



The influence of radial shear rolling on the structure and properties of 58Ni-Cr-Mo-B-Al-Cu superalloy

A. A. Bikmukhametova[†], E. V. Galieva, I. Sh. Valeev, E. Yu. Klassman,
I. I. Musabirov, V. A. Valitov

[†]Bikmukhametova_aa@mail.ru

Institute for Metals Superplasticity Problems, RAS, Ufa, 450001, Russia

The work is devoted to a study of the microstructure and microhardness of the heat-resistant nickel-based superalloy 58Ni-Cr-Mo-B-Al-Cu after radial-shear rolling. The radial-shear rolling was carried out at a temperature of 925°C in three stages. It was found that the grain size of γ -phase after all rolling stages decreased in the radial direction from the center to the periphery from 62 ± 2 to 2.5 ± 0.2 μm . At the third rolling stage, the grains size was reduced twice in the billet center in a comparison to the initial state. At the same time, individual grains with a size of 100 μm in the billet surface are observed, and grains with a size of 1 ± 0.1 μm are present at the periphery. The microhardness changes in inverse proportion to the grain size, the values increase in the radius direction. The maximum microhardness value is achieved in the peripheral part of the sample and is equal to 5.4 ± 0.6 GPa. According to the EBSD analysis, there is no texture at the billet periphery, but at the middle of the radius there is a two-component axial texture of the $\langle 001 \rangle + \langle 111 \rangle$ type along the billet axis. The billet center is characterized by the presence of a more pronounced two-component axial texture of the $\langle 001 \rangle + \langle 111 \rangle$ type along the billet axis. The obtained results indicate the possibility of the gradient structure formation by radial-shear rolling in the heat-resistant nickel-based superalloy 58Ni-Cr-Mo-B-Al-Cu with the initial coarse-grained structure.

Keywords: heat-resistant nickel-based superalloy, ultrafine grained, coarse grained, microstructure, radial-shear rolling.

1. Introduction

It is known [1] that superalloys (heat-resistant nickel-based alloys) are used for manufacturing of various parts of gas turbine and rocket engines operating at high temperatures. These alloys are difficult to deform and are characterized by low technological plasticity. An increase in technological properties in such superalloys is possible by the formation of an ultrafine-grained (UFG) or even nanocrystalline structure [2–5]. Currently, there are many different severe plastic deformation methods to produce relatively homogeneous UFG structure throughout the entire volume in samples and billets of heat-resistant superalloys such as multiple isothermal forging, torsion straining on Bridgman anvils, etc. [2, 6–9]. However, in some cases it is desirable to form a gradient structure along the section of a billet. For instance, it is useful to form an UFG structure only in the surface layers of a billet keeping the initial coarse-grained one in the billet center. Such a microstructural state can be achieved by means of the radial-shear rolling method. The feature of radial-shear rolling is a complex diagram of the stress-strain state that leads to the appearance of maximum shear deformations in the peripheral surface zone of a rolled billet, which decreases in the central zone direction [10–13]. Such processing may result in a gradient structure formation across the rolled billet section. This fact is accompanied by the gradient properties obtaining varying from the surface to rolled billet center.

The aim of this work is the gradient microstructure formation in 58Ni-Cr-Mo-B-Al-Cu superalloy billet by radial-shear rolling.

2. Materials and methods

The 58Ni-Cr-Mo-B-Al-Cu superalloy with a coarse-grained microstructure was chosen as a material for the study. Cylindrical specimens of the alloy with a diameter of $d = 21$ mm and a length of $L = 65$ mm were deformed on the RSP-30 mill [14] at the temperature of $T = 925^\circ$ in three stages. The elongation ratio K_B was calculated by the formula: $K_B = (D/d)^2$, where D and d are the values of the diameter of billet before and after deformation, respectively.

The diameter of billet in the course of radial-shear rolling in three passes gradually decreased to 17.3 mm after the first rolling stage, to 13.6 mm after the second, and to 12.7 mm after the third rolling stage. Correspondingly, the elongation ratio K_B for each stage of rolling was equal to 1.47, 1.62, and 1.14, respectively, with the total value of K_B due to all stages equal to 2.73.

The microstructure study was carried by scanning electron microscope Mira 3 SBH (Tescan) using the BSE mode. The EBSD analysis was performed using a Nordlys Detector (Oxford Instruments) adjusted on this microscope at an accelerating voltage of 20 kV. Orientation maps were obtained from areas of 300×300 μm^2 with a scanning step of 0.5 μm in the central area, 300×300 μm^2 with a scanning step

of $0.5\ \mu\text{m}$ at a distance of $0.5R$ from the axis, and $60 \times 60\ \mu\text{m}^2$ with a scanning step of $0.1\ \mu\text{m}$ at $0.99R$.

Microhardness test involved the use of a diamond indenter to make a microindentation into the surface of the billet of 58Ni-Cr-Mo-B-Al-Cu superalloy. The hardness value is measured by Vickers method.

3. Results and discussion

The initial microstructure of the 58Ni-Cr-Mo-B-Al-Cu superalloy was a coarse-grained one with an average size of γ -phase grains of $62 \pm 2\ \mu\text{m}$ and sizes of semi-coherent particles of intragranular γ'' -phase of $40 \pm 2\ \text{nm}$ [4].

The radial-shear rolling leads to significant microstructure changes. After rolling of the sample from the diameter of 21 to 17.3 mm (rolling stage 1), the maximum grain size of $51.2 \pm 3.7\ \mu\text{m}$ is observed in the center of the sample (Fig. 1). One can also notice a gradual decrease in the size of recrystallized grains along the radius of the sample size in the transition from its center to the periphery, where the average grain size of the γ -phase is $15.2 \pm 2.1\ \mu\text{m}$ (Fig. 1c).

As one can see from Fig. 2, further radial-shear rolling of the superalloy from the diameter of 17.3 to 13.6 mm (rolling stage 2) leads to the formation of a gradient structure similar to that formed after the first stage of rolling, but with smaller grain sizes. The average size of γ -phase grains in the billet center was equal to $47.2 \pm 3.6\ \mu\text{m}$ and it monotonously decreased in the radial direction to the values of $10.4 \pm 2.3\ \mu\text{m}$ in the billet surface zone.

The most significant changes occurred after the third stage of radial-shear rolling, i.e. from the diameter of 13.6 mm to the final diameter of 12 mm (Fig. 3). After rolling to the final diameter with the total elongation ratio $K_B = 2.74$, the grain size of the γ -phase in the center of the billet was equal to $34.1 \pm 4.7\ \mu\text{m}$ and monotonously decreased in the radial direction down to $2.4 \pm 0.2\ \mu\text{m}$. So, an UFG structure was formed in the surface layers of the rolled billet.

The temperature of rolling, 925°C , is close to the higher border of the temperature range for superplasticity of the alloy 58Ni-Cr-Mo-B-Al-Cu with the UFG structure. Therefore, the surface layers of the sample continue to deform superplastically that is why work hardening does not occur and only previously formed recrystallized grains are observed. In the central part of the billet where insignificant deformation takes place, low-angle boundaries are observed. According to the EBSD analysis (Fig. 4), the fraction of high-angle boundaries increases from the center to the periphery by almost twice while the fraction of low-angle boundaries decreases significantly, from 52% to about 10%. Such a significant change from the coarse grained structure to the ultrafine grained one along the radius of the rolled billet from its center to the periphery is probably due to the features of the trajectory and velocity and the diagram of the stress-strain state during this severe plastic deformation process, which, as a consequence, leads to a difference in the plastic flow and the formation of structure in different zones of the billet [11,13].

A texture analysis of the selected areas shows the presence of a crystallographic texture in the center and in the middle

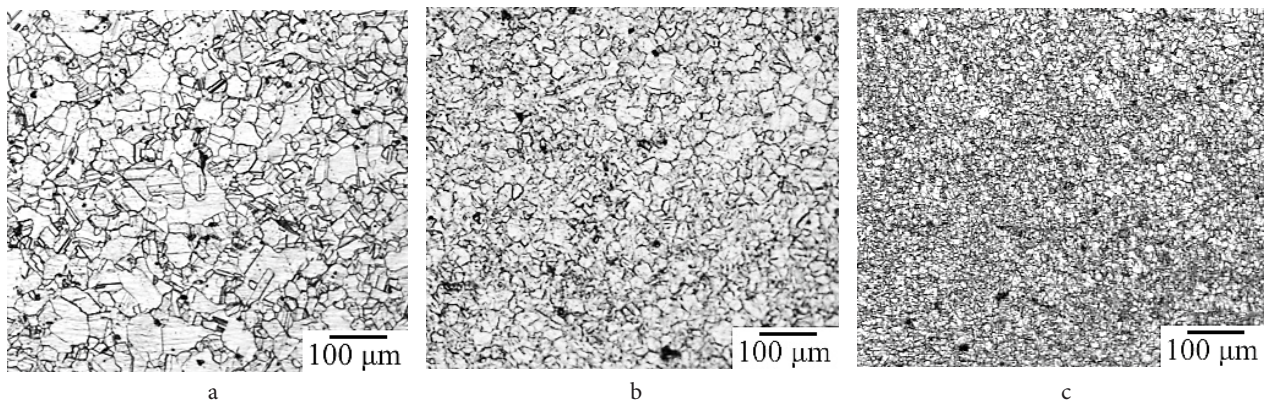


Fig. 1. Microstructure (optical microscopy) of 58Ni-Cr-Mo-B-Al-Cu superalloy ($K_B=1.47$): the center of sample (a), at a distance $0.5R$ (b), at a distance $0.99R$ (c).

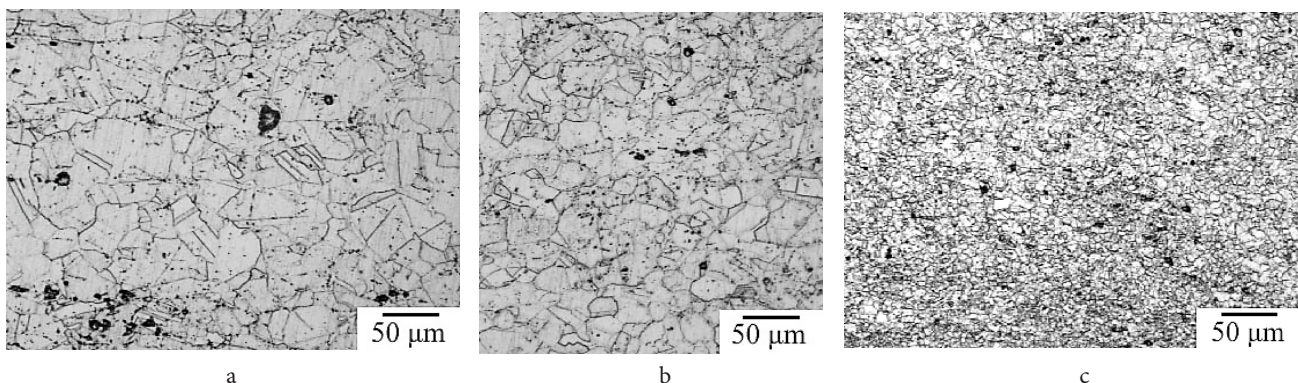


Fig. 2. Microstructure (optical microscopy) of 58Ni-Cr-Mo-B-Al-Cu superalloy ($K_B=1.62$): the center of sample (a), at a distance $0.5R$ (b), at a distance $0.99R$ (c).

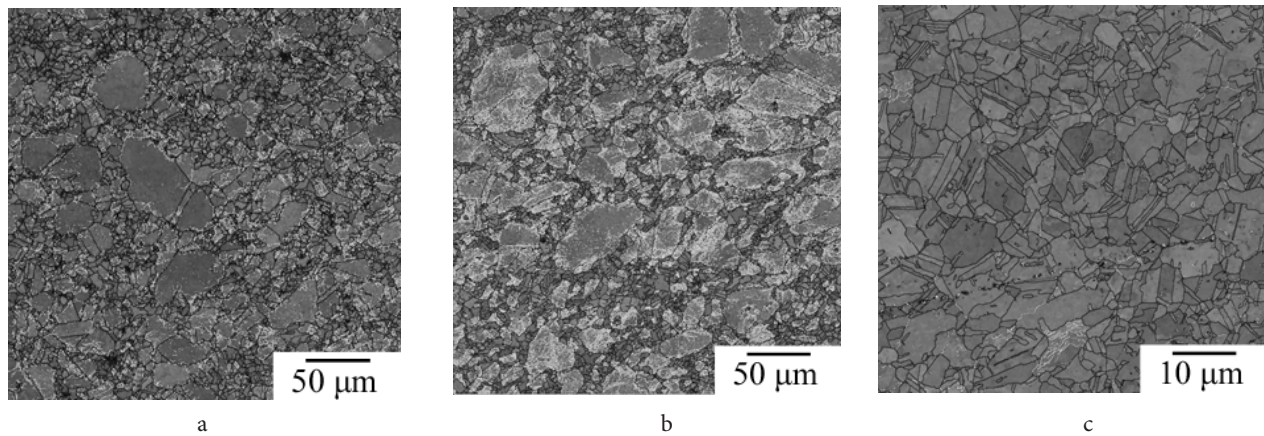


Fig. 3. Contrast maps of 58Ni-Cr-Mo-B-Al-Cu superalloy after radial-shear rolling ($K_r=1.14$): sample center (a), 0.5R (b), 0.99R (c).

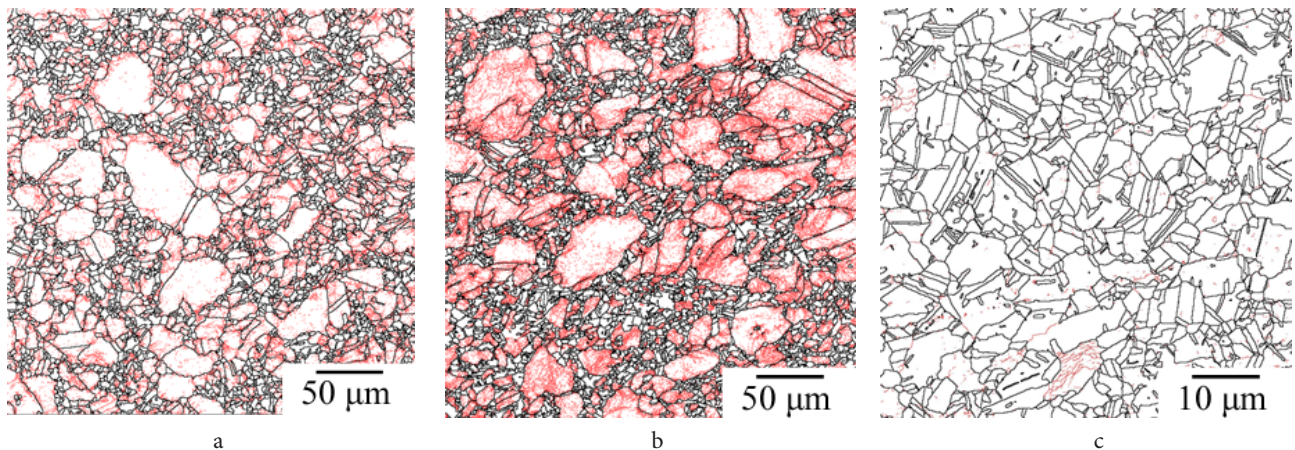


Fig. 4. (Color online) EBSD-maps for various part of the cross-section of the billet made from 58Ni-Cr-Mo-B-Al-Cu superalloy after radial-shear rolling $K_r=1.14$: sample center (a), 0.5R (b), 0.99R (c).

of the radius of the sample cut across the billet axis (Fig. 5). Direct pole figures (DPFs) are constructed for the planes of $\{100\}$, $\{110\}$ and $\{111\}$ families. For the area located at the sample periphery, the DPFs demonstrate the absence of texture. For all plane families, there is the lack of significant orientation localizations. The occurrence of several area with an orientation localization is due to the relatively small area of EBSD scan and presence of large grains. The DPFs for the area in the middle of the sample radius demonstrate a crystallographic axial texture. The DPF shows the orientation localization in the center of the $\{100\}$ plane family indicates that they lie in the plane of the section, that is, across the rolled billet axis. In this case, the presence of a ring at the periphery of the DPF for $\{110\}$ indicates that the texture is axial, in which the crystallites rotate around the $\langle 001 \rangle$ axes. Thus, this group of localizations indicates the presence of an axial texture of the $\langle 001 \rangle$ type along the billet axis.

The second component of the crystallographic texture is represented by the localization of the density of orientations on the DPF for $\{111\}$. In this case, the presence of an axial texture is shown, in which crystallites rotate around the $\langle 111 \rangle$ axes. Thus, a two-component axial texture of the $\langle 001 \rangle + \langle 111 \rangle$ type in the sample in the middle of the radius along the billet axis is observed. The maximum density of orientations is about 7. The study of the DPF for the area taken in the center of the sample also shows the presence of a two-component axial texture of the $\langle 001 \rangle + \langle 111 \rangle$ type along the billet axis. However, in

comparison with the previous case, it is more pronounced, since the maximum density of orientations is about 14.

It has been found that there is a noticeable decrease in the γ -phase grain size from the center to the peripheral surface billet part at each stage of the radial-shear rolling. Such a gradient decrease in the grain size in the cross section of the rolled billet leads to a gradient increase in the microhardness. Fig. 6 presents the dependences of microhardness in the radial direction of the rolled billet. The figure shows that the microhardness changes monotonically from the center to the periphery. The minimum microhardness value of 3.0–3.6 GPa in the sample center is observed, where the coarse-gradient structure is retained. The maximum values of microhardness of 5.2–5.5 GPa are observed at the periphery, where the structure is finer. Probably, such a change in the microhardness is associated with the Hall-Petch effect, according to which the characteristics of the yield point and, in this case, the microhardness of the material increase with decreasing grain size.

The 58Ni-Cr-Mo-B-Al-Cu superalloy is widely used for the manufacture of various parts of rocket engines from sheet, bar, and bulk materials, i.e. from various assortment of deformed semi-finished products. Therefore, the results obtained can significantly expand the range of parts that can be manufactured using superplasticity that will be observed in the rolled billet surface layer, in which the UFG structure is formed during radial-shear rolling.

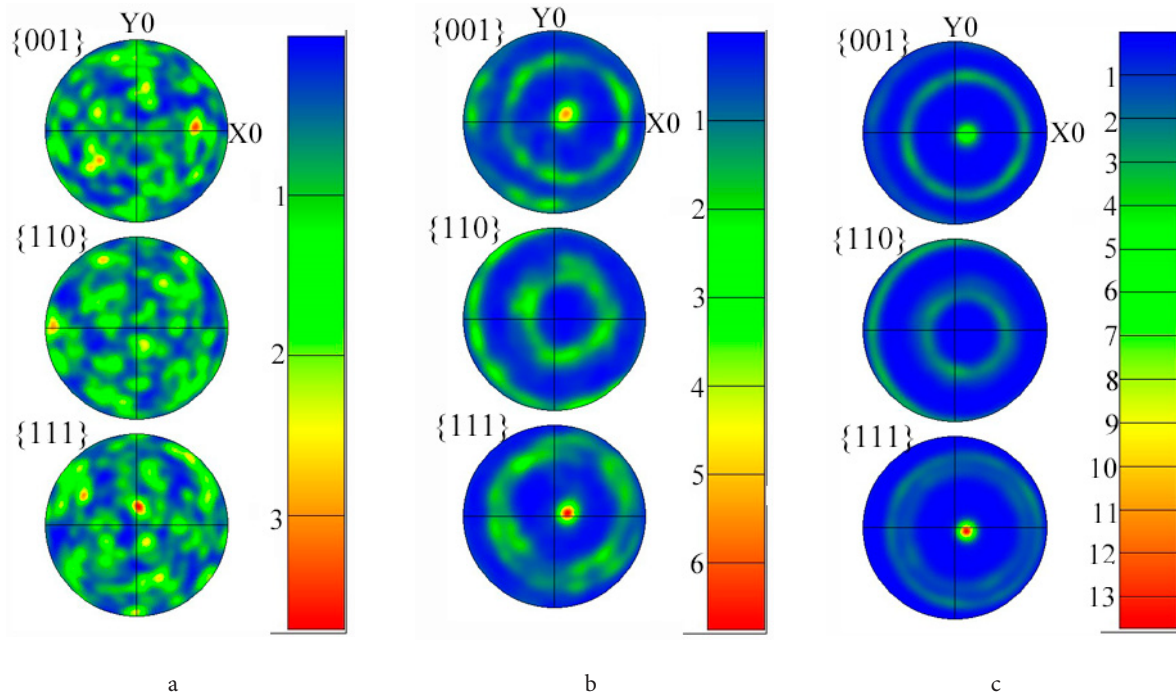


Fig. 5. (Color online) Direct pole figures of the cross-section of the billet made from 58Ni-Cr-Mo-B-Al-Cu superalloy after radial-shear rolling ($K_B=1.14$): sample center (a), 0.5R (b), 0.99R (c).

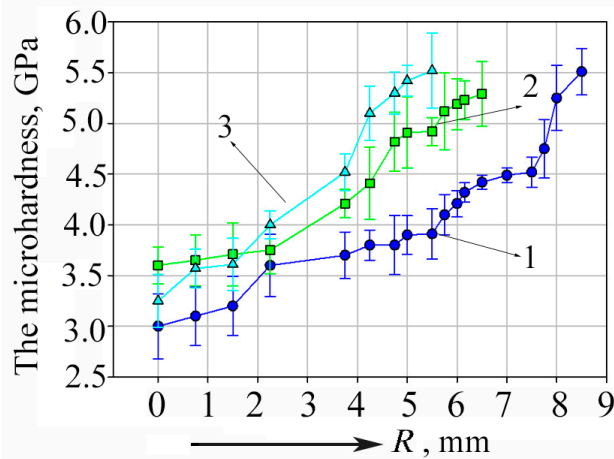


Fig. 6. (Color online) The change of microhardness in radial direction during three stages of rolling.

4. Conclusions

Radial shear rolling makes it possible to form a gradient structure in 58Ni-Cr-Mo-B-Al-Cu superalloy billets, in which the microstructure of the γ -phase changes in the radial direction from the coarse-grained ($34.1 \pm 4.7 \mu\text{m}$) one in the center to the UFG one ($2.4 \pm 1.7 \mu\text{m}$) in the billet surface layers. The fraction of high angle boundaries increases from the center to the periphery by almost twice and the fraction low-angle decreases approximately five times.

There is no texture in the surface layer of the rolled billet. There is a two-component axial texture of the $\langle 001 \rangle + \langle 111 \rangle$ type along the billet axis. The sample center is characterized by the presence of a two-component axial texture of the $\langle 001 \rangle + \langle 111 \rangle$ type along the rolled billet axis, which is more pronounced compared to one on the distance of 0.5R.

The gradient change in the microstructure in the radial direction of the billet after radial shear rolling leads to a gradient change in the microhardness. In the billet center, where the coarse-grained microstructure is formed, the microhardness value is 3.3 GPa, and in the peripheral surface zone, where the UFG structure is formed, the microhardness increases to 5.4 GPa.

Acknowledgment. This study was supported by IMSP RAS state assignment No. AAAA-A17-117041310215-4. Experimental studies were carried out on the facilities of shared services center of the Institute for Metals Superplasticity Problems of Russian Academy of Sciences "Structural and Physical-Mechanical Studies of Materials".

References

1. Ch. Sims, T. Stoloff, V. Hagel. Superalloys II: High-Temperature Materials for Aerospace and Industrial Power. John Wiley & Sons, New York (1987).
2. R. R. Mulyukov, R. M. Imayev, A. A. Nazarov, V. M. Imayev, M. F. Imayev, V. A. Valitov, R. M. Galeev, S. V. Dmitriev, A. V. Korznikov, A. A. Kruglov, R. Ya. Lutfullin, M. V. Markushev, R. V. Safiullin, O. Sh. Sitdikov, V. G. Trifonov, F. Z. Utyashev (ed. by R. R. Mulyukov, R. M. Imayev, A. A. Nazarov, V. M. Imayev, M. F. Imayev). Sverkhplastichnost' ul'tramelkoznistykh splyavov: eksperiment, teoriya, praktika. Moscow, Nauka (2014) 284 p. (in Russian)
3. V. A. Valitov. Letters on Materials. 3 (1), 50 (2013). (in Russian) [Crossref](#)
4. E. V. Valitova, R. Ya. Lutfullin, V. A. Valitov. Perspektivnye materialy. 15, 30 (2013). (in Russian)
5. E. Yu. Klassman, V. V. Astanin. Letters on Materials, 10 (1), 10 (2020). (in Russian) [Crossref](#)
6. R. R. Mulyukov, R. M. Imayev, A. A. Nazarov. Scientific and

- technical statements SPbPU. Physical and mathematical sciences. 4–1 (182), 190 (2013). (in Russian)
7. R.Z. Valiev, I.V. Alexandrov. Bulk nanostructured metallic materials. Akademkniga, Moscow (2007) 392 p. (in Russian)
 8. E. V. Valitova, R. Ya. Lutfullin, M. H. Muhametrakhimov, V. A. Valitov. Perspektivnye materialy. 15, 35 (2013). (in Russian)
 9. G. F. Korznikova, A. P. Zhilyaev, A. A. Sarkeeva, R. Ya. Lutfullin, R. U. Shayahmetov, G. R. Khalikova, R. Kh. Khisamovg, K. S. Nazarov, R. R. Mulyukov. Metallic Composites, Prepared by Deformation Processing. Materials Science Forum. 1016, 1759 (2020). [Crossref](#)
 10. I. A. Ditenberg, S. A. Malakhova, A. N. Tyumencev, A. V. Korznikov. Perspektivnye materialy. 12, 306 (2011). (in Russian)
 11. I. Sh. Valeev, A. H. Valeeva, R. F. Fazlyakhmetov, G. R. Halikova. Materials science. 7, 27 (2010). (in Russian)
 12. A. K. Valeeva, I. S. Valeev. Letters on Materials. 3 (1), 38 (2013). (in Russian) [Crossref](#)
 13. S. Dobatkin, S. Galkin, Y. Estrin, V. Serebryany, M. Diez, N. Martynenko, E. Lukyanova, V. Perezhogin. Journal of Alloys and Compounds. 774, 969 (2019). [Crossref](#)
 14. B. A. Romancev, V. K. Mikhailov, S. P. Galkin, M. G. Degtyarev, B. V. Karpov, A. P. Chistova. Patent RF № 2009737, 30.03.1994. (in Russian)