text{m}$.

The dynamics of the diffraction pattern variation indicates a pronounced trend to intensity increase for (200) reflection I_{200} with decreasing ribbon thickness, i.e. increase the cubic texture degree. This conclusion is well illustrated by the dependences $I_{200}(d)$ and $I_{311}(d)$ shown in Fig. 8: I_{200} increases significantly as d decreases, while I_{311} is practically constant. The whole data shown in Figs. 6–8 give a possibility to

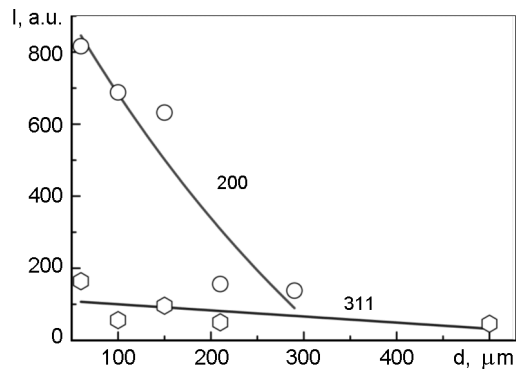


Fig. 8. (200) and (311) diffraction line intensities depending on the ribbon thickness for Ni-9.5 at. % W ribbons obtained by the technology "route" No.1. Annealing conditions: 1150°C/1 h.

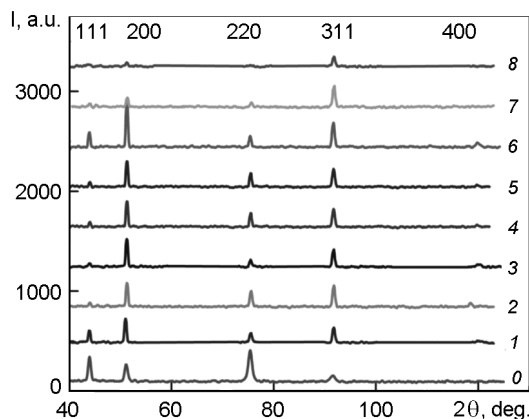


Fig. 9. X-ray diffraction patterns for Ni-9.5 at. % W ribbons prepared by the technology "route" No.3. Annealing conditions: 0 — without annealing; 1 — 1000 °C/2 h; 2 — 1150°C/1 h; 3 — 1150°C/2 h; 4 — 1200°C/0.5 h; 5 — 1200°C/1 h; 6 — 1200°C/2 h; 7 — 1300°C/0.5 h; 8 — 1300°C/2 h.

conclude, that the rolling by No.1 "route" on the rolling mill with small diameter of rolls and very large number of passages provides preparation of the ribbon with final thickness $\sim 50\text{--}100 \mu\text{m}$ and with enough perfect cubic texture.

The ribbon obtained from Ni-9.5 at. % W alloy using the "route" No.2, that is rolling using the commercial mill with large diameter rolls and small number of passages, at first sight, has less perfect cubic texture. However, additional investigations are necessary in order to open all potential abilities of this technology process. An undoubted advantage is the possibility to use standard commercial equipment and small

number of passages need for production of the ribbon with thickness $\sim 100 \mu\text{m}$.

Application of the "route" No.3 (see Fig. 4), i.e., cool rolling of the "large" ingot on the rolling mills with large diameter of rolls and large number of passages for preparation of the ribbon with $\sim 100 \mu\text{m}$ thickness gave no positive result. From the X-ray diffraction patterns shown in Fig. 9 it can be seen that annealing at temperatures $1000\text{--}1300^\circ\text{C}$ did not result in strong enough cubic texture.

5. Conclusions

The conception was developed on the possibility of obtaining the cubic texture $\{100\}\langle 001\rangle$ in the ribbon based on paramagnetic Ni–W alloys with high W content by means of creating the high level compressive stress resulting in increasing the stacking fault energy E_{sf} , and following high temperature annealing. It was established that the optimal method for obtaining workpieces for remaking into ribbons by rolling is the powder metallurgy. Based on the developed conception, two different ways were realized for production of ribbons based on paramagnetic Ni–9.5 at. % W alloys: deposition of thin TiN layers, and deformation of the ribbon in order to create high density of dislocations ("extreme" rolling). The optimal schemes for mechanical and thermal treatment of Ni–9.5 at. % W ribbons for 2nd generation HTSC-coated conductors (2G HTS) were developed including choosing the parameters for untraditional regimes of rolling, ribbon thickness, and annealing time, etc. The work was carried out with support of Science and Technology Center in Ukraine (Project STCU# P424).

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Одержання текстурованої стрічки на основі парамагнітних сплавів Ni–W з високим вмістом вольфраму

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Метою роботи є пошук шляхів одержання підкладки для ВТНП-покритих провідників другого покоління на основі парамагнітних сплавів Ni–W з високим вмістом вольфраму, що мають кубічну текстуру типу {001}<100>. В основу роботи покладено наступну робочу гіпотезу: підвищення енергії дефектів упакування E_{sp} , як наслідок, формування кубічної текстури можливо в результаті створення досить сильних стискаючих напруг у стрічці. На основі розвинених уявлень реалізовано два нових шляхи одержання кубічної текстури стрічки: у результаті плазмового осадження тонких шарів TiN ($a_{TiN} \ll a_{Ni-W}$) і в результаті проведення прокатки сплаву Ni–W в умовах, що забезпечують створення високої щільності дислокацій ("екстремальна прокатка"). Розроблено оптимальні схеми механічної й термічної обробки стрічки на основі парамагнітного сплаву Ni–9.5 at. % W, що включають вибір методу синтезу, параметрів режимів прокатки, товщини стрічки, температури й час відпалу.