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Thermal stability of a submicrocrystalline structure formed by high-pressure torsion in Ni and Ni–2 % Cr alloy

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Abstract: The main problem of submicrocrystalline (SMC) materials formed as a result of severe plastic deformation is their thermal stability. The large stored energy and the formation of strongly disordered microcrystallites in the structure lead to a decrease in the recrystallization onset temperature and, therefore, possibly decrease the structure stability. In the work, severe plastic deformation by high-pressure torsion and annealing of pure nickel and an alloy containing 2 at. % chromium were carried out. The structure of both deformed and annealed material was studied by scanning and transmission electron microscopy. The dependence of hardness on the square root of true strain and structure evolution were analyzed to identify the boundaries of the stages of structural states. The energy stored during deformation was estimated using differential scanning calorimetry by the amount of absorbed heat energy. The author studied the behaviour of materials during annealing depending on the stored strain energy at the SMC structure stage. Three stages of structural stats were identified in pure nickel: cellular, mixed, and SMC structure, while in the alloy containing 2 at. % chromium, a cellular structure stage was not detected. A decrease in the stored strain energy was found at the stage of the SMC structure for both materials. Alloying nickel with 2 at. % chromium increases its thermal stability, which increases the temperature when the grain growth becomes intensive by 150 °C. The amount of stored strain energy affects grain growth in the alloy containing 2 at. % chromium, whereas in pure nickel no effect was detected. In the Ni–Cr alloy, greater stored energy corresponds to larger recrystallized grain size.

Keywords: nickel; Ni–Cr alloy; high-pressure torsion; submicrocrystalline structure; stored strain energy.

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INTRODUCTION

Industrial development requires the creation of new materials with unique physical and mechanical properties. One of the approaches to achieve these properties is the formation of submicrocrystalline (SMC) and nanocrystalline (NC), structural states in these materials, for which severe plastic deformation (SPD) can be used [1; 2], in particular, high-pressure torsion (HPT) [3; 4]. The structure formation directly depends on the stacking fault energy (SFE) of the studied materials. Thus, in materials with a medium SFE during deformation at room temperature, the structure evolution appears to be three successive stages: cellular, mixed, and SMC structure [1; 5; 6]. The cellular-type structure is formed due to the movement of dislocations. At the second stage, along with the dislocation mechanism, rotational modes of deformation appear leading to the formation of individual microcrystallites [7]. Such a structure contains both high- and low-angle boundaries; it is usually called a mixed structure [8]. Finally, at the last stage, the structure consists of microcrystallites only – this is the SMC structure stage. In materials with high SFE during HPT, the cellular structure stage is not registered; after small angles of anvil rotation, a mixed structure appears [9].

The presence of an SMC structure leads to a significant (by about 150 °C) decrease in the recrystallization temperature, which may be associated with a decrease in the thermal stability of the structure [10]. On the other hand, in [11–13] it is shown that grinding the structural elements below a certain critical size, leads to an increase in thermal stability. In the works [14; 15], it was found that the formation of a honeycomb-type structure as a result of SMC structure recrystallization gives the material high thermal stability. The SMC structure can be obtained in the material surface layer by frictional processing [16]. This treatment

of martensitic and austenitic steels delays the softening (by 200–300 °C) of the material surface layer [17].

Another characteristic of thermal stability is the grain growth rate during recrystallization. It is possible to reduce the grain growth rate by alloying to form a solid solution. It is important to limit the content of the alloying element to prevent such a decrease in the SFE, that would lead to a change in the strain mechanism.

The stored strain energy also affects recrystallization [18]. In a number of materials, SPD can lead to the development of relaxation processes, such as dynamic recovery and dynamic recrystallization [3; 5; 19]. As a result of these processes, with an increase in true strain, a decrease in the stored strain energy can be observed, and, consequently, a decrease in the recrystallization driving force.

The purpose of this work is to study the influence of alloying Ni–2 at. % Cr on the SMC structure formation, the amount of stored energy during HPT deformation, as well as on the thermal stability of the resulting structure upon heating.

METHODS

To carry out the study, single-crystalline nickel (99.98 wt. % Ni) and a polycrystalline single-phase Ni–Cr alloy, with a chromium content of 2 at. % (Ni–2Cr) were selected. The samples had a diameter of 5 mm and a thickness of 0.3 mm. They were deformed by HPT in Bridgman anvils at room temperature under a pressure of 8 GPa by upsetting and with anvil rotation: for Ni – from 15° to 7 revolutions, and for Ni–2Cr – from 15° to 10 revolutions. The true strain was calculated using the formula

$$e = \ln\left(1+\left(\frac{\varphi r}{h_i}\right)^2\right)^{\frac{1}{2}} + \ln\left(\frac{h_0}{h_i}\right),$$

where φ is the angle of anvil rotation, rad.;
r_i is the radius from the center of the sample, mm;
h_0 is the sample thickness before deformation, mm;
h_i is the thickness of the sample after deformation at the i-radius, mm.

The hardness of the studied materials was measured after deformation, using a METOLAB 502 device under a load of 0.5 N along two mutually perpendicular diameters of the sample with a step of 0.25 mm. Hardness values were averaged over true strain intervals Δe=0.4. The boundaries of the deformation stages were determined by the inflection points in the hardness dependence on the square root of true strain $H=f(e^{1/2})$ in accordance with the technique described in [20]. To assess the mechanical properties of nano- and submicrocrystalline materials, hardness values were used as flow stress, as substantiated in [21]. Before measuring hardness, the samples were ground and electropolished.

Calorimetric studies were carried out using a STA 449 F3 thermal analyser, with a heating rate of 20 °C/min in the temperature range of 25–1000 °C. Two successive heatings were carried out in the same temperature range with the sample being cooled to room temperature after each heating. During the first heating, heat release peaks were recorded on the curves. The peak temperatures were determined by the secant method, and the amount of released thermal energy was determined as the area under the peak. This energy was then associated with the stored strain energy [22]. Reheating was carried out to ensure that the heat release process was irreversible. The error, when determining the amount of energy released during calorimetric measurements did not exceed 2.5 %.

The samples were annealed in a vacuum furnace at the following temperatures: for Ni – 200–350 °C with a step of 50 °C for 1 h; for Ni–2Cr – 200–400 °C with a step of 100 °C for 1 h. Samples with the maximum stored energy (for Ni – e=7.0 and for Ni–2Cr – e=9.3, which corresponds to the anvil rotation by 2 and 10 revolutions), and SMC structure samples, where the maximum stored energy has not been reached (for pure nickel – with the anvil rotation by 7 revolutions (e=8.3), and for Ni–2Cr – by 5 revolutions (e=8.5)), were selected for annealing.

The resulting structures were assessed using QUANTA 200 and TESCAN MIRA scanning electron microscopes, and a JEM200CX transmission electron microscope. Using scanning electron microscopy (SEM), the structure was examined at a distance of 1.0 mm from the centre of the samples. Based on the SEM results, the spectra of grain-boundary angles, and grain sizes were determined. Using transmission electron microscopy, the structure was examined at the distances of 1.5 mm from the centre of the samples. The sizes of structural elements (dislocation cells, microcrystallites, and recrystallized grains) were determined from bright-field images, as well as from dark-field images in the {111} type reflection, based on the results of more than 400 measurements, which ensured an error of less than 10 %.

The results were processed using the STATISTICA software.

RESULTS

Fig. 1 shows a diagram of the hardness dependence on the true strain of the studied materials. It can be seen that alloying with 2 % of Cr only slightly influences the strain, hardening of Ni in the region of relatively small strains (up to e=6). At e>6, alloying, ensures an increase in the hardness growth coefficient; in Ni, hardness stabilisation is registered after e=5.

To identify the stages of deformation in the studied materials, the dependences shown in Fig. 2 were obtained. For pure Ni, three stages were identified, their change occurs at e=0.8 and e=5. For the alloy containing 2 % of Cr, only two stages were obtained; the transition from one stage to another in Ni–2Cr occurred at e=8.5.

Fig. 3 shows the structures of pure Ni and the Ni–2Cr alloy deformed in this work. It can be seen that the evolution of the pure nickel structure goes through three stages. First, a cellular-type structure is formed up to e=0.8 (Fig. 3 a). In the interval 0.8<e<5, individual microcrystallites with high-angle boundaries are formed in the structure, which is a sign of a mixed-type structure (Fig. 3 b). Moreover, after e=5, the structure contains only microcrystallites, which is a sign that the SMC structure has been achieved (Fig. 3 c). Unlike pure nickel, in Ni–2Cr, the cellular structure stage was not registered, therefore, only one boundary is identified in this alloy, at which the mixed structure is replaced by the SMC structure at e=8.5 (Fig. 3 d, 3 e).

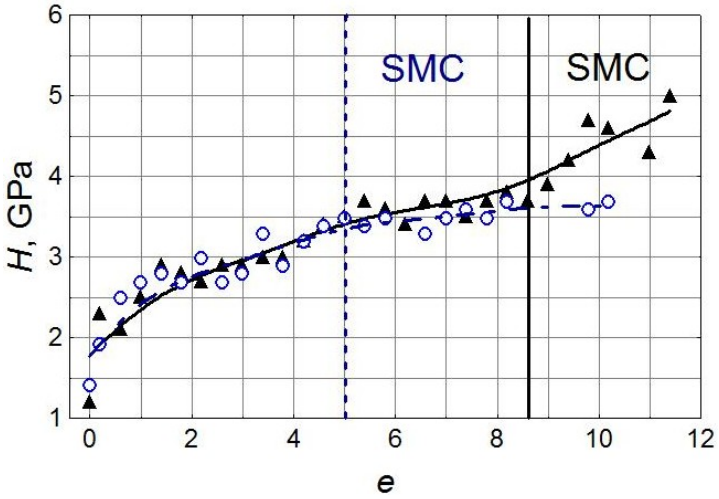


Fig. 1. *Microhardness dependence on true strain: ○ – pure Ni; ▲ – Ni–2Cr alloy*
Рис. 1. *Зависимость твердости от истинной деформации: ○ – чистый Ni; ▲ – сплав Ni–2Cr*

Fig. 4 shows the dependence of the average size of structural elements on true strain. It has been found that alloying with chromium in an amount of 2 % does not lead to a decrease in the size of microcrystallites in the single-phase Ni–2Cr alloy compared to pure nickel: after e=9, the average size of microcrystallites in both cases is (0.14±0.01) μm. It can be seen that the output of the dependencies shown in Fig. 4 to saturation in grinding is typical for both studied materials.

Fig. 5 shows that the stored energy (E) in the studied materials continuously increases with increasing deformation up to e=7 in pure Ni and e=9 in Ni–2Cr, and with continued deformation at the SMC structure stage it decreases in both materials. Thus, the maximum stored energy was recorded after deformation e=7.0 for Ni and e=9.3 for Ni–2Cr, which corresponds to the anvil rotations for 2 and 10 revolutions.

Fig. 6 gives images of the pure nickel structure after deformation and annealing. Annealing pure nickel at 150 °C leads to the recrystallization onset (Fig. 6 a). The average size of structural elements is 0.2 μm. Increasing the annealing temperature to 200 °C leads to the recrystallization completion (Fig. 6 b). The average size of recrystallized grains is 6 μm, while the maximum size of recrystallized grains is about 40 μm. After annealing at 300 °C, a decrease in the average size of recrystallized grains to 5.5 μm is observed, apparently due to the appearance of nuclei through the thermal activation mechanism (Fig. 6 c). At the annealing temperature of 350 °C, the grain growth rate becomes greater than the rate of nucleation of new recrystallization centres (Fig. 6 d), and the average grain size increases again.

Fig. 7 shows that in nickel, the average grain size obtained through annealing the material with the maximum (2 revolutions) and lower (7 revolutions) stored strain energy is almost the same. The temperature when rapid grain growth begins also does not depend on the strain amount (stored energy). Thus, no effect of the difference in the stored strain energy on recrystallization in pure nickel with an SMC structure was detected.

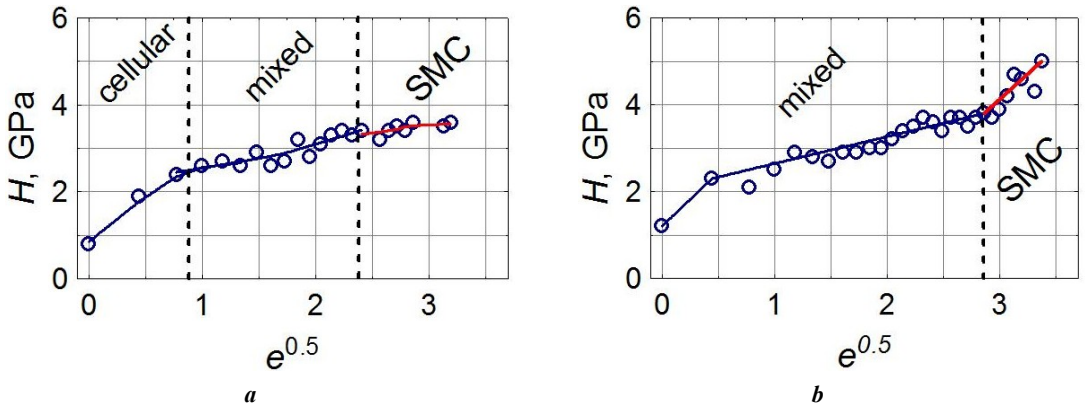


Fig. 2. *Diagram of microhardness dependence on the square root of true strain:* ***a*** *– pure Ni;* ***b*** *– Ni–2Cr alloy*
Рис. 2. *График зависимости твердости от квадратного корня из истинной деформации:* ***a*** *– чистый Ni;* ***b*** *– сплав Ni–2Cr*

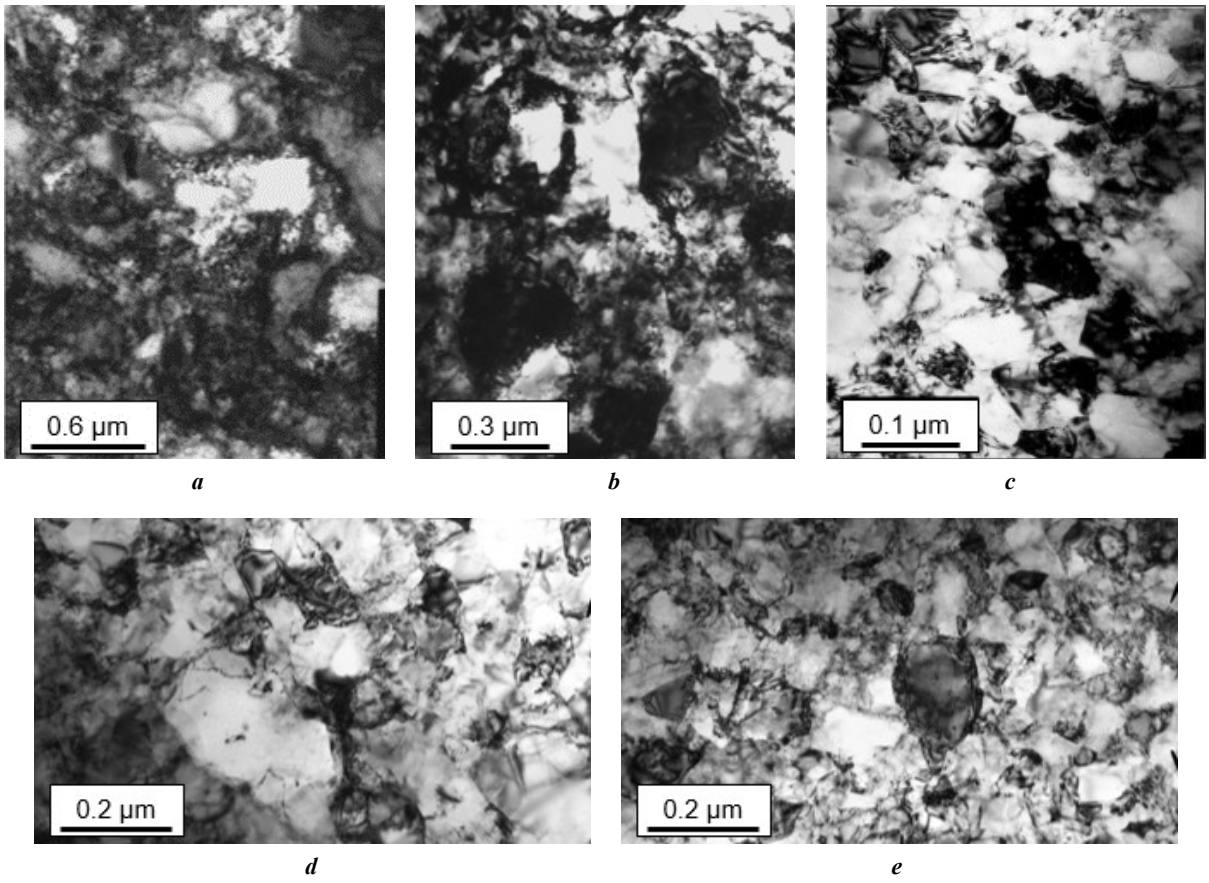


Fig. 3. *Fine structure of pure nickel and Ni–2Cr alloy deformed by high-pressure torsion:* ***a*** *– Ni, e=0.3;* ***b*** *– Ni, e=4.2;* ***c*** *– Ni, e=6.9;* ***d*** *– Ni–2Cr, e=4.0;* ***e*** *– Ni–2Cr, e=8.6*
Рис. 3. *Тонкая структура чистого никеля и сплава Ni–2Cr, деформированных СПД:* ***a*** *– Ni, e=0,3;* ***b*** *– Ni, e=4,2;* ***c*** *– Ni, e=6,9;* ***d*** *– Ni–2Cr, e=4,0;* ***e*** *– Ni–2Cr, e=8,6*

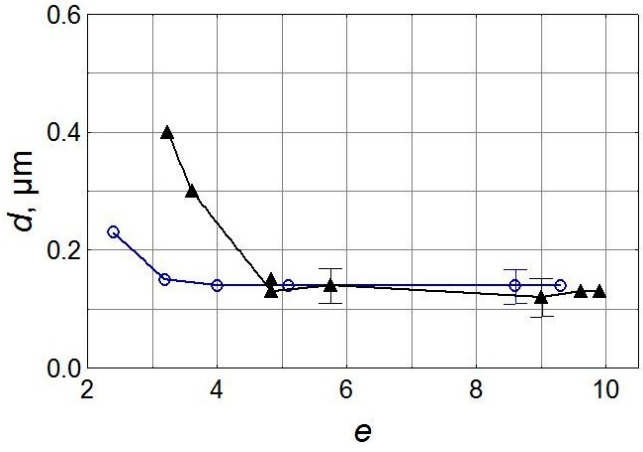


Fig. 4. *Dependence of average size of structural elements on true strain:*
○ – pure Ni; ▲ – Ni–2Cr alloy
Рис. 4. *Зависимость среднего размера элементов структуры от истинной деформации:*
○ – чистый Ni; ▲ – сплав Ni–2Cr

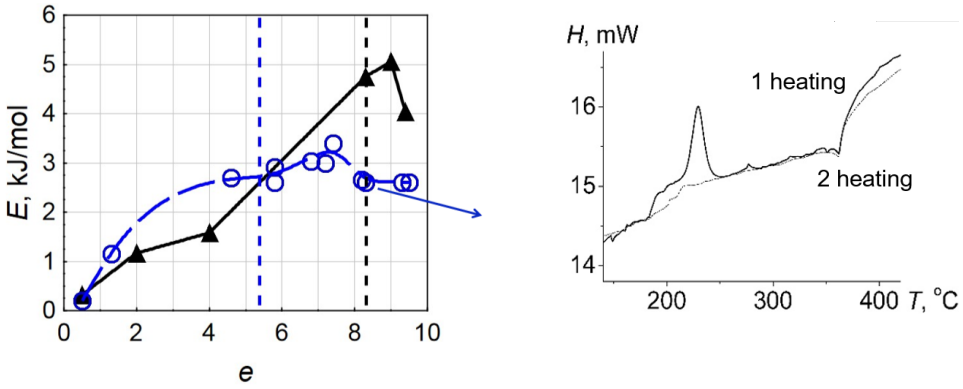


Fig. 5. *Dependence of stored strain energy on true strain: ○ – pure Ni; ▲ – Ni–2Cr alloy; the inset shows an example of a DSC (differential scanning calorimetry) curve for Ni deformed by 5 anvil revolutions*
Рис. 5. *Зависимость запасенной энергии деформации от истинной деформации: ○ – чистый Ni; ▲ – сплав Ni–2Cr; на вставке пример ДСК (дифференциальной сканирующей калориметрии) кривой для Ni, деформированного на 5 оборотов наковальни*

There are individual recrystallized grains with a size of about 0.7 μm in the Ni–2Cr alloy after annealing at 200 °C (Fig. 8 a, 8 b). The average size of the structural elements did not change much, relative to the size of the microcrystallites in the deformed state. It amounts (0.16±0.03) μm. This shows that the number of recrystallized grains is small. Thus, recrystallization in this material just begins at a temperature of 200 °C.

In turn, annealing at 300 °C shows that in the Ni–2Cr alloy, recrystallization occurs more completely after deformation with e=9.3 (10 anvil revolutions, Fig. 8 c). In this case, in the sample that was deformed by HPT for 5 revolutions, a large fraction of the non-recrystallized matrix is retained (Fig. 8 d). The structure contains large deformed structure areas, as well as individual grains larger than 1 μm in size, which do not contain dislocations. SEM images demonstrate that annealing led to the bimodal structure formation (Fig. 9). The average size of the structural elements for both treatments is close: 1.2 μm after deformation with e=8.5 and 0.8 μm with e=9.3, while the maximum size differs by a factor of 2 and is 5 and 9 μm, respectively.

Increasing the annealing temperature to 400 °C increases the dimensional heterogeneity of the structure of the alloy with 2 % of Cr deformed by both 5 and 10 anvil revolutions (Fig. 10); along with large grains, small crystallites are observed. It can be seen that after deformation by 10 revolutions, and annealing at 400 °C, individual coarse grains larger than 50 μm in size appeared (Fig. 10 a), the average grain size is approximately 8 μm. After

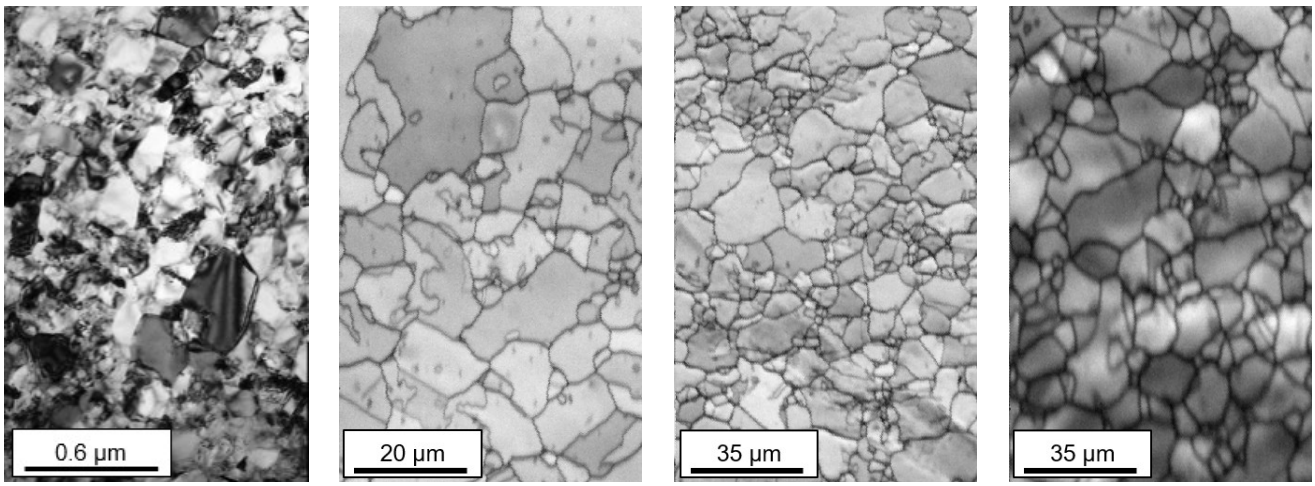


Fig. 6. *Electron microscope images of the structure of pure nickel deformed with e=7 (2 anvil revolutions) and following annealing at:*
a *– 150 °C;* ***b*** *– 200 °C;* ***c*** *– 300 °C;* ***d*** *– 350 °C*
Рис. 6. *Электронно-микроскопические изображения структуры чистого никеля, деформированного с e=7 (2 оборота наковальни), и последующего отжига при:*
a *– 150 °C;* ***b*** *– 200 °C;* ***c*** *– 300 °C;* ***d*** *– 350 °C*

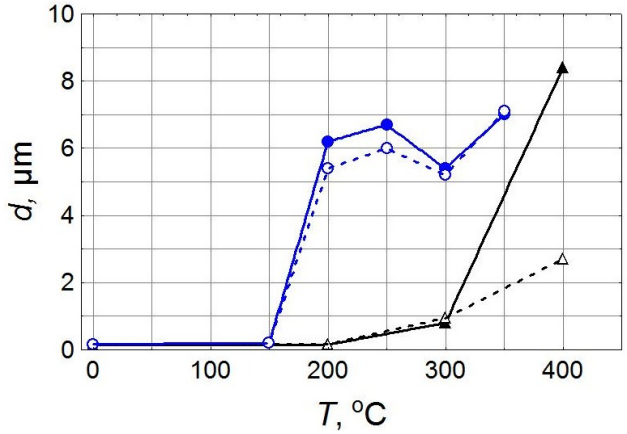
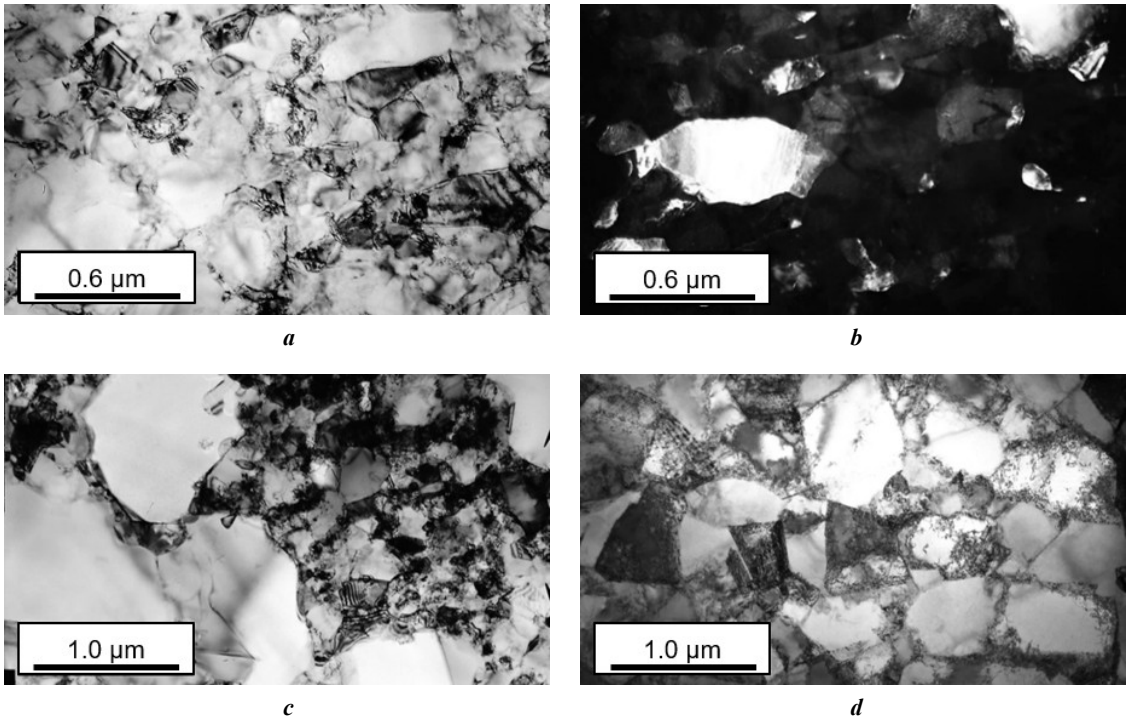


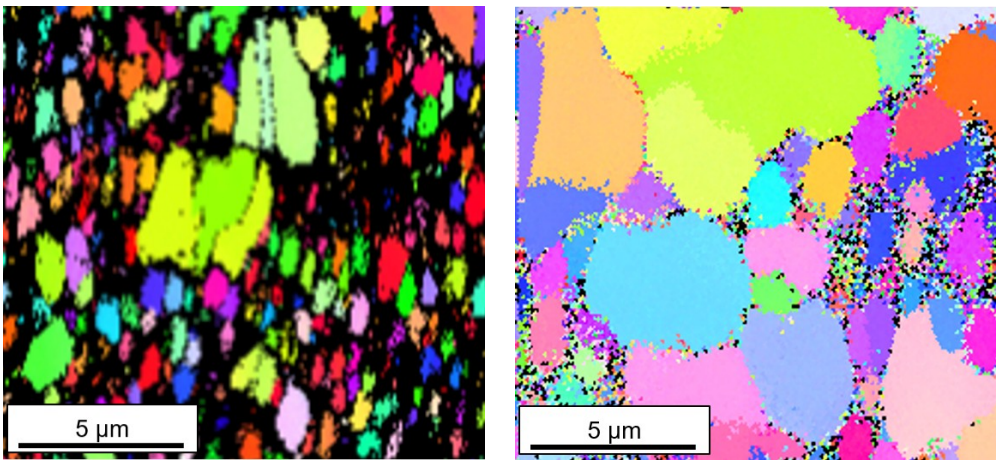
Fig. 7. *Dependence of average size of structural elements on annealing temperature:*
● – pure Ni; ▲ – Ni–2Cr alloy;
filled markers correspond to the deformation when the stored energy is maximum according to DSC data
Рис. 7. *Зависимость среднего размера элементов структуры от температуры отжига:*
● – чистый Ni; ▲ – сплав Ni–2Cr;
закрашенные маркеры соответствуют деформации,
при которой запасенная энергия максимальна, согласно данным ДСК



a ***b***

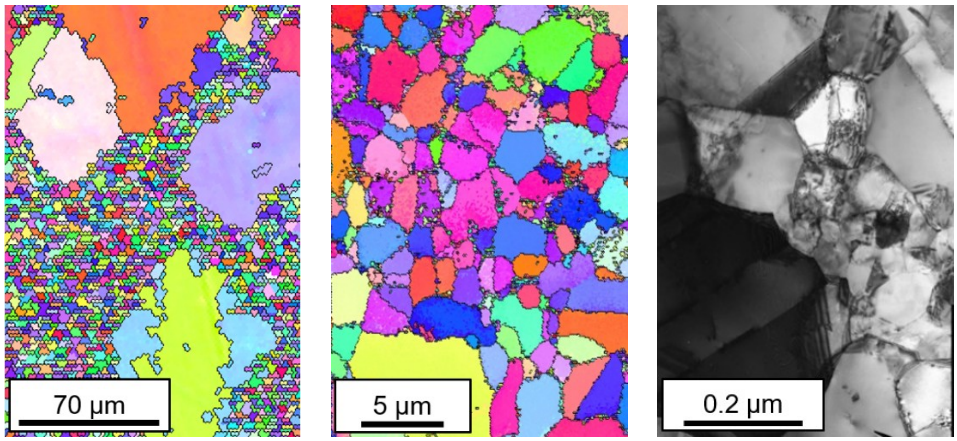
c ***d***

Fig. 8. *Microstructure of the Ni–2Cr alloy after deformation*
*and subsequent annealing at 200 (**a**, **b**) and 300 °C (**c**, **d**):*
a *– bright-field image, e=8.5;* ***b*** *– dark-field image, e=8.5;* ***c*** *– bright-field image, e=9.3;* ***d*** *– bright-field image, e=8.5*
Рис. 8. *Микроструктура сплава Ni–2Cr после деформации*
*и последующего отжига при 200 (**a**, **b**) и 300 °C (**c**, **d**):*
a *– светлопольное изображение, e=8,5;* ***b*** *– темнопольное изображение, e=8,5;*
c *– светлопольное изображение, e=9,3;* ***d*** *– светлопольное изображение, e=8,5*



a *b*

Fig. 9. *Orientation map in colours of the inverse pole figure of the Ni–2Cr alloy after deformation and subsequent annealing at 300 °C:* ***a*** *– e=8.5;* ***b*** *– e=9.3*
Рис. 9. *Ориентационная карта в цветах обратной полюсной фигуры сплава Ni–2Cr после деформации и последующего отжига при 300 °С:* ***a*** *– e=8,5;* ***b*** *– e=9,3*



a *b* *c*

Fig. 10. *Orientation map in colours of the inverse pole figure and fine structure of the Ni–2Cr alloy after deformation and subsequent annealing at 400 C:* ***a*** *– e=9.3;* ***b, c*** *– e=8.5;* ***a, b*** *– SEM;* ***c*** *– TEM*
Рис. 10. *Ориентационная карта в цветах обратной полюсной фигуры и тонкая структура сплава Ni–2Cr после деформации и последующего отжига при 400 С:* ***a*** *– e=9,3;* ***b, c*** *– e=8,5;* ***a, b*** *– СЭМ;* ***c*** *– ПЭМ*

deformation by 5 revolutions and annealing at 400 °C, relatively large grains up to 8 μm in size and small areas of non-recrystallized structure are also observed (Fig. 10 b, 10 c), but the average grain size is several times smaller – 2.5 μm. Thus, in this alloy, primary recrystallization does not complete even at 400 °C. For the Ni–2Cr alloy, the dependences of the average size of structural elements on the annealing temperature obtained by SEM and TEM methods are shown in Fig. 7.

DISCUSSION

Pure nickel deformation at the SMC structure stage does not lead to a change in hardness (Fig. 1). At the same time, the dimensions of the nickel structural elements also remain constant (Fig. 4), and calorimetric studies show a decrease in the stored strain energy (Fig. 5). Based on these results, one can conclude that in nickel deformed by HPD at the SMC structure stage, the dominant structure-forming process is dynamic recovery. This is consistent with data known from the literature [23].

The results of the work confirmed that alloying with a small amount of chromium did not cause a change in the strain mechanism, which is observed, for example, in the Ni–20Cr alloy [24]. In this work, the author managed to identify the influence of solid solution strengthening on the formation of the structure of the studied materials during deformation and subsequent annealing. In the Ni–2Cr alloy, the SMC structure is formed at a significantly higher true strain (e=8.5) than in pure Ni (e=5). In this case, deformation at the SMC structure stage does not lead to stabilisation of the alloy hardness value. However, the average size of the SMC structure elements, just as in nickel, does not change (Fig. 1 and 2). Despite the stabilisation of sizes

in Ni–2Cr, according to calorimetric studies, the stored energy changes: it increases to e=9.3 (10 anvil revolutions), and then decreases. Consequently, the structure continues to change. A decrease in the stored strain energy, at the SMC structure stage indicates the occurrence of dynamic recovery in the alloy (Fig. 5). However, the increase in hardness shows that in this case, dynamic recovery is not the dominant process.

Annealing of SMC nickel showed that recrystallization begins at 150 °C (Fig. 6 a) and proceeds as the growth of individual centres. This leads to strong grain heterogeneity: a small number of large grains are located in a fine-grained matrix. Recrystallization is completed at 200 °C (Fig. 6 b). After annealing at 300 °C, a decrease in the average size of recrystallized grains is observed (Fig. 6 c). A similar effect was recorded in [15] as a result of annealing the iron SMC structure. In the latter case, the decrease in size was associated with the appearance of thermally activated recrystallization nuclei. A further increase in the annealing temperature of nickel shows a tendency towards grain structure coarsening (Fig. 6 d).

Nickel alloying with 2 at. % of chromium increases the temperature of the recrystallization onset from 150 to 200 °C, and the temperature of the beginning of intensive grain growth – from 150 to 300 °C (Fig. 7 and 8). Therefore, one can conclude that solid solution strengthening increased the thermal stability of the SMC structure. Just like in Ni, in the Ni–2Cr alloy, recrystallization occurs according to the mechanism of accelerated growth of individual centres, which does not allow obtaining a homogeneous submicrogranular recrystallized structure, as, for example, in iron [14; 15]. In this work, it was not possible to determine the temperature of the end of recrystallization in the Ni–2Cr alloy.

In pure nickel, a change in the stored strain energy at the SMC structure stage does not affect recrystallization, while in Ni–2Cr the stored strain energy affects the size of the recrystallized grain: greater stored energy corresponds to a larger recrystallized grain size (Fig. 7).

CONCLUSIONS

1. During high-pressure torsion deformation, in the Ni–2Cr alloy, in contrast to pure nickel, a continuous increase in hardness is observed throughout the entire studied deformation range. Alloying with chromium significantly inhibits the transition to the SMC structure stage: in the Ni–2Cr alloy, the transition to the submicrocrystalline structure stage occurs at a true strain e=8.5±0.3 – higher than for pure nickel (e=5.3).

2. Nickel alloying with chromium in an amount of 2 at. % does not lead to an increase in the submicrocrystalline structure dispersion; in both cases, after deformation with e=9, the microcrystallite size is (0.14±0.01) μm.

3. Alloying with chromium affects the recrystallization temperature of nickel. The temperature, when the recrystallization of nickel with a submicrocrystalline structure begins, is 150 °C, and that of the Ni–2Cr alloy is 200 °C. The temperature of the intensive grain growth onset increases from 150 °C in pure nickel, to 300 °C in the Ni–2Cr alloy.

4. In the Ni–2Cr alloy, preliminary high-pressure torsion deformation and the energy stored during this, influence the recrystallized grain size. As a result of annealing of the Ni–2Cr alloy, in which the maximum energy was accumulated during deformation, the largest recrystallized grains, and high dimensional heterogeneity of the structure are observed, whereas in pure nickel, the dependence was not detected.

5. In both studied materials, submicrocrystalline structure recrystallization occurs through the accelerated growth of individual centres. This makes it impossible to obtain a submicrogranular recrystallized structure uniform in size.

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Термическая стабильность субмикрокристаллической структуры, сформированной методом «сдвиг под давлением» в Ni и сплаве Ni–2 % Cr

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Аннотация: Основной проблемой субмикрокристаллических (СМК) материалов, сформированных в результате большой пластической деформации, является их термическая стабильность. Большая запасенная энергия и формирование в структуре сильно разориентированных микрокристаллитов ведет к уменьшению температуры начала рекристаллизации и, как следствие, возможно, к снижению стабильности структуры. В работе проведена большая пластическая деформация методом «сдвиг под давлением», а также отжиг чистого никеля и его сплава, содержащего 2 ат. % хрома. Исследование структуры как деформированного, так и отожженного материала осуществляли методами сканирующей и просвечивающей электронной микроскопии. Анализ зависимости твердости от квадратного корня из истинной деформации совместно с анализом структурных изменений позволил выделить границы стадий структурных состояний. Запасенную при деформации энергию оценивали с помощью дифференциально-сканирующей калориметрии по количеству поглощенной тепловой энергии. Исследовано поведение материалов при отжиге в зависимости от запасенной энергии деформации на стадии СМК-структуры. В чистом никеле выделены три стадии структурных состояний: ячеистой, смешанной и СМК-структуры, тогда как в сплаве, содержащем 2 ат. % Cr, стадия ячеистой структуры не зафиксирована. Обнаружено снижение запасенной энергии деформации на стадии СМК-структуры для обоих материалов. Легирование никеля 2 ат. % хрома повышает термическую стабильность, что проявляется в повышении температуры начала интенсивного роста зерна на 150 °С. Величина запасенной энергии деформации оказывает влияние на рост зерна в сплаве с содержанием хрома 2 ат. %, тогда как в чистом никеле влияние не зафиксировано. В сплаве Ni–Cr большая запасенная энергия соответствует большему размеру рекристаллизованного зерна.

Ключевые слова: никель; сплав Ni–Cr; сдвиг под давлением; субмикрокристаллическая структура; запасенная энергия деформации.

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