THE INCREASING OF THE THERMAL STABILITY OF THE CRUSHED SUBSTRUCTURE OF STEELS

ПІДВИЩЕННЯ ТЕРМІЧНОЇ СТАБІЛЬНОСТІ ЗДРІБНЕНОЇ ПОЛІГОНІЗАЦІЙНОЇ СУБСТРУКТУРИ СТАЛЕЙ

DOI 10.15589/SMI.2018.02.18

Aleksandr M. Dubovoy
О. М. Дубовий, д-р техн. наук, проф.¹
oleksandr.dubovyj@nuos.edu.ua
ORC ID: 0000-0002-2843-1879

Tetiana O. Makruha
Т. О. Макруха, провідний фахівець¹
tmakruha@gmail.com
ORC ID: 0000-0001-8841-1699

Oleksandr V. Chechel
О. В. Чечель, канд. техн. наук, наук. співроб.²
achechel@trestmag.kiev.ua
ORC ID: 0000-0002-0175-714X

¹Admiral Makarov National University of Shipbuilding, Mykolaiv
²Frantsevich Institute for Problems of Materials Science of NAS of Ukraine, Kyiv

Abstract. The influence of the combined deformation on the thermal stability of the poligonization substructure of the technically clean iron and steels are shown in the article. So, the purpose of the work is the increasing of the thermal stability of the poligonization substructure by combined deformation of iron, steel 20, 45, У8, 12Х13 and 40Х. The possibility of the increasing hardness on the maximum indexes are investigated and installed the optimum magnitude and species of the deformation and modes, namely temperature and time of the exposure, of the heat treatment. The possibility of the thermal stability of the poligonization substructure during pre-recrystallization heat treatment of the Fe and carbon and alloyed steels by using combined deformation is established. The decreasing of the sizes of the regions of the coherent scattering of the values of the physical and mechanical properties, such as hardness, is proved. Indexes of the quantities of nanoscale substructure elements and angle of orientation of the subgrains are calculated by methods of the harmonic analysis. The combined deformation allows the using this method for machine parts and appliances. It was shown that pre-crystallization thermal treatment allows to form a substructure with nanosized elements of size 80 nm (steel 45) in a combination of deformed steels, and their quantity can reach 75 % of the total number of structural components with a maximum angle of divergence of subgrains 2.16º (technically pure iron).

Keywords: combined deformation; problem of Material Science; subgrains; nanostructure materials.

Анотація. Розглянуто вплив комбінованої деформації на термічну стабільність технічно чистого заліза, сталей 20, 45, У8, 12Х13 та 40Х шляхом комбінування різновидів та величин деформації. Метою роботи є підвищення термічної стабільністі полігоналізаційної субструктури технічно чистого заліза, сталей 20, 45, У8, 40Х та 12Х13 шляхом комбінованої деформації. Експериментально досліджено вплив розміру областей когерентного розсіювання, кількості наноструктурних елементів та кута деорієнтації субзерен на фізико-механічні властивості, а саме твердість заліза та сталей. Установлена можливість термічної стабільності полігоналізаційної субструктури в процесі переддекристалізаційної термічної обробки після попередньої комбінованої деформації, а саме поєднання динамічної деформації на 30 % та статичної деформації на 30 % з наступною переддекристалізаційною термічною обробкою за температури 500 °C. Досліджено розміри областей когерентного дослідження та встановлено, що найменші розміри спостерігаються при переддекристалізаційній термічній обробці, яка забезпечує максимум твердості. Показано, що переддекристалізаційна термічна обробка дозволяє формувати в комбіновані деформованих сталях субструктуру з нанорозмірними елементами величиною 80 нм (сталь 45), а кількість їх може досягати 75 % від загальної кількості структурних складових з максимальним кутом деорієнтації субзерен 2,16º (технічно чисте залізо).
Ключові слова: комбінована деформація; проблеми матеріалознавства; субзерна; наноструктурні матеріали.

Аннотація. Рассмотрено влияние комбинированной деформации на термическую стабильность технически чистого железа, сталей 20, 45, У8, 40Х и 12Х13 путем комбинирования разновидностей и величины деформаций. Целью работы является повышение термической стабильности полигонизационной субструктуры технически чистого железа, сталей 20, 45, У8, 40Х и 12Х13 путем комбинированной деформации. Экспериментально исследовано влияние размера области когерентного рассеивания, количества наноструктурных элементов и угла разориентировки субзёрёна на физико-механические свойства, а именно твердость железа и сталей. Установлена возможность термической стабильности полигонизационной субструктуры в процессе предкристаллизационной термической обработки после предыдущей комбинированной деформации, а именно сочетание динамической деформации на 30 % и статической деформации на 30 % с последующей предкристаллизационной термической обработкой при температуре 500 °С. Исследованы размеры областей когерентного рассеивания и установлено, что наименьшие размеры наблюдаются при предкристаллизационной термической обработке, которая обеспечивает максимум твердости. Показано, что предкристаллизационная термическая обработка позволяет формировать в комбинированно деформированных сталях субструктуру с наноразмерными элементами величиной 80 нм (сталь 45), а количество их может достигать 75 % от общего количества структурных составляющих с максимальным углом разориентировки субзёрёна 2,16° (технически чистое железо).

Ключевые слова: комбинированная деформация; проблемы материоведения; субзерна; наноструктурные материалы.

References


Problem statement. The problem of modern machine building is increasing of the reliability and work life of machine parts and mechanisms. This requires constant improvement, the application of new materials. The operational characteristics of the machine parts as a whole, their work life are determined predominantly by the physical and mechanical properties of the metals and alloys from which they are made. For solving this problem, mainly steel and alloys with increased strength, in particular hardness, are used. The increase of physical and mechanical parameters is possible due to pre-recrystallization heat treatment, which ensures the formation of a crushed substructure and nanostructure inclusive [1–3]. Pre-recrystallization heat treatment provides increased hardness due to the formation of polygonal substructure. However, further heating or shutdown at a temperature close to the initial recrystallization threshold leads to the leveling of the resulting results due to the development of the processes of harvest polygonization and recrystallization. The dislocation boundaries of sub-lands, which are formed during polygonization, are mobile and at elevated temperatures easily move, while the size of sub-grains increases and, as a result, hardness, strength decreases. All this makes it impossible pre-recrystallization heat treatment of large parts (more than 10 mm).

Latest research and publications analysis. In recent years, considerable progress has been made in the creation of nanostructured materials. Particular attention is paid to the methods of intensive plastic deformation (IOP). This group of methods for obtaining materials is based on conducting large-scale plastic deformation (>90 %) under high applied pressure at relatively low temperatures [4, 5]. In such conditions of deformation there is a grinding of microstructural elements in metals and alloys to a nanoscale size. The methods of IOP allow to obtain impervious metallic nanostructured materials. However, the range of sizes of sub-grained nanomaterials, as a rule, is greater than 100 nm. The structure obtained in the IOP differs greatly in nonequilibrium due to the low density of free dislocations and, mainly, the high-angle nature of the boundaries of sub-grains [4, 5].

Since IOP methods are characterized by high cost, labor-intensiveness and complexity of equipment, and are suitable only for details of a small section (up to 10 mm), one of the ways of solving this problem may be the use of pre-recrystallization thermal treatment of materials. It provides the opportunity to form a polygonal substructure [4]. As established in [1, 5], pre-recrystallization thermal treatment of statically deformed specimens by 75 % from Y8 steel and technically pure iron at a temperature that corresponds to the temperature threshold of recrystallization leads to an increase in hardness by 20 and 30 %, respectively, due to the formation of a crushed polygonization substructure, the maximum value of which is observed at a 2 minute lengthening time. Hardness decreases with increasing the aging time or when the temperature rises due to the increase in the size of the sub-grains, which indicates the instability of the substructure at elevated temperature, [6] has established the possibility of thermal stabilization of the polygonal substructure in the process of pre-recrystallization thermal treatment of technically pure iron and nickel for 20...70 and 10...60 minutes respectively, which consists of conducting a cold dynamic deformation of 30 % and a subsequent static deformation of 30% and pre-recrystallization heat treatment. It was found that increasing the thermal stability of the polygonal substructure of steels 20 and 45 is possible by combining dynamic and static deformations in total by 60 % and by subsequent pre-recrystallization thermal treatment, respectively, within 10...60 min and 5...60 minutes, while the hardness decreases somewhat, but remains higher compared with the state after deformation by 14 and 27 % respectively.

Separation of previously unsolved parts of the general problem. However, the influence of the magnitude and varieties of combined deformation on the thermal stability of the polygonal substructure is not yet sufficiently studied.

THE ARTICLE AIM — increasing of the thermal stability of the polygonization substructure of technically pure iron, steels 20, 45, Y8, 40X and 12X13 by the combined deformation.

Methods, object and subject of research. Since iron is the base of steels and castings, which today make up about 90 % of all structural materials used in technology and everyday life, then in the further research used technically pure iron grades E12 (GOST 3836–83); steels 20, 45 (GOST 1050–88); steel Y8 (GOST 1435–99); steel 40X (GOST 2591–2006) and steel 12X13 (GOST 2590–2006).

Annealed at 800 °C for 1 hour of iron samples of 6×6×8 mm were subjected to cold dynamic deformation by impact cyclic action to a given deformation value. Static deformation was carried out using a hydraulic press Losen Housen WLRK (Dusseldorf, 35 t) with a load of 20 t. Cold rolled was carried out in a laboratory horizontal position. Thermal treatment of samples was carried out in a laboratory electric oven CHOJ-1.6.2.0.08/9-M1. Hardness HV, was determined on a Vickers type device with a load of 5 kg indenter (DSTU ISO 6507-4: 2008), 10 measurements were made for each experimental point. The analysis of the structure of deformed and heat-treated specimens was carried out using an Optical Metallographic Microscope using the Delta Optical HDCE-20C Digital Camera equipped with the Scopeimage 9.0 image processing software and the scanning electron microscope ZEISS Gemini SEM 500. The size of the coherent X-ray scattering regions) were determined by the Sherrer formula and the method of harmonic analysis of the diffraction profile. The shooting of the diffractograms was carried out according to the reflection on the DРОН-3.0 device.

The object of the research are technically pure iron (E12), carbon (20, 45, U8) and alloy (40X, 12X13) steel.

The subject of the research are the regularities of the process of formation of a crushed po-
lygonal substructure with increased physical and mechanical properties and its thermal stabilization in plastic-deformed, technically pure iron and steels as a result of pre-crystallization thermal treatment.

**Basic material.** For annealed samples of technically pure iron, cold rolling was performed at 40 %, after which the sample was subjected to a static deformation of 40 %. Subsequently, the samples were subjected to pre-recrystallization heat treatment at a temperature of 500 °C with an endurance of up to 60 minutes. The results of the influence of pre-crystallization thermal treatment on hardness are shown in Fig. 1.

From Fig. 1 it can be seen that the dependence of hardness on the duration of pre-recrystallization heat treatment is extreme. The highest value of hardness of 2.08 GPa is observed at an endurance of 1 min, which is 19 % more than after rolling; and by 12 percent more than after a combination of rolling and static deformation.

Stabilization of the substructure, judging by the hardness, occurs in the interval between 1.5 and 10 minutes, then the hardness decreases. At 60 minutes of exposure, it is 1.91 GPa and approaches the value that corresponds to the hardness value after the combined deformation. Thus, there is no significant stabilization of the substructure.

To determine the possibility of combining rolling and static deformation, an experiment with a total deformation of 60 % was conducted, namely, samples of technically pure iron deformed by 30 % in the rolling mill, and then by another 30 % in the hydraulic press. For deformed samples, pre-crystallization heat treatment was performed at a temperature of 500 °C, the results of the study are shown in Fig. 2.

From Fig. 2 it can be seen that the dependence of hardness on the duration of pre-crystallization heat treatment is also extreme. The highest hardness is observed 2.33 GPa, which is 25 % more than after rolling; and by 15 % more than after a combination of rolling and stiff deformation.

Stabilization of the substructure occurs in the interval between 7 and 20 minutes, the hardness at this is 1.71 GPa, which is only 4 % more than after combined deformation in total by 60 %.

Next, the possibility of combining dynamic deformation with the following static deformation was checked after changing the direction to a 90° angle: for annealed samples of technically pure iron, dynamic deformation was performed with a shock cyclic action of 40 %, after which the sample was reversed by 90° and subjected to a static deformation of 40 % with hydraulic press. Thus the total value of the deformation was 80 %. Subsequently, pre-crystallization heat treatment was carried out at a temperature of 500 °C with a shutter speed of 60 minutes. The results of the influence of pre-recrystallization thermal treatment on hardness are shown in Fig. 3.

Fig. 3 shows that the dependence of hardness on the duration of exposure in the pre-crystallization heat treatment is also extreme. At an endurance of 5 minutes, the highest hardness is observed 2.33 GPa, which is 25 % more than after rolling; and by 15 % more than after a combination of rolling and stiff deformation.

A characteristic feature of such processing is 2 sites with a stabilized structure: the first — from 7 to 20 minutes, the second — from 30 to 60 minutes. Combining in the range of 7 to 20 minutes makes sense, because the hardness increase relative to the total combined deformation is 11 %, in contrast to the endurance from 30 to 60 minutes, where the gain is only 6 %.

Combining dynamic deformation by 40 % and static deformation by 40 % at an angle of 90° is more promising due to greater hardness.

As a combination of dynamic deformation with a static angle 90° allows for greater hardness than combining with rolling, but complicated for implementation, therefore, in order to reduce the number of operations, it is decided to combine two static deformations at an angle of 90° relative to one another. Pre-crystallization heat treatment was carried out at a temperature of 500 °C with an exposure of 1 to 60 minutes. The results of the study are shown in Fig. 4.

![Fig. 1](image.png)

**Fig. 1.** The effect of combined deformation (rolling on 40 % + static deformation by 40 %) and the following pre-crystallization thermal treatment on the hardness of technically pure iron: 1 — hardness after rolling; 2 — hardness after rolling and subsequent static deformation; 3 — hardness after combined deformation and subsequent heat treatment

![Fig. 2](image.png)

**Fig. 2.** The effect of combined deformation (rolled on 30 % + static deformation by 30 %) and the following pre-crystallization thermal treatment on the hardness of technically pure iron: 1 — hardness after rolling; 2 — hardness after rolling and subsequent static deformation; 3 — hardness after combined deformation and subsequent heat treatment
ment, the polygonization substructure is stabilized, since the hardness remains at one level with an elongation of 20 to 60 minutes.

The determination of the effect of hot dynamic deformation at 880 °C on the stability of the polygonal substructure was carried out in the same manner as in [6] (dynamic deformation of 30 % and static deformation by 30 %). The maximum hardness of 2.22 GPa was reached at 10 minutes, after 60 minutes the hardness decreased to the initial values after the combined deformation is likely due to the process of dynamic polygonization.

A combination of warm dynamic deformation (300 °C) followed by cold static deformation of technically pure iron was studied. Hardness after warm dynamic strain was 1.95 GPa, after combined deformation — 2.04 GPa. After that, pre-recrystallization heat treatment was carried out at a duration of 10 minutes, which was chosen based on previous experiments. Hardness after heat treatment was 2.08 GPa, which is only 2 % more than hardness after combined deformation.

Therefore, in the following studies, the optimal type of deformation is the combination of cold dynamic 30 % and static 30 % deformation (60 % total) is chosen, since such combination provides high hardness indexes, allows to stabilize polygonization substructure of purely technical iron.
pure iron, the method is simple in its execution. Then under the combined deformation we mean this method — combining dynamic deformation by 30 % and static deformation by 30 %.

To determine the dependence of the amount of carbon on the hardness of the combined deformed alloys after pre-crystallization heat treatment, samples from carbon steels 20, 45 and steel Y8 with a total deformation value of 60 %.

The investigated carbon steel has a ferrite-perlite and perlite structure. This, as a rule, leads to the localization of deformation and the formation of an inhomogeneous structure. Therefore, to ensure a uniform distribution of carbides, steel was subjected to pre-heat treatment — annealing. The hardness of steel 20 after annealing at a temperature of 850 °C for 60 minutes is 1.45 GPa, steel 45 (810 °C) — 1.7 GPa, steel Y8 (750 °C) — 1.87 GPa. After combined deformation, the hardness of the samples was: in the steel 20 — 1.97 GPa, in the steel 45 — 2.24 GPa, in the steel Y8 — 2.57 GPa.

After the combined deformation, pre-crystallization heat treatment was carried out at a temperature of 500 °C with an endurance of up to 60 minutes to verify the possibility of stabilizing the polygonal substructure. The results of the effect of the duration of the pre-crystallization heat treatment endurance are shown in Fig. 6.

Fig. 6 shows that increasing the concentration of carbon contributes to increased hardness. The greater the carbon, the more so in the steel solid cementite, which causes the growth of hardness. The duration of exposure to obtain the highest hardness values is reduced. It is also evident that increasing the thermal stability of the polygonal substructure of steel 20, 45 and Y8 can be combined by dynamic and static deformation by a total of 60 % and the subsequent pre-crystallization heat treatment according to 10...60 minutes, 5...60 minutes and 1...60 minutes. Also, from Fig. 6 it is seen that, with increasing amount of carbon, the duration of exposure decreases, which provides the maximum value of hardness.

To determine the dependence of the influence of doping elements on the hardness of the combined deformed alloys after pre-recrystallization thermal treatment, steel 40X and 12X13 were investigated.

Alloy steel was subjected to preliminary annealing, hardness after which was: steel 40X — 1.86 GPa, steel 12X13 — 2.02 GPa.

The hardness after the combined deformation in total by 60 % was: steel 40X — 2.38 GPa, steel 12X13 — 2.47 GPa.

After the combined deformation, pre-recrystallization heat treatment was carried out at a temperature of 500 °C with an endurance of up to 60 minutes to verify the possibility of stabilizing the polygonal substructure. The results of the effect of the duration of the pre-crystallization heat treatment are shown in Fig. 7.

Fig. 7 shows that with the increase in the amount of chromium in steel — the hardness of combined deformed samples increases after pre-recrystallization heat treatment.

Thus, the combined deformation of the samples, which consists of the previous cold dynamic deformation and the subsequent static deformation, is likely to contribute to the formation of sub-regions with an increased angle of divergence. It is obvious that an additional static deformation increases the number of structural imperfections in the form of dislocation crossings.

It is known for example [5] that the movement of dislocations is prevented by the boundaries of sub-grains, particles of another phase, concentration heterogeneity, structural imperfections (dislocations), fluctuations in the lattice, associated with uneven distribution of energy and impurities.

Also, dislocations that were introduced during deformation are blocked by impurity atoms, so at the next load, these dislocations do not participate in sliding, they inhibit newly formed dislocations or parts of dislocations that are unlocked after deformation loading. Atoms of penetration in metals cause more tetragonal and lead to a sharp increase in strength.

Thus, the stabilization of the substructure of technically pure iron and steels is to reduce the mobility of the boundaries of sub-grains by creating triple nodes (crossings) of dislocations and inhibition of dislocations by admixing atoms, predominantly those dissolved by penetration (in technically pure iron — 0.009 % C).

As in the course of the study, a significant change in the physical and mechanical properties of metals and
alloys [7], in particular hardness, was established, therefore the scientific interest is the change in the substructure, which led to changes in properties, namely the size of the coherent scattering regions, the number of nanostructured elements, and the angle of subregion orientation.

Since the CSR corresponds to the internal ordered grain region and does not include highly distorted boundaries, the size of the CSR is identified with the average grain size (sub-grains) [8].

Determination of the average size of coherent scattering regions of deformed, technically pure iron was carried out in three ways: according to Scherrer’s formula, using the method of harmonic analysis of the shape of the diffraction profile and the method of electron microscopy (Table 1).

From the data presented in Table 1, it can be seen that the size of CSR after pre-recrystallization heat treatment at the appropriate time of exposure decreases in comparison with the deformed state of the metal. Also, the size of CSR correlates with the values of hardness: the more hardness, the smaller the size of the CSR. This indicates that an increase in the hardness values results from the pre-recrystallization heat treatment due to the crushing of the substructure of the pre-deformed metal and confirms the validity of the conclusions regarding the stabilization of the polygonal substructure with a change in hardness.

Determination of the average size of coherent dispersion of carbon steels 20, 45 were also conducted using the Sherrer formula.

From the data given in Table 2 it can be seen that in the heat treatment, which provides maximum hardness, the size of the CSR decreases to 119 nm.

Subsequently, the average sizes of sub-steel in steel 45 were determined. The results are given in Table 3.

The data in Table 3 indicate that the application of combined deformation, which consists of a dynamic 30 % and a static of 30 %, followed by pre-crystallization heat treatment at a temperature of 500 °C for 2 minutes, provides a subzero size of 80 nm.

The average size of the CSR for the steel Y8 was also determined by Scherrer's mold and by scanning electron microscopy. Fig. 8 shows the microstructure of the treated steel U8 with an increase of 20,000 times.

Figure 8 shows that there is a crushing of substructural elements after pre-recrystallization thermal treatment with respect to the deformed state, which confirms the definite dimensions of CSR by methods of X-ray diffraction analysis (Table 4), according to which their reduction after pre-crystallization thermal treatment relative to the deformed state occurs. This is a direct proof of the crushing of the substructure of plastically deformed steel Y8 in the process of pre-crystallization thermal treatment.

The results of the determination of the average size of CSR (subgrains) steel Y8 are given in Table 4.

These data indicate that combined deformation and pre-recrystallization thermal treatment, which provides maximum hardness values, ensures crushing of substructural elements, in some cases, to nanoscale size.

These data indicate that combined deformation and pre-recrystallization thermal treatment, which provides maximum hardness values, ensures crushing of substructural elements, in some cases, to nanoscale size.

In the table 5 shows a change in the average size of the OCP alloy steel 40X after deformation and deformation and the pre-recrystallization heat treatment.

Data of the table 5 indicate that the combined deformation followed by pre-crystallization thermal treatment provides crushing of the substructure after pre-recrystallization thermal treatment relative to the deformed state.

In table 6 shows a change in the average size of CSP of steel 12X13 after deformation and deformation and pre-recrystallization heat treatment.

**Table 1. Average size of CSR, combined deformed samples of technically pure iron before and after pre-recrystallization heat treatment**

<table>
<thead>
<tr>
<th>Processing mode</th>
<th>Hardness, GPa</th>
<th>Size of CSR, nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>1.65</td>
<td>190</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 10 min</td>
<td>2.22</td>
<td>125</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 60 min</td>
<td>2.12</td>
<td>175</td>
</tr>
</tbody>
</table>

**Table 2. Average size of CSR, combined deformed samples of steel 20 before and after pre-recrystallization heat treatment**

<table>
<thead>
<tr>
<th>Processing mode</th>
<th>Hardness, GPa</th>
<th>Size of CSR, nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>1.97</td>
<td>145</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 5 min</td>
<td>2.32</td>
<td>120</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 60 min</td>
<td>2.28</td>
<td>190</td>
</tr>
</tbody>
</table>

**Table 3. Average size of CSR, combined deformed samples of steel 45 before and after pre-recrystallization heat treatment**

<table>
<thead>
<tr>
<th>Processing mode</th>
<th>Hardness, GPa</th>
<th>Size of CSR, nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>2.24</td>
<td>110</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 2 min</td>
<td>2.55</td>
<td>80</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 60 min</td>
<td>2.44</td>
<td>180</td>
</tr>
</tbody>
</table>

**Table 4. Average size of CSR, combined deformed samples of Y8 before and after pre-recrystallization heat treatment**

<table>
<thead>
<tr>
<th>Processing mode</th>
<th>Hardness, GPa</th>
<th>Size of CSR, nm*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>2.57</td>
<td>130/190</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 2 min</td>
<td>3.20</td>
<td>115/150</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 60 min</td>
<td>3.00</td>
<td>130/160</td>
</tr>
</tbody>
</table>

*The w/o indicates the size of the CSR, which is determined using the Scherrer formula and by scanning electron microscopy.
Table 5. Average size of CSR, combined deformed samples of steel 40X before and after pre-recrystallization heat treatment

<table>
<thead>
<tr>
<th>Processing mode</th>
<th>Hardness, GPa</th>
<th>Size of CSR, nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>1.86</td>
<td>130</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 2 min</td>
<td>2.53</td>
<td>120</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 60 min</td>
<td>2.52</td>
<td>120</td>
</tr>
</tbody>
</table>

Table 6. Average size of CSR, combined deformed samples of steel 12X13 before and after pre-recrystallization heat treatment

<table>
<thead>
<tr>
<th>Processing mode</th>
<th>Hardness, GPa</th>
<th>Size of CSR, nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>2.02</td>
<td>120</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 2 min</td>
<td>3.00</td>
<td>100</td>
</tr>
<tr>
<td>Combined deformation and heat treatment: 500 °C, 60 min</td>
<td>2.83</td>
<td>110</td>
</tr>
</tbody>
</table>

Data of the table 6 indicate that the combined deformation followed by pre-crystallization heat treatment provides the size of the CSR 100 nm, since the Sherrer formula defines only the mean value of CSR, we can say that such treatment provides 12X13 steel of nanoscale size of the substructure.

Technically pure iron and steel deformed by the combined method are characterized by certain features of the structure, which differs significantly from the structure of these materials which were statically deformed, which is conditioned not by equilibrium conditions of their formation (high rate of deformation). The structure determines their operational properties, for it characterized by increased concentration of various defects, which leads to the formation of nanostructured elements of the substructure. Properties of materials to a large extent depend on the relative number of nanostructured elements, therefore, the determination of the fraction of the nanostructural component allows a certain degree of prediction of the properties of materials [9, 10].

In the table 7 shows the relative number of nanostructured elements calculated by the method of approximation of the diffraction profile for deformed technically pure iron and steels before and after pre-recrystallization heat treatment.

The above data is shown in Table 7 indicate that the maximum size of the diverting angle provides combined deformation with a heat treatment that provides maximum hardness. At 60 minutes, the orientation angle decreases slightly relative to the maximum value, but remains larger than after deformation.

DISCUSSION. Thus, the combined deformation of the samples, which consists of the previous cold dynamic deformation and the subsequent static deformation, is likely to contribute to the formation of sub-regions with an increased angle of divergence. It is obvious that an additional static deformation increases the number of structural imperfections in the form of dislocation crossings.

It is known for example [5] that the movement of dislocations is prevented by the boundaries of sub-grains, particles of another phase, concentration heterogeneity, structural imperfections (dislocations), fluctuations in the lattice, associated with uneven distribution of energy and impurities.

Also, dislocations that were introduced during deformation are blocked by impurity atoms, so at the next load, these dislocations do not participate in sliding, they inhibit newly formed dislocations or parts of dislocations that are unlocked after deformation loading. Atoms of penetration in metals cause more tetragonal and lead to a sharp increase in strength.
Thus, the stabilization of the substructure of technically pure iron and steels is to reduce the mobility of the boundaries of sub-grains by creating triple nodes (crossings) of dislocations and inhibition of dislocations by admixing atoms, predominantly those dissolved by penetration (in technically pure iron — 0.009 % C).

**CONCLUSIONS.**

1. It was established that obtaining a thermally stable polygonal substructure for 60 minutes at a temperature of 500 °C provides a combination of cold dynamic deformation of 30 % and static deformation of 30 % followed by pre-crystallization thermal treatment for technically pure iron, steels 20, 45, U8, 40X and 12X13. The smallest OCP sizes (about 100 nm) are observed in pre-crystallization heat treatment, which provides maximum hardness, with an increase of 10 % on the substructure of OCP stabilization.

2. Combined deformation and subsequent pre-crystallization thermal treatment providing maximum hardness values provide an increase in the relative number of nanoscale sub-grains by at least 18 % (steel 20), a maximum of 89 % (steel 12X13) compared to the deformed state.

3. It was established that the maximum dimension of the angle of diorientation of sub-grains 2,16° was recorded after combined deformation with thermal treatment, which provides maximum hardness for technically pure iron. At 60 minutes, the orientation angle slightly decreases relative to the maximum value, but remains larger relative to the deformed state.

### Table 7. Relative amount of nanosized sub-grains, %, in deformed technically pure iron and steels before and after pre-crystallization heat treatment

<table>
<thead>
<tr>
<th>Processing</th>
<th>Fe</th>
<th>Steel 20</th>
<th>Steel 45</th>
<th>Y8</th>
<th>40X</th>
<th>12X13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>54</td>
<td>17</td>
<td>15</td>
<td>28</td>
<td>37</td>
<td>18</td>
</tr>
<tr>
<td>Combined deformation and heat treatment, which</td>
<td>75</td>
<td>20</td>
<td>22</td>
<td>42</td>
<td>65</td>
<td>34</td>
</tr>
<tr>
<td>provides maximum hardness</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Combined deformation and heat treatment for</td>
<td>58</td>
<td>16</td>
<td>18</td>
<td>19</td>
<td>16</td>
<td>12</td>
</tr>
<tr>
<td>60 minutes</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Table 8. Middle angle of divergence of sub-grains, degrees, in deformed technically pure iron and steels before and after pre-crystallization heat treatment

<table>
<thead>
<tr>
<th>Processing</th>
<th>Fe</th>
<th>Steel 20</th>
<th>Steel 45</th>
<th>Y8</th>
<th>40X</th>
<th>12X13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined deformation</td>
<td>1,4</td>
<td>0,04</td>
<td>0,04</td>
<td>0,14</td>
<td>0,05</td>
<td>0,08</td>
</tr>
<tr>
<td>Combined deformation and heat treatment, which</td>
<td>2,16</td>
<td>0,1</td>
<td>0,88</td>
<td>0,21</td>
<td>0,08</td>
<td>0,18</td>
</tr>
<tr>
<td>provides maximum hardness</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Combined deformation and heat treatment for</td>
<td>1,8</td>
<td>0,08</td>
<td>0,24</td>
<td>0,19</td>
<td>0,06</td>
<td>0,14</td>
</tr>
<tr>
<td>60 minutes</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Список літератури**


© О. М. Дубовий, Т. О. Макруха, О. В. Чечель
Статтю рекомендує до друку
d-р техн. наук, проф. В. Ф. Квасницький