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Quantitative analysis of deformation texture and primary recrystallization after inclined rolling and annealing of the $(Fe_{83}Ga_{17})_{99}B_1$ magnetostrictive alloy

Ivan R. Strizhachenko*[1], engineer
Irina V. Gervasyeva[2], Doctor of Sciences (Physics and Mathematics), leading researcher
Vasily A. Milyutin[3], PhD (Physics and Mathematics), senior researcher
Denis I. Devyaterikov[4], PhD (Physics and Mathematics), researcher
M.N. Mikheev Institute of Metal Physics of the Ural Branch of RAS, Yekaterinburg (Russia)

***E-mail: strizhachenko@imp.uran.ru**

[1]ORCID: https://orcid.org/0009-0009-5217-230X
[2]ORCID: https://orcid.org/0000-0001-8928-1707
[3]ORCID: https://orcid.org/0000-0002-5808-3959
[4]ORCID: https://orcid.org/0000-0002-6909-7678

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Abstract: The Fe–Ga alloy is a promising magnetostrictive material thanks to of the optimal combination of functional properties and relatively low price due to the absence of rare-earth elements in the composition. To obtain the maximum magnetostriction in Fe–Ga polycrystals, it is necessary to create a crystallographic texture with a predominance of the <100> direction, since the tetragonal magnetostriction constant is the largest. Traditional methods of thermomechanical treatment do not lead to the formation of such a texture in a bcc alloy. In this paper, for the first time, the authors propose to use inclined rolling to increase the proportion of favorable texture components. Warm rolling with a deformation degree of 70 % was carried out at angles of 0, 30 and 90° to the direction of hot rolling. The deformation texture was examined using X-ray texture analysis and the texture and structure of the material after recrystallization was analyzed by electron backscatter diffraction (EBSD) on a scanning electron microscope. Quantitative texture analysis was carried out using the orientation distribution function (ODF) method using the ATEX software. The volume fraction of some texture components was calculated. The study shows that a significant change in the deformation textures and primary recrystallization occurs during rolling at an angle of 90°. The sample after such rolling contains the largest amount of the planar component {100}. The study identified a relationship between the texture of deformation and recrystallization in Fe–Ga: to increase the proportion of components with the <001> crystallographic direction during recrystallization, the presence of planar components {111} in the deformation texture is necessary, which is associated with the predominant growth of favorable components in the deformation matrix with such a texture.

Keywords: Fe–Ga alloy; $(Fe_{83}Ga_{17})_{99}B_1$ alloy; texture quantitative analysis; inclined rolling; primary recrystallization; magnetostriction.

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INTRODUCTION

Alloys with high magnetostriction are widely used in the electrical industry. Actuators and linear motors, sound and ultrasound transducers, sensors for various purposes, devices for converting mechanical energy into electrical one, etc. are produced on their base. In recent years, Fe–Ga alloy, which has an optimal combination of properties and a much lower cost compared to the popular Fe–Dy–Tb alloy, is considered a promising replacement for traditional magnetostrictive materials [1]. For this reason, today a large number of studies on the Fe–Ga alloy have appeared. For example, methods to increase magnetostriction by creating an optimal phase composition, have been developed [2], phase transformations [3] and the reasons for abnormally high magnetostriction in the alloy have been studied in detail [4]. The most common form of use of magnetostrictive alloys (and many other soft magnetic alloys) is sheets, which are separated from each other by an insulation layer and then assembled into positions of a given shape and size. Such configuration allows creating volumetric devices, that can operate under conditions of increased reversal magnetization frequencies without large eddy current losses, which is especially important in the context of many magnetostrictive devices.

To produce Fe–Ga alloy sheets, rolling deformation followed by recrystallization annealing is usually used. It is very important in the process of thermomechanical processing to create a certain crystallographic texture necessary to achieve the best properties (magnetostriction). In the case of Fe–Ga, such texture is <100> // RD (rolling direction), at which the magnetostriction magnitude is maximum [5]. The problem is that the Fe–Ga alloy with a Ga content of up to 19 at. % (the first peak of the magnetostriction dependence on the composition [6]) used for rolling has a body-centered cubic (bcc) structure, in which it is quite difficult to obtain a large volume of grains with the <100> // RD orientation primary recrystallization. To achieve this goal, it is necessary to carry out a detailed study of the features of the formation of texture components, in the Fe–Ga alloy at various stages of thermomechanical treatment with varying modes. Such research has been actively conducted in recent years [7–9], but the problem has not yet been fully resolved. It is known that by selecting, rolling, and annealing modes, it is possible to increase significantly the content of the <100> // RD component in the final texture, but even in this case its proportion remains quite low [9; 10]. Moreover, in terms of engineering, rolling of the Fe–Ga alloy is complicated by its tendency to brittle fracture, so the binary alloy is often doped with a small amount of boron, which minimizes the problem [11]. There are studies demonstrating the possibility of creating a favourable texture with a predominance of the so-called Goss orientation (this is the name of the {110}<100> orientation), or an orientation close to it, due to secondary recrystallization, similar to the mechanism implemented in Fe–3%Si anisotropic electrical steel [12; 13]. However, this is a technologically difficult process, which requires, among other things, the introduction of additional inclusions to inhibit the normal growth of grains at certain annealing stages.

To solve the problem of creating a crystallographic texture, optimal for the formation of magnetic properties in the Fe–Ga alloys, further research is required, including the use of new non-standard approaches. One of such approaches is inclined rolling [14]. It consists in changing the angle of the cold rolling direction relative to the hot rolling direction. This allows making changes in the processes of grain nucleation and growth, which in some cases can lead to an increase in the amount of <100> orientation. Previously, this approach was used for Fe–Si bcc alloy rolling and showed its effectiveness [15–17]. Since this alloy is similar to Fe–Ga from the point of view of texture formation features, one can hope that inclined rolling will be effective in this case as well. Such experiments had not been carried out previously, so the idea of their implementation formed the basis of this paper.

To increase ductility and minimize the risk of fracture during rolling, an alloy doped with 1 at. % of boron, namely $(Fe_{83}Ga_{17})_{99}B_1$ was selected as a study material.

The purpose of this work is to study the influence of rolling at an angle on the crystallographic texture in the Fe–Ga alloy.

METHODS

An ingot of $(Fe_{83}Ga_{17})_{99}B_1$ composition (atomic percentages are used in the paper) was melted by induction in a protective argon atmosphere and poured into a copper mold using centrifugal casting. As was found earlier, such casting method allows reducing the number of pores in Fe–Ga ingots, and achieving a uniform distribution of chemical elements [18]. The addition of boron is intended to increase the ductility of the alloy, which will simplify the task of its plastic deformation. A cylindrical ingot with a height of 50 mm and a diameter of 14 mm was cut into three equal parts across the long side. The blanks were forged to a thickness of 6 mm and hot rolled to a thickness of about 2.7 mm. Forging and hot rolling were carried out at a temperature of 1000 °C. Then, using an electric spark machine, rectangles 8 mm wide and 20 to 30 mm long were cut from the resulting sheets at different angles to the hot rolling direction (Fig. 1).

The samples were rolled in several passes on a double rolling mill heated to 350 °C before each pass to minimize the risk of cracking. The overall degree of deformation using warm rolling was 70 %. The deformation texture was studied using a PANalytical Empyrean Series 2 diffractometer. The pole figures of the samples were obtained in a configuration with a focusing polycapillary lens.

The sample was fixed on a measuring table with three degrees of freedom. An X-ray tube operating at the characteristic wavelength of CoKα, λ=1.789 Å served as the radiation source. Based on the experimental pole figures {110}, {200} and {112}, Bunge orientation distribution functions (ODFs) were built using ATEX software[1]. Using a specially created program, volume fractions of specified orientations {hkl}<uvw>±10° were obtained from ODF data for deformed samples, in the same way that it was implemented in our previous work [19].

Recrystallization annealing of deformed samples was carried out in vacuum at a temperature of 900 °C for 30 min. Sample preparation for structural studies was carried out by mechanical grinding, and electropolishing in a 90 % $C_2H_4O_2$ + 10 % $HClO_4$ solution. The structure and texture of the recrystallized samples were studied using the electron backscatter diffraction (EBSD) method, on a FEI Quanta 200 scanning electron microscope using an EDAX attachment. On recrystallized samples subjected to chemical polishing, a zone of the same area was scanned at a relatively low magnification of ×100, which allows covering a larger number of grains and obtain better statistics. In total, using the EBSD method, an area of about 25 mm^2 was examined on each sample. Applying the TSL OIM Analysis program, the volume fractions of planes or directions in a plane, as well as the average grain diameter were determined.

RESULTS

Fig. 2 shows the ODF sections at an angle of φ2=45° with relevant information.

The ODF 45° section (Fig. 2 a) presents the orientations most characteristic of the deformation of bcc materials. It can be observed that in all three variants of inclined rolling (Fig. 2 b–d), orientations with an octahedral plane {111} parallel to the rolling plane (horizontal line at angle of Φ=54.7°) are predominantly present. Moreover, orientations with a cubic plane {100} parallel to the rolling plane (upper side of the orientation cube at Φ=0°) occur with significantly greater intensity. In this case, in the sample rolled transverse to the rolling direction (Fig. 2 c), there are significantly more orientations with {111} planes. The sum of the volume fractions of {111}<110> and {111}<112> in this sample is 6.9 %, and in samples rolled at angles of 0

[1] *Beausir B., Fundenberger J.-J. Analysis Tools for Electron and X-ray diffraction. ATEX-software. URL: www.atex-software.eu.*

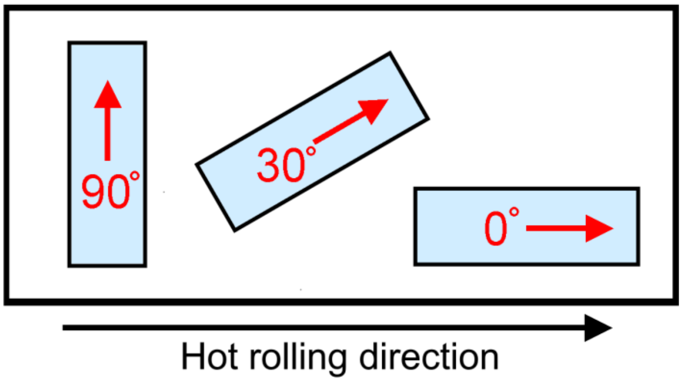


Fig. 1. *The scheme of cutting samples from a hot-rolled sheet at different angles to the hot rolling direction*
Рис. 1. *Схема вырезания образцов из горячекатанного листа под разными углами*
по отношению к направлению горячей прокатки.
Горизонтальная стрелка внизу рисунка указывает направление горячей прокатки

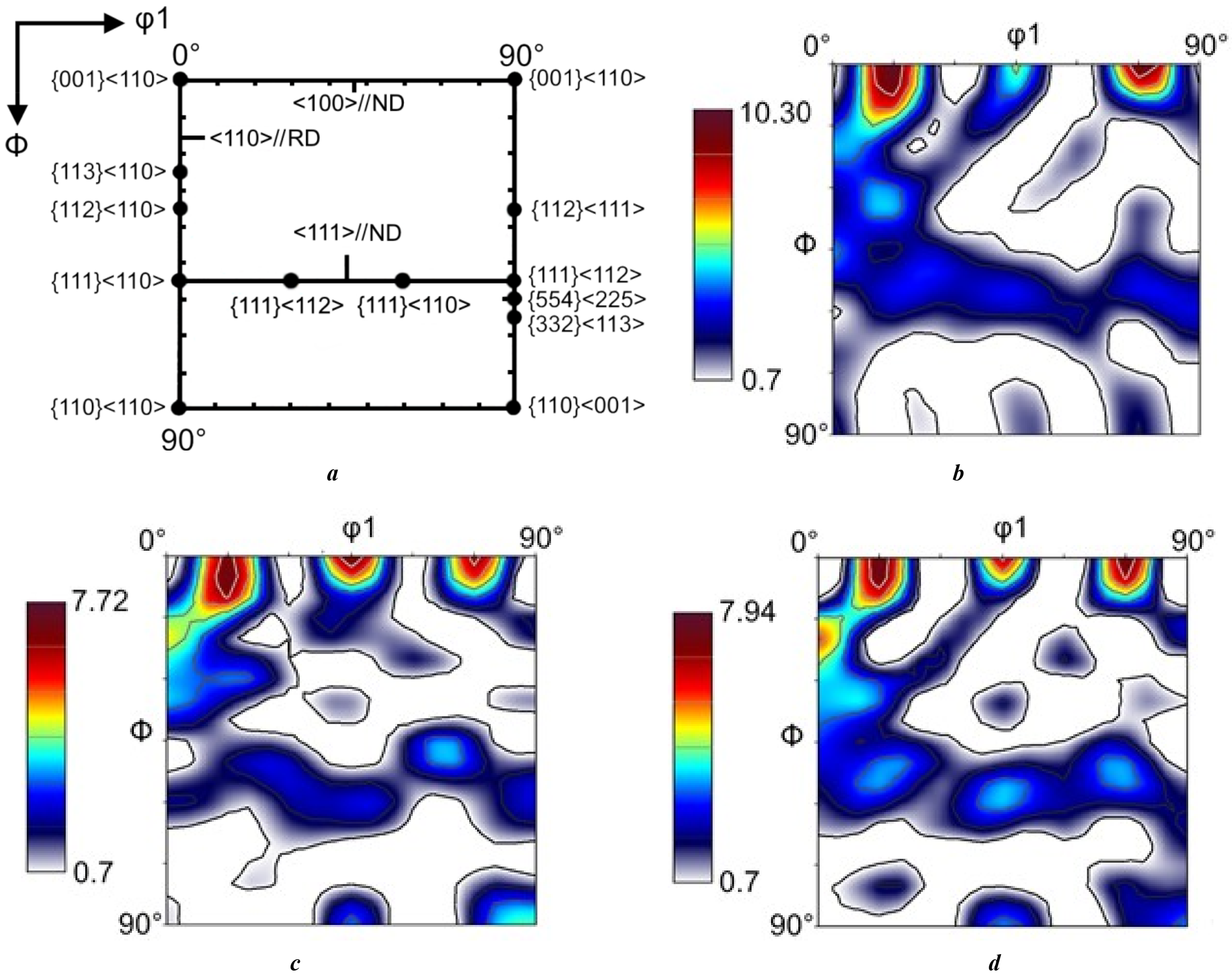


Fig. 2. *The location of the most important orientations (at φ2=45°) on the ODF specific to rolling and recrystallization of bcc metals (**a**) and ODF sections at φ2=45° for samples rolled at different angles to hot rolling direction: 0° (**b**); 30° (**c**); 90° (**d**)*
Рис. 2. *Положение наиболее важных ориентировок (при φ2=45°) на ФРО, характерных для прокатки и рекристаллизации ОЦК-металлов (**а**) и сечения ФРО при φ2=45° для образцов, прокатанных под разными углами к направлению горячей прокатки: 0° (**b**); 30° (**c**); 90° (**d**)*

and 30°, is 5.8 and 5.7 %, respectively. Moreover, the {111}<132> orientation is clearly visible in this sample (7.1 %, which is higher than for other variants) (Fig. 2, Table 1). As for cubic orientations, their volume fraction is significantly higher than of octahedral ones, and is approximately the same in all studied samples. In addition, Fig. 2 b and 2 c show a Goss orientation {110}<001> (lower right corner in the section), which, like cubic, is desirable for the formation of magnetic properties.

The study of recrystallized samples allows tracing the crystallographic texture evolution depending on the angle between cold and hot rolling. The recrystallization texture was studied by EBSD, since in this case the samples are less suitable for X-ray diffraction (XRD) studies. This is related to the fact that the grains of a certain orientation formed as a result of recrystallization are much larger in area than the cells in the deformation structure, which does not provide a good level of statistical sampling using the XRD method. The EBSD method makes it possible to analyze simultaneously the grain structure and crystallographic texture and suits well for the study of recrystallized samples. The results of the study by this method are shown in Fig. 3 in the form of fragments of orientation maps.

Upon visual analysis of the orientation maps, one can conclude that the sample rolled at an angle of 90°, contains a larger number of "red" grains, i. e., those in which the {100} planes lie in the sheet plane. Of course, to make an unambiguous conclusion, it is necessary to carry out a detailed quantitative analysis of the content of one or another texture component in the texture of samples. The results of such an analysis for the deformation texture and recrystallization texture are given in Table 1. Since the predominant directions are of greatest importance in terms of the formation of the Fe–Ga functional properties, their analysis was carried out in recrystallized samples with a scattering of ±15°.

It can be observed from Table 1 that the use of inclined rolling, significantly improves the texture of the samples in terms of the relation between the main crystallographic directions. Moreover, the effect when changing an angle between hot and warm rolling by 90° is more visible than by 30°. Compared to rolling along the hot rolling direction (0°), rolling at 90° leads to a twofold decrease in the undesired <111> // RD orientation, and a significant increase in the fraction of <100> // RD. At the same time, the proportion of grains with <110> // RD directions is unchanged under any of the studied rolling modes.

DISCUSSION

It is known that in the Fe–19%Ga alloy, the magnetostriction constant λ_{100} is maximal and is about 250 ppm. At the same time, λ_{110} is also quite high – about 150 ppm. Finally, λ_{111} is minimal and, according to various sources, varies from −20 to 0 [10]. To achieve high magnetostriction values in a polycrystalline sample, it is necessary to combine in its texture as many <100> orientations as possible and as few <111> orientations as possible, while the presence of <110> is acceptable. The work revealed the influence of the angle between hot and warm rolling on the number of these orientations in the $(Fe_{83}Ga_{17})_{99}B_1$ alloy.

To identify the reasons for changes in the recrystallization texture of samples rolled at different angles, it is necessary to analyze their deformation texture. Since the recrystallization annealing of all samples was carried out under identical conditions, it is the initial texture (in this case, the texture of the samples after final rolling), which is responsible for the observed phenomena. Changing the angle of the final rolling direction relative to the hot rolling direction is equivalent to rotating the sample around the normal direction (ND). This leads to changes in the rotation trajectories of various orientations, during plastic deformation. As can be observed, the deformation texture of a sample rolled transverse to the hot rolling direction contains significantly more {111} planar orientations.

It is well known from the literature [20] that in the Fe–3%Si bcc alloy, the grains disordered around the <110> axis by 27–35° have the boundaries of greatest mobility. This explains the growth of the {110}<001> component into a matrix with a strong {111}<112> orientation, during secondary recrystallization in electrical steel. Perhaps, in the Fe–Ga alloy, it is possible to find other favourable correlations for the growth of the {100}<hkl> component into any of the components of the {111} axial texture, for example, into the {111}<321> orientation predominant in the variant with rolling at an angle of 90° (Fig. 2 c). This will be the subject of further research.

The orientation criterion although is the main one, but not the only one, for the growth of grains with a favourable <100> // RD direction. It is also necessary to take into account the features of mesostructural elements, such as deformation bands, shear bands, and transition bands in the deformation structure. Moreover, an important role is played both by the relationship between the deformation texture components, and the distance between potential recrystallization nuclei with a certain orientation, as well as by other parameters. Therefore, identifying definite mechanisms responsible for the observed change in texture during recrystallization requires additional research. This work shows for the first time that rolling at an angle of 90° with respect to the hot rolling direction can be a promising tool for creating a favourable texture in a Fe–Ga alloy.

The authors studied samples cut at angles of 0, 30 and 90° relative to the hot rolling direction. There were no samples cut at other angles, such as 60°, due to technical limitations. All samples in the study were cut from the same ingot (to maintain equal experimental conditions for all samples). The size of the ingot and the hot-rolled sheets made from it did not allow cutting out another full-scale sample at an angle of 60°. It was not possible to reduce the size of the cut samples, since otherwise it would be problematic to carry out subsequent cold rolling. In connection with the prospects of the proposed approach, further research using other angles between the directions of hot and cold rolling, including 60°, is certainly necessary.

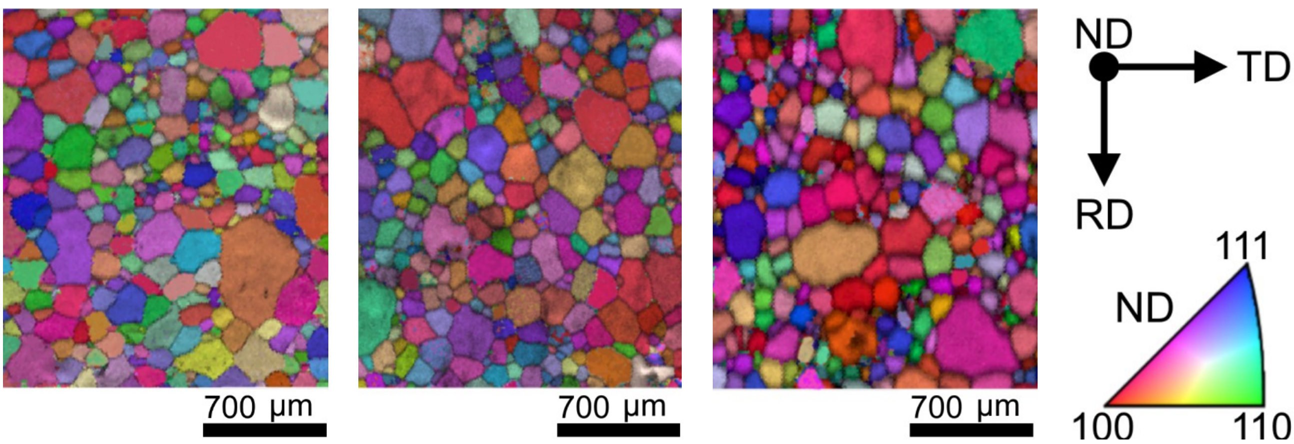
CONCLUSIONS

1. The $(Fe_{83}Ga_{17})_{99}B_1$ alloy melted by induction and cast using centrifugal casting, was successfully deformed by hot and warm rolling at different angles (0, 30, and 90°) to the hot rolling direction.

2. It is shown that the deformation texture after 70 % compression, alongside a noticeable amount of the {111} planar component, contains a large number of cubic orientations that are atypical for the deformation texture of bcc

Table 1. *Texture quantitative characteristics (volume fraction of components {hkl}<uvw>±10°, % in deformation texture and volume fraction of directions <uvw>±15° in recrystallization texture) and average grain size after recrystallization*
Таблица 1. *Количественные характеристики текстуры (объемная доля компонент {hkl}<uvw>±10°, % в текстуре деформации и объемная доля направлений <uvw>±15° в текстуре рекристаллизации) и средний размер зерна после рекристаллизации*

Studied characteristics	**An angle between cold and hot rolling directions, deg.**		
	0	**30**	**90**
Rolling deformation			
{100}<001> orientation fraction	1.6	3.4	2.1
{100}<230> orientation fraction	7.1	8.9	8.0
{100}<120> orientation fraction	7.5	8.9	8.4
{110}<001> orientation fraction	2.6	2.0	1.5
{111}<112> orientation fraction	3.4	3.0	3.0
{111}<110> orientation fraction	2.4	2.7	3.9
{111}<132> orientation fraction	6.3	5.7	7.1
Recrystallization 900 °C, 30 min			
Grain size (by the cutting line), μm	123.6	99.3	129.0
Grain size (by average diameter), μm	149.2	130.3	153.2
Fraction of <100> // RD directions	15.3	18.9	24.4
Fraction of <110> // RD directions	15.6	15.5	15.9
Fraction of <111> // RD directions	7.8	5.7	3.5



a *b* *c*

Fig. 3. *Fragments of orientation maps EBSD obtained from recrystallized samples.*
*Rolling at angles to the hot rolling direction: 0° (**a**), 30° (**b**), 90° (**c**).*
Sample orientation during scanning: RD – rolling direction; ND – normal direction to a sheet; TD – transverse direction
Рис. 3. *Фрагменты ориентационных карт EBSD, полученных с рекристаллизованных образцов.*
*Прокатка под углами к направлению горячей прокатки: 0° (**a**), 30° (**b**), 90° (**c**).*
Ориентация образцов при съемке: RD – направление прокатки; ND – направление нормали к листу; TD – поперечное направление

alloys. Rolling at an angle of 90° results in the largest amount of {111}<132> confined component within the {111} axial one.

3. It has been found that recrystallization annealing at 900 °C of samples rolled at an angle of 30 and 90° leads to a more favourable texture in terms of the formation of Fe–Ga properties, compared to traditional rolling (0°). The most visible effect is observed in the sample rolled at an angle of 90° to the hot rolling direction. This work shows for the first time that angular rolling is an effective tool to improve the primary recrystallization texture in Fe–Ga alloys.

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Количественный анализ текстуры деформации и первичной рекристаллизации при угловой прокатке и отжиге магнитострикционного сплава $(Fe_{83}Ga_{17})_{99}B_1$

Стрижаченко Иван Романович*[1], инженер
Гервасьева Ирина Владимировна[2], доктор физико-математических наук, ведущий научный сотрудник
Милютин Василий Александрович[3], кандидат физико-математических наук, старший научный сотрудник
Девятериков Денис Игоревич[4], кандидат физико-математических наук, научный сотрудник
Институт физики металлов имени М.Н. Михеева Уральского отделения РАН, Екатеринбург (Россия)

***E-mail: strizhachenko@imp.uran.ru**

[1]ORCID: https://orcid.org/0009-0009-5217-230X
[2]ORCID: https://orcid.org/0000-0001-8928-1707
[3]ORCID: https://orcid.org/0000-0002-5808-3959
[4]ORCID: https://orcid.org/0000-0002-6909-7678

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Аннотация: Сплав Fe–Ga является перспективным магнитострикционным материалом благодаря оптимальному сочетанию функциональных свойств и относительно низкой цены за счет отсутствия редкоземельных элементов в составе. Для получения максимальной магнитострикции в поликристаллах Fe–Ga необходимо создавать кристаллографическую текстуру с преобладанием направления <100>, поскольку наибольшей является константа тетрагональной магнитострикции. Традиционные методы термомеханической обработки не приводят к формированию такой текстуры в сплаве с ОЦК-решеткой. В работе впервые предложено использовать угловую прокатку с целью увеличения доли благоприятных текстурных компонент. Теплая прокатка со степенью деформации 70 % была реализована под углами 0, 30 и 90° по отношению к направлению горячей прокатки. Текстура деформации анализировалась с помощью рентгеновского текстурного анализа, а текстура и структура материала после рекристаллизации – методом дифракции обратно рассеянных электронов (EBSD) на сканирующем электронном микроскопе. Количественный анализ текстур проводился с помощью метода функции распределения ориентаций с использованием программного обеспечения ATEX. Количественно определена объемная доля некоторых компонент. Показано, что существенное изменение в текстурах деформации и первичной рекристаллизации происходит при прокатке под углом 90°. Образец после такой прокатки содержит наибольшее количество плоскостной компоненты {100}. Установлена зависимость между текстурой деформации и рекристаллизации в Fe–Ga: так, для повышения доли компонент с кристаллографическим направлением <001> при рекристаллизации необходимо присутствие в текстуре деформации плоскостных компонент {111}, что связано с преимущественным ростом благоприятных компонент в деформационной матрице с такой текстурой.

Ключевые слова: сплав Fe–Ga; сплав $(Fe_{83}Ga_{17})_{99}B_1$; количественный анализ текстуры; угловая прокатка; первичная рекристаллизация; магнитострикция.

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