

Article

Superior Strength of Austenitic Steel Produced by Combined Equal-Channel Angular Pressing and Rolling Processing

Marina V. Karavaeva *, Marina M. Abramova, Nariman A. Enikeev, Georgiy I. Raab and Ruslan Z. Valiev

Ufa State Aviation Technical University, 12 K. Marx str., Ufa 450008, Russia, abramovamm@yandex.ru (M.M.A.); nariman.enikeev@gmail.com (N.A.E.); giraab@mail.ru (G.I.R.); rzvaliev@yahoo.com (R.Z.V.)

* Correspondence: karma11@mail.ru; Tel.: +7-917-781-7784

Abstract: Enhancement in the strength of austenitic steels with a small content of carbon can be achieved by a limited number of methods, among which is the ultrafine-grained (UFG) structure formation, especially efficient with the use of severe plastic deformation (SPD) processing that enables increasing significantly the contribution of grain-boundary strengthening, and also involves a combination of other strengthening factors (work hardening, twins, etc.). In this paper, we demonstrate that the use of SPD processing combined with conventional methods of deformation treatment of metals, such as rolling, may lead to an additional strengthening of UFG steel. Analysis of the results of the study on the change of the microstructure and mechanical properties of the Cr-Ni stainless austenitic steel after a combined deformation reveals a substantial increase in the strength properties of this steel, resulting from a consecutive application of SPD processing via equal-channel angular pressing and rolling at a temperature of 400 °C, yielding a strength more than 1.5 times higher than that produced by any of these two methods used separately.

Keywords: stainless steel; severe plastic deformation; strength

1. Introduction

Severe plastic deformation (SPD) processing of a broad range of metals and alloys enables increasing their mechanical properties significantly due to the formation of an ultrafine-grained structure, ensuring a concurrent action of several mechanisms of strengthening, such as solid solution strengthening, precipitation hardening, dislocation strengthening and, primarily, grain-boundary strengthening [1-7]. A high-strength state is provided by a scientifically-based control of the microstructural parameters that are most sensitive to processing regimes. Such SPD processing regimes that have the most effect on the microstructure are strain, temperature, loading rate and type. The latter has an effect on both the kinetics of microstructure evolution and the homogeneity of the produced microstructure. For example, when studying the microstructure transformation of Ti alloys with a change of deformation type, it was shown that the substitution of a monotonic loading with essentially non-monotonic loading enabled activating new slip systems and thus intensifying the process of microstructure evolution [8, 9]. With respect to SPD processing, it was demonstrated that the so-called route of equal-channel angular pressing (ECAP) has a great effect on the evolution of structure [10, 11]. The best results, in terms of microstructure refinement and enhancement of mechanical properties, were obtained when using routes B and Bc, in which the billet is rotated about its axis between ECAP passes. Such a turn changes the schemes of the principal stresses and strains in a material, and as a result, the deformation process becomes

non-monotonic. Similar result was obtained for the cyclic HPT when the sufficient grain refinement in Ni and Fe was reported to be achieved at the less deformation level then for the one-direction HPT [12]. A vivid example of non-monotonic loading is the SPD technique of multiple forging, in which the change of the scheme of principal stresses is effected as a result of a consecutive rotation of the billet about three axes [13, 14].

It is possible to realize the non-monotonic loading process through a consecutive processing of billets by different methods. This path has already been tested successfully for Ti-based [15], Cu-based [16] and Al-based alloys [17, 18]. At the first stage of processing, SPD processing (ECAP-Conform) was conducted, and at the second stage, rolling (drawing) was performed. It is marked in all studies that a change in the type of loading has a beneficial effect on the properties of materials. At the second stage of deformation processing, there is observed an additional increase in the microhardness and strength of ultrafine-grained materials, produced by SPD at the first stage of processing. It is more difficult to determine unambiguously the effect of a change in the deformation type of UFG materials on the features of microstructure. At the present time, the experimental data reported in literature are not sufficient to summarize the results. Besides, of great importance is the microstructure formed immediately during SPD processing, as well as the nature of the material itself. After the rolling of even an equiaxed UFG structure, a structure was observed that was elongated in the direction of plastic straining. For copper, an increase in the structural homogeneity was revealed [16], and on the contrary, for an Al alloy, a division of microstructure into two fractions was observed, one of which contained shear bands, and the other one contained equiaxed grains [17].

In this paper, we investigate the possibility of increasing, through the use of a combined processing, the strength of an austenitic stainless steel, for which it is practically impossible to increase strength by thermal treatment, and thus microstructure refinement by deformation processing is an efficient means of strengthening.

2. Materials and Methods

As the material for the study, the austenitic stainless steel was selected. The chemical composition of the steel is given in Table 1.

In order produce a single-phase austenitic structure, prior to deformation processing the steel was water-quenched from a temperature of 1050 °C (holding time 1 h).

The SPD processing of rods with a diameter of 10 mm and a length of 100 mm was effected by ECAP through 8 passes at a temperature of 400 °C, the intersection angle of channels in the die-set was 120 ° (Fig.1), route Bc.

Table 1. Chemical composition of the austenitic steel under investigation

C	Cr	Ni	Ti	Si	S	P	Fe
0.08	16.19	9.13	0.3	0.58	0.03	0.08	bas.

The thermal conditions of ECAP processing were selected in accordance with earlier studies [6, 19] that demonstrated the efficiency of SPD processing for microstructure refinement and enhancement of the mechanical properties of the austenitic stainless steel at the given temperature, as well as for the formation of grain-boundary segregations and nanotwins resulting in an

additional enhancement of the strength properties. The number of passes was selected in such a way as to be sufficiently large to produce such a strain under which the hardness and strength of a UFG billet reach saturation. This condition is further referred to as «ECAP».

Rolling was conducted in smooth rolls at the same temperature of billet heating, 400 °C, through 15 passes to a final strip thickness of 2.3 mm. The total relative strain was 77 % (Fig.1). This condition is further referred to as «ECAP+Rol». This regime was selected on the basis of the above-mentioned considerations, as well as to preserve the integrity of the billet.

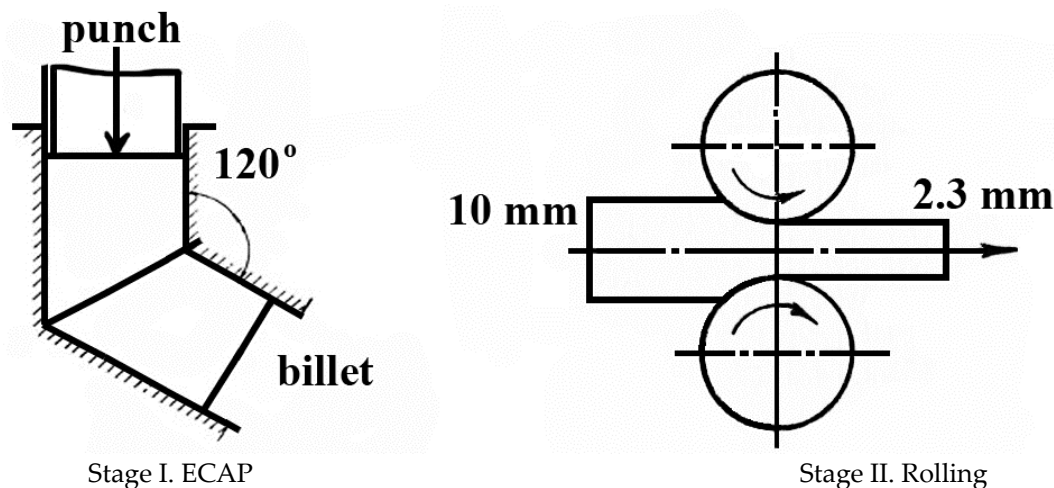


Figure 1. Principle of the combined processing of the steel

To study the effect of the combined processing on the microstructure and properties, also investigated were billets subjected to rolling under the same regimes, but without a preliminary deformation processing by ECAP. This condition is further referred to as «Rol».

The microstructure was studied in the longitudinal section of a rod and a strip. To reveal the structure, electrolytic etching was performed in a chemically pure nitric acid (the mass fraction of the acid was not less than 65%). The etching time was from 5 to 10 seconds under a voltage of 13-20 V. Structural studies were performed using an Olympus GX51 optical microscope, a JEOL JSM-6490VL scanning electron microscope and a JEOL JEM-2100 transmission electron microscope.

The grain sizes were determined from the dark-field images of the structure. At least 300 grains were measured for each condition.

The dislocation density ρ_{xrd} was determined from the results of X-ray studies according to the expression:

$$\rho_{xrd} = \frac{2\sqrt{3} \langle \epsilon^2 \rangle^{\frac{1}{2}}}{b d_{xrd}},$$

where $\langle \epsilon^2 \rangle^{1/2}$ is the level of elastic microdistortions of the crystal lattice; b is the Burgers vector of dislocations; d_{xrd} is the size of coherent scattering regions.

Microhardness was measured on a Micromet-5101 device in the longitudinal directin. At least 30 measurements were made for each condition.

Uniaxial tensile testing was performed on an INSTRON 8801 tensile testing machine at room temperature. For the tensile tests, flat samples with a gauge length of 4 mm were used, the strain rate was 10^{-3} s^{-1} .

3. Results

The microstructure of the steel in the as-received condition was represented by equiaxed austenite grains with a mean size of $(9\pm2) \mu\text{m}$ (Fig. 2, a). In some grains, twins were observed. The volume fraction of grains containing twins was about 10 %.

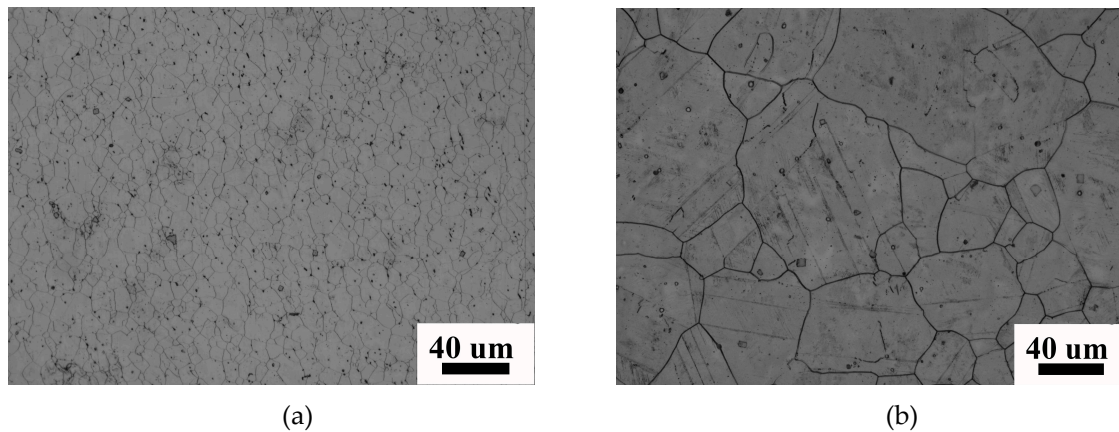


Figure 2. Microstructure of the austenitic steel: as-received condition (a); after quenching (b)

After quenching, the sizes of austenite grains increased to an average value of $(40\pm11) \mu\text{m}$. Practically all grains contained wide twins. At the boundaries of austenite grains, at twin boundary/grain boundary intersections, serrations were observed.

- Microstructure of the austenitic steel after SPD processing and rolling

After SPD processing by ECAP, within the austenite grains there was observed the formation of differently-directed shear bands (Fig.3, a). As a result of intersection of these bands, new boundaries form and grain refinement takes place. The microstructure is characterized by non-homogeneity. At 10000 times magnification (Fig.3, b), there are visible relatively coarse grains with sizes of several μm and fine grains with sizes much smaller than $1 \mu\text{m}$. The coarse grains are elongated in the direction of the sample axis (Fig. 3, a). The volume fraction of the regions with relatively coarse grains amounts to about 10 %.

When the structure is examined in detail by TEM, structural non-homogeneity is also revealed (Fig.3, c). A large volume of the structure (about 60 %) is represented by shear bands with thin boundaries, within which there is observed a developed dislocation structure in the form of wide dislocation boundaries. These boundaries divide the bands into non-equiaxed cells. The cell sizes amount to, on average, about 180 nm in the transverse direction and 370 nm in the longitudinal direction (Fig.3, c). Alongside with shear bands, in the structure there are present practically equiaxed grains with a reduced dislocation density and thin equilibrium boundaries. The grain sizes are about 350 nm. In the grains, there are present separate very thin (about 10 nm in thickness) deformation twins (Fig.3, d). The fraction of grains with twins does not exceed 5 %. The average distance between the twin boundaries in grains with twins is about 75 nm. The electron diffraction pattern shown in the insert in Fig.3 c reveals separate reflections located circumferentially, which indicates high-angle misorientations of grains.

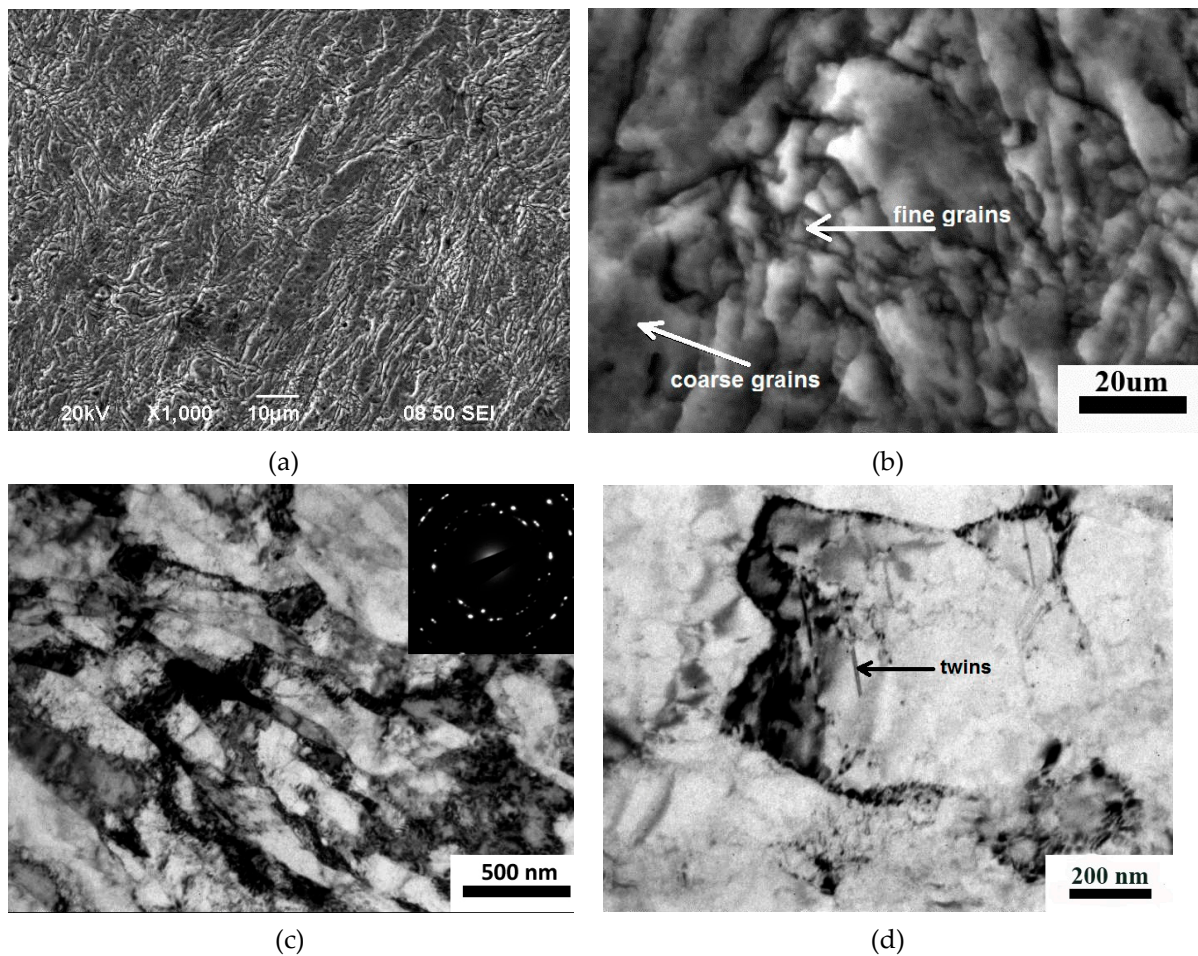
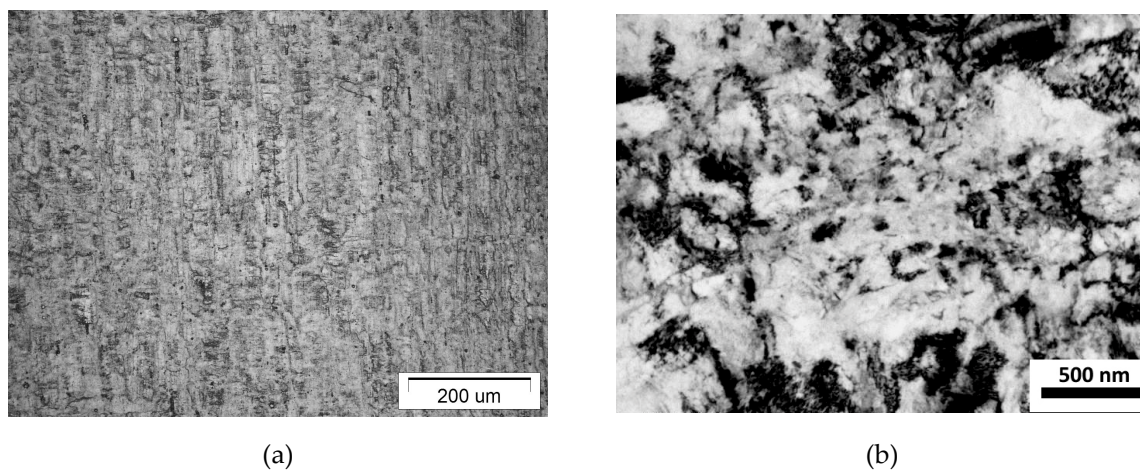


Figure 3. Microstructure of the steel after SPD processing (via ECAP) in the longitudinal section («ECAP» condition): (a), (b) – SEM; (c),(d) – TEM

Thus, after SPD processing via ECAP, there is formed a non-homogeneous austenitic UFG structure, elongated in the direction of straining, with a developed network of grains/subgrains and a small number of twins.

After rolling of the ECAP-processed steel, further grain refinement is observed (Fig.4). Individual grains are practically not identified by an optical microscope. The boundaries of the original austenite grains are not visible either (Fig.4, a).



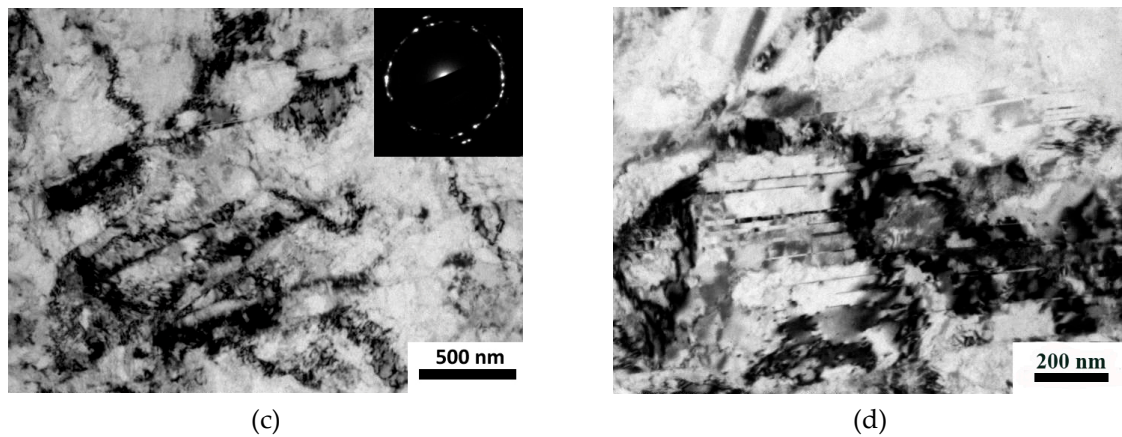
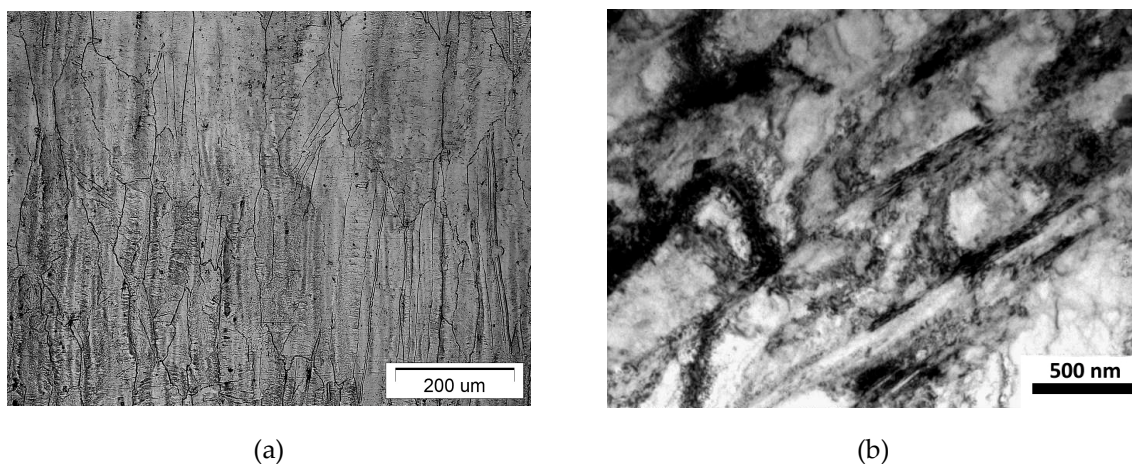


Figure 4. Structure of the austenitic steel after ECAP and subsequent rolling to a total strain of 77 % («ECAP+Rol» condition): a – optical microscopy; b – d - TEM

When the microstructure is examined by TEM, it can be seen that the microstructure has become more homogeneous (Fig.4, b) as compared to the one observed in the «ECAP» condition (Fig.3, c). The structure has a grain/cellular character. Shear bands are preserved in separate regions, but the fraction of banded structure is only about 10 %. The dislocation density increases, while the size of structural elements decreases to 110 nm. In the grains, there are observed thin twins (Fig.4, c). The fraction of grains containing twins increases to 14 %. The average distance between the twin grains decreases to 30 nm. The electron diffraction pattern shown in the insert of Fig.4, b has a ring-shaped form, which indicates high-angle misorientation.

Thus, combined loading leads to a further microstructure refinement – the mean grain size decreases to 110 nm, the fraction of nanotwins grows.

In the steel samples after rolling («Rol» condition) there can be seen distinctly the boundaries of original austenite grains (Fig.5, a), elongated in the rolling direction. Within the grains there form shear bands. At the boundaries of the original austenite grains, at the shear band/grain boundary intersections, steps are observed.



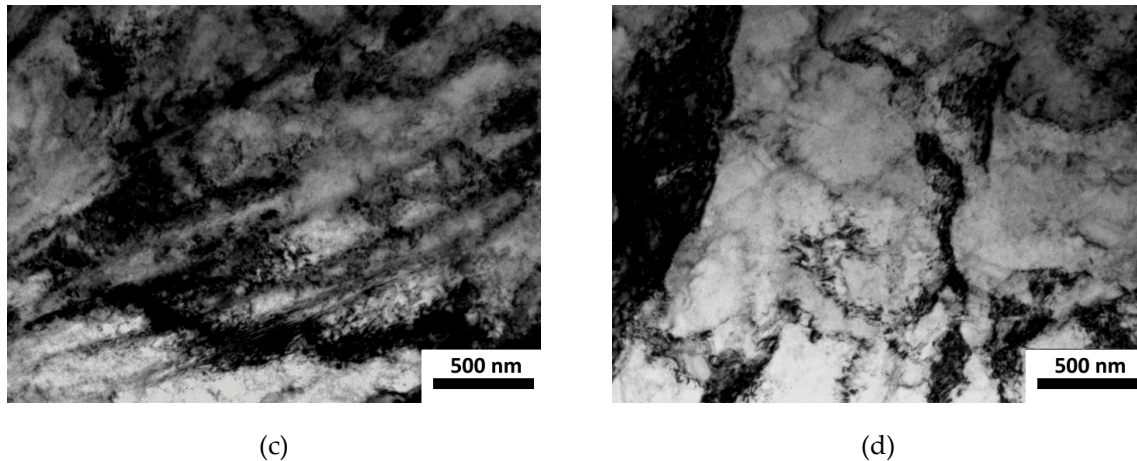


Figure 5 Structure of the austenitic steel after rolling to a total strain of 77 % («Rol» condition): (a) – optical microscopy; (b) – (d) - TEM

There is also observed a banded structure (Fig.5, b, c). Inside the bands there are wide boundaries dividing grains into cells (Fig.4, b, c, d). The average cell size amounts to 560 nm. The structure is characterized by an increased dislocation density. Twins are practically absent.

Thus, in the «Rol» condition, the steel is characterized by a banded cellular structure with a cell size of 560 nm, which does not contain twins.

- Mechanical properties of the austenitic steel

The average microhardness value of the austenitic steel in the as-received condition is (1970±60) MPa (Fig.6). After quenching microhardness declines slightly to a value of (1820±30) MPa, which is related to the growth of austenite grains, as well as to a more complete dissolution of excess phases during heating prior to quenching.

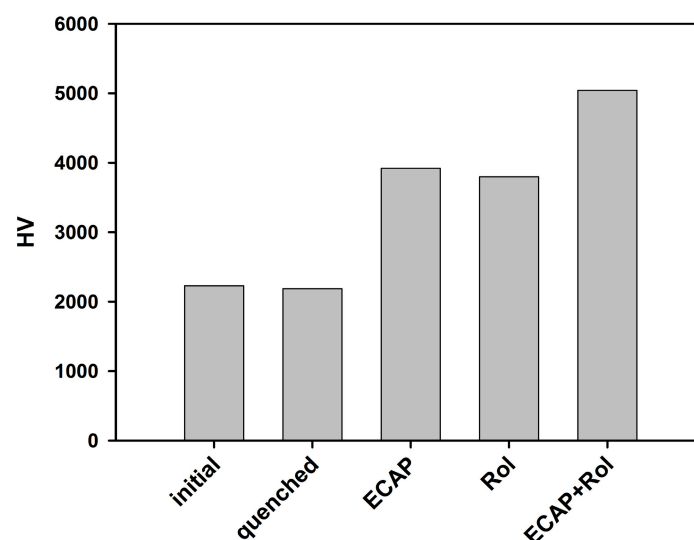


Figure 6. Microhardness of the austenitic steel after different types of processing

As a result of microstructure refinement, in the «ECAP+Rol» condition the average microhardness value of the steel grows two-fold, reaching (3920±50) MPa. After rolling to 77 % without a preliminary ECAP processing («Rol»), there is also observed an increase in hardness, very

similar to ECAP processing – to 3800±50 MPa. A combination of ECAP and rolling results in an additional increase in microhardness by 35-40 %, reaching (5040±40) MPa (Fig.6).

In a similar manner, straining has an effect on the steel's strength as well. Fig. 7 shows the engineering stress-strain curves obtained during tensile tests of the steel samples in different conditions. It is obvious that the deformation behavior of the material changes depending of the type of processing of the steel.

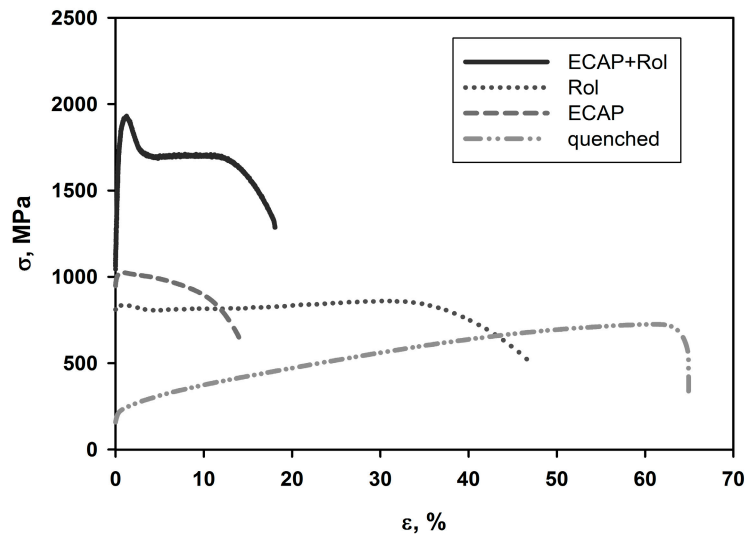


Figure 7. Engineering stress-strain curves of the steel after different types of processing

The values of the steel's mechanical parameters in different conditions are given in table 2.

Table 2. Mechanical properties of the steel after different types of processing

Condition	Offset yield stress $\sigma_{0.2}$, MPa	Upper yield stress (corresponds to yield)	Lower yield stress (corresponds to yield)	Ultimate tensile strength σ_{ult} , MPa	Uniform elongation $\delta_{uniform}$, %	Elongation to failure δ , %	Microhardness HV, MPa
quenching	200	-	-	720	62	65	1820
ECAP	950	-	-	1020	1	14	3920
Rol	-	830	800	855	33	47	3800
ECAP + Rol	-	1925	1700	1720	11	18	5040

Peculiar of the quenched condition is a significant strengthening together with a large elongation value: the yield stress is $\sigma_{0.2}$ =200 MPa, the ultimate tensile strength (UTS) is 2.5 times higher – σ_{ult} =720 MPa; the elongation is δ =65 %.

For the steel in the «ECAP» condition, peculiar is a high value of yield stress $\sigma_{0.2}$ =950 MPa and a very short period of insignificant strengthening: the uniform elongation is only 1 %, the UTS is σ_{ult} =1020 MPa. After that, a rapid strain localization takes place in the neck, corresponding to the region with a stress decline in the diagram. The total elongation is δ =14 %.

In spite of similar values of microhardness and yield stress in the conditions «ECAP» and «ECAP+Rol», the deformation behavior for these two conditions differs significantly. In the curve of the rolled sample, there appears a weakly expressed yield drop, to which corresponds the upper yield stress $\sigma_u=800$ MPa. After that there is observed a yield plateau, to which corresponds the lower (physical) yield stress $\sigma_l=800$ MPa and the region of weak strengthening to a strain of 33 %. The UTS is $\sigma_{ult}=855$ MPa. The elongation to failure of the sample is $\delta=47$ %.

The highest strength is exhibited by the samples after the combined loading «ECAP+Rol». This condition is displayed by the curve with a distinct yield drop. The upper yield stress is $\sigma_u=1925$ MPa, and the lower yield stress, corresponding to the yield plateau, is $\sigma_l=1700$ MPa. The curve does not demonstrate any strengthening, strain localization starts as elongation reaches the value $\delta=11$ %, and the total elongation is $\delta=18$ %.

Thus, the type of deformation processing determines not only the level of properties, but also the tensile mechanical behavior of the steel.

4. Discussion

Numerous studies on the SPD processing of bulk billets of metals and alloys via ECAP have demonstrated that the greatest microstructure refinement and, hence, an increase in hardness and strength is observed after the initial 1-2 passes, after which strengthening becomes much slower [20-22]. Meanwhile, the possibilities for strength enhancement in a material have not yet been exhausted. This is suggested by the fact that under processing by high-pressure torsion, as a rule, the observed hardness values are significantly larger than the ones that can be attained by ECAP processing [10].

The strength of metals and alloys is determined by the strengthening mechanisms that are active under the given structure of a material. It is evident that it is possible to «turn on» this or that mechanism through a directed action on the microstructural features due to the loading regimes and type under SPD.

In UFG materials produced by SPD, strengthening is achieved due to several mechanisms [1-7, 22]:

1. Grain-boundary and dislocation strengthening. During the formation of a submicrocrystalline structure, the density of grain boundaries, which are an efficient impediment for dislocation movement, significantly increases. For the formation of new boundaries of deformation origin, dislocation generation in various slip systems is necessary.

When analyzing the types of loading realized in the course of SPD processing, it is necessary to mention two distinctive features typical for SPD processing:

- 1) a high hydrostatic constituent, which is especially significant in high-pressure torsion, but present in all deformation techniques;
- 2) an essential non-monotony of strain, typical for most SPD techniques, such as ECAP or multiple forging.

Both of these features enable activating additional slip systems, thus leading to an increase in dislocation density, formation of new interfaces and refinement of structural elements.

2. Solid-solution strengthening and precipitation hardening. These mechanisms are competing ones, since when the alloying of as solid solution (solid-solution strengthening) grows,

the quantity of dispersed particles (precipitation hardening) decreases. The contribution of this constituent into strengthening is determined primarily by the deformation temperature. It is shown [23, 24] that at room temperature the dissolution of second-phase particles prevails, but as the deformation temperature is increased, precipitation of dispersed particles is also observed.

3. Formation of segregations at grain boundaries. This process is also connected with the dissolution of particles during SPD and a transfer of solute atoms to the deformation boundaries. The action of this mechanism is also determined to a great extent by the deformation temperature: at room temperature the formation of segregations is observed, and at elevated temperatures second-phase particles are observed more often at grain boundaries [25]. It is not known how hydrostatic pressure or a change in the loading scheme influences the formation of segregations or dispersed particles.

4. Formation of twins. For a number of materials, including austenitic steels, it is typical that during SPD processing there form nanotwins. The high-angle boundaries of nanotwins are also impediments for dislocation movement and, consequently, lead to strengthening. Twinning may be activated when possibilities for slip are limited. One of the factors activating twinning is the hydrostatic constituent of loading.

During ECAP processing via route Bc, the billet's rotation by 90° in each new pass activates new slip planes. However, these planes are located at the same angle with respect to the billet axis, since the billet's axis orientation with respect to the direction of load application does not change. Therefore, strengthening takes place during the initial passes of deformation processing until a deformation texture is formed, where the location of predominant slip systems along the direction of action of the maximum tangential stresses is observed. After the formation of such a texture, slip is concentrated in these planes.

When the scheme of the stress and strained state is changed (in this particular case, by changing the type of loading), the direction of action of the maximum tangential stresses changes with respect to the sample's axis. As a result of such a change, new slip systems should be activated, and the activation of twinning is also possible.

Thus, a change in the loading scheme may activate at least two of the above-mentioned factors of strengthening: an increase in dislocation density and an increase in the length of grain boundaries. These conclusions are confirmed by studies conducted on various materials. For instance, in the paper [17], in the Al alloy 5083 after ECAP processing and additional compression, imitating rolling conditions, an increase in dislocation density was observed. An enhancement of strength after ECAP-Conform processing and compression of Ti [15], after ECAP processing and rolling of Cu [16], was accounted for by the formation of additional low-angle boundaries within grains and a transformation of the low-angle boundaries into high-angle ones.

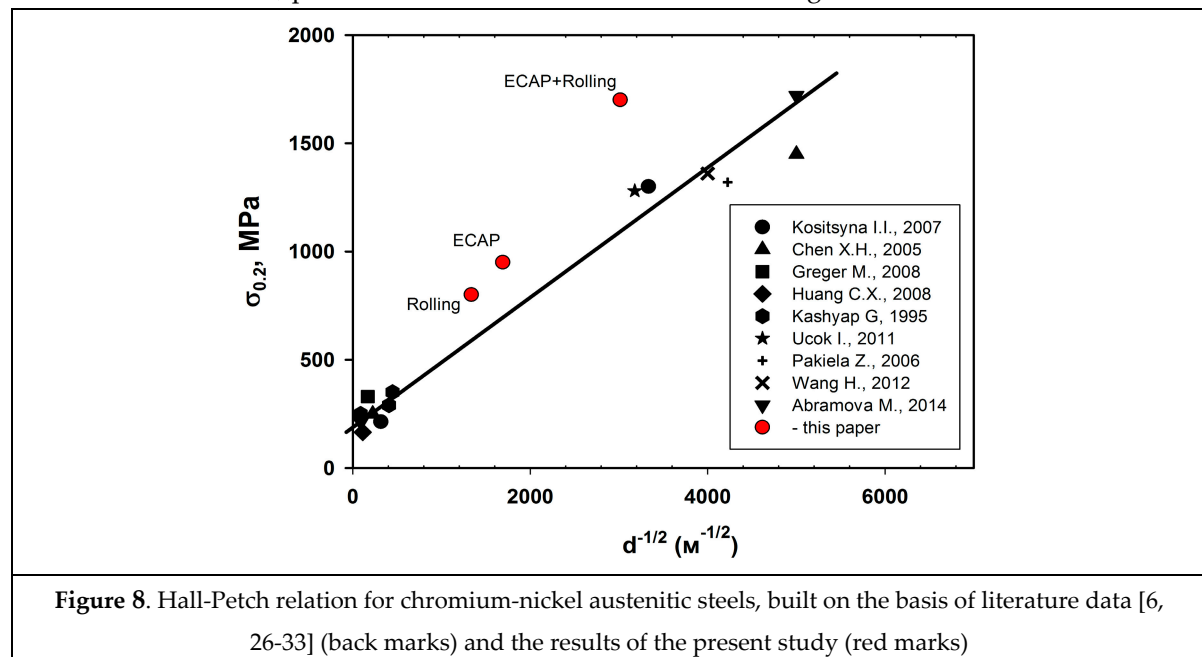
This conclusion is also confirmed by the studies on the microstructure of austenitic steel produced by such techniques as ECAP, rolling and ECAP+rolling (see table 3).

Table 3. Features of the structure of steel after straining

Condition	Dislocation density, m ⁻²	Grain/cell size, nm	Fraction of shear bands	Fraction of grains with twins, %	Twin spacing, nm
ECAP	1.28*10 ¹⁴	350	60	5	75
ECAP+ Rol	7.19*10 ¹⁴	110	10	14	30
Rol	4.27*10 ¹⁴	560	80	-	-

Comparison of the microstructural parameters of steel in different conditions demonstrates that as a result of combined loading, the size of structural elements considerably decreases as compared with the «ECAP» condition and the «rolling» condition. This leads to a considerable increase in the density of grain/cell boundaries.

Let us consider a generalized dependence of yield stress on grain size in accordance with the Hall-Petch relation, presented on the basis of literature data in Fig.8.



The results obtained in the present study are also presented in the graph. It can be seen that the points corresponding to the conditions «ECAP» or «Rol» have a certain deviation from the line summarizing literature data towards larger values of yield stress. However, the point corresponding to the «ECAP+Rol» condition is located much higher than expected in accordance with the Hall-Petch relation.

It is known that strengthening of nanostructured steels is provided not only by grain boundaries. For austenitic steels, additional strengthening is introduced by the dislocation mechanism, as well as twin boundaries, as it was demonstrated in [6]. In the general case, the contributions of different mechanisms follow linear additivity [2, 3, 5-7, 22]:

$$\sigma_T = \Delta\sigma_{FS} + \Delta\sigma_{SS} + \Delta\sigma_p + \Delta\sigma_{GB} \quad (1)$$

where σ_{FS} is the friction stress of α -iron's lattice; $\Delta\sigma_{SS}$ is solid-solution strengthening; $\Delta\sigma_{\rho}$ is dislocation strengthening; $\Delta\sigma_{GB}$ is grain-boudary strengthening.

Let us estimate the contribution of these mechanisms the into yield stress of the investigated steel in each condition.

The total strengthening can be defined from the Hall-Petch relation, displayed in Fig.8, as the stress corresponding to the infinitely large grain (single crystal). In the given case, it is $\sigma_0 = 195$ MPa.

Dislocation strengthening is defined as:

$$\Delta\sigma_{\rho} = \alpha M b G \rho^{1/2},$$

where $\alpha=0.3$ is a constant; $M=3.05$ is the Taylor factor; $G=77$ GPa is the shear modulus and $b=\sqrt{2}/2 a$ is the Burgers vector for the investigated steel.

Let us define grain-boundary strengthening, taking into account the presence of twins, as follows [6]:

$$\Delta\sigma_{GB} = (1-f)k_y d^{-1/2} + f k_y \lambda^{-1/2},$$

where f is the fraction of grains with twins; $k_y=0.3$ MPa*m^{1/2} is a constant derived from the dependence in Fig.8; d is the average grain/cell size; λ is the average distance between twin boundaries in grains containing twins.

The estimation results are given in table 4.

Table 4. Estimation results for the contribution of different mechanisms into the strengthening of the steel

Condition	$\Delta\sigma_{\rho}$	$\Delta\sigma_{GB}$			Calculated value	Experimental value
		$\Delta\sigma_d$	$\Delta\sigma_{tw}$	$\Delta\sigma_d + \Delta\sigma_{tw}$		
ECAP	202	481	55	536	933	950
ECAP+Rol	480	778	242	1020	1688	1700
Rol	370	401	-	401	771	800

The estimated results are very close to the experimental values of yield stress. Microstructural studies and the presented estimations show that strength enhancement of the steel under a combined loading is provided predominantly by the grain-boundary constituent in accordance with the Hall-Petch equation. Besides, unlike in ECAP processing, after rolling at a temperature of 400 °C no twins were observed in the structure of the steel. After a combined loading, the fraction of twin structure increases even as compared to the «ECAP» condition, and this component also makes a significant contribution to the steel's strengthening (see table 4). The dislocation contribution into yield stress grows almost two-fold.

In addition to further strengthening, the combined loading of «ECAP+Rol» has an effect also on the steel's deformation behavior, which is principally different not only from the quenched condition, but also from the steel's behavior in the conditions «ECAP» and «Rol». In the quenched condition, the steel's structure is characterized by a small density of grain boundaries and wide twins. Under deformation, dislocation density increases in such a structure, and as a result, an extensive region of strengthening and a high ductility are observed in the curve.

In the «ECAP» condition, we observed the view of the stress-strain curve typical for materials subjected to SPD – the maximum stress under small strains, rapid macrolocalization of strain and failure.

After «ECAP+Rol», in the curve there is observed a distinct yield drop. Its appearance could be caused by segregations. The formation of segregations during SPD processing was found in recent years in many materials, including austenitic steels [6]. In the samples after rolling, the appearance of a weakly expressed yield drop indicates that the formation of segregations or atmospheres on dislocations is peculiar for rolling. Evidently, the formation, during ECAP processing, of an ultrafine-grained structure with a high density of grain boundaries stimulates segregation formation during subsequent rolling, which is expressed in the yield phenomenon observed in the curve. The contribution of segregations can be estimated as the difference between the upper and the lower yield stresses, which amounts to 225 MPa for the steel in the «ECAP+Rol» condition. However, this issue requires an additional detailed study.

Note should also be made that for the steel after «ECAP+Rol» peculiar are rather high values of both uniform (11 %) and total (18 %) elongation. This may also be related to the blocking of dislocations by atmospheres or segregations of alloying element atoms: after the disruption of the blocking of a large quantity of dislocations, their free movement is possible, thus ensuring an additional deformation of the sample.

Thus, the application of the combined technique of ECAP+Rol results in a considerable growth in the density of grain/cell boundaries and increases the fraction of twins in the microstructure, which enables enhancement of the strength characteristics, at the same time preserving the ductility of the UFG austenitic steel.

5. Conclusions

1) A combination of SPD processing and conventional metal forming techniques, realized through a combined loading via «ECAP+Rol» of the austenitic steel, leads to a further refinement of a homogeneous UFG cell-granular microstructure with a high density of grain boundaries and a large fraction of twin structure produced by ECAP processing.

2) As a result, the tensile mechanical behavior of the UFG steel samples produced by the combined loading changes – there appears a yield drop, to which corresponds the upper yield stress of 1925 MPa, as well as the yield plateau, the yield stress amounted to 1700 MPa. The reached values of strength are 1.5 time higher than the values of yield stress obtained when using only the ECAP technique (950 MPa) or only rolling (~815 MPa). Besides, in the UFG sheet produced by the combined loading a rather high level of ductility is preserved: a uniform elongation of 11 %, a total elongation of 18 %.

3) The enhancement of the strength characteristics is achieved as a result of a combined action of several strengthening mechanisms: grain-boundary strengthening, dislocation strengthening,

twinning-induced strengthening and, presumably, strengthening due to the formation of segregations of alloying element atoms.

Acknowledgments: The authors acknowledge the financial support by the Ministry of Science and Education of Russian Federation under Grant agreement no.14.583.21.0012 (unique identification number RFMEFI58315X0012) and by the International Research & Development Program of the National Research Foundation of Korea (NRF) funded by the Ministry of Science, ICT and Future Planning (MSIP) of Korea 20 (Grant no.K1A3A1A49.070466, FY2014). A part of investigations was conducted using the facilities of collective research Nanocentre (Ufa State Aviation Technical University).

References

1. Valiev R.Z., Estrin Y., Horita Z., Langdon T.G., Zehnbauer M.J., Zhu Y.T. Fundamentals of superior properties in bulk nano SPD materials. *Mater.Res.Lett.*, 2016, Vol.4, No 1, pp.1-21.
2. Valiev R.Z., Enikeev N.A., Langdon T.G. Towards superstrength of nanostructured metals and alloys, produced by SPD. *Kovove Mater.* 49, 2011, pp. 1-9.
3. Hasan H.S., Peet M.J., Avettand-Fénoël M.-N., Bhadeshia H.K.D.H. Effect of tempering upon the tensile properties of a nanostructured bainitic steel. *Mat.Sci. and Eng.A* 615 (2014), pp.340-347.
4. R. Z. Valiev, Bulk nanostructured materials: fundamentals and applications / by Ruslan Z. Valiev, Alexander P. Zhilyaev, Terence G. Langdon. 2013, pp. 456. - ISBN: 978-1-118-09540-9.
5. Kamikawa N., Abe Y., Miyamoto G., Funakawa Y., Furuhashi T. Tensile behavior of Ti,Mo-added low carbon steels with interphase precipitation. *ISIJ International*, Vol.54 (2014), No.1, pp.212-221.
6. Abramova M.M., Enikeev N.A., Valiev R.Z., Etienne A., Radigue B., Ivanisenko Y., Sauvage X. Grain boundary segregation induced strengthening of an ultrafine-grained austenitic stainless steel. *Materials letters* 136 (2014), pp. 349-352.
7. Ganeev A.V., Karavaeva M.V., Sauvage X., Courtois-Manara E., Ivanisenko Y., Valiev R.Z. On the nature of high-strength of carbon steel produced by severe plastic deformation. *IOP Conf. Series Materials Science and Engineering* 63 (2014), 012128. Doi:10.1088/1757-899X/63/1/012128.
8. Bylja O.I., Vasin R.A., Ermachenko A.G., Karavaeva M.V., Muravlev A.V., Chistjakov P.V. The influence of simple and complex loading on structure changes in two-phase titanium alloy. *Scripta Materialia* Vol.36 (1997), № 8, pp.949-954.
9. Berdin, V.K., Karavaeva, M.V., Syutina, L.A. Effect of the type of loading on the evolution of microstructure and crystallographic texture in VT9 titanium alloy. (2003) *Metal Science and Heat Treatment* 45 (11-12) PP. 423 - 427 doi: 10.1023/B:MSAT.0000019195.58304.6e
10. R.Z. Valiev, A.P. Zhilyaev, T.G. Langdon, Bulk Nanostructured Materials: Fundamentals and Applications, 2014 by John Wiley & Sons, Inc., 456 pages..
11. Iwahashi Y., Horita Z., Nemoto M., Langdon T.G. The process of grain refinement in equal-channel angular pressing. *Acta mater.* Vol.46, No.9, pp.3317-3331, 1998.
12. Wetscher F., Pippan R. Cyclic high-pressure torsion of nickel and armc iron. *Philosophical Magazine*, Vol.86, No.36, 2006, pp.5867-5883.

13. G.Salischev, R.Zaripova, R.Galeev, O.Valiahmetov Nanocrystalline structure formation during severe plastic deformation in metals and their deformation behavior. *Nanostructured Materials*, Vol.6, pp.913-916, 1995.
14. A.Belyakov, K.Tsuzaki, R.Kaibyshev Nanostructure evolution in an austenitic stainless steel subjected to multiple forging at ambient temperature. *Mat.Sci.Forum*, Vols.667-669 (2011), pp.553-558.
15. Polyakov A., Gunderov., Sitdikov V., Valiev R., Sevenova I., Sabirov I. Physical Simulation of hot rolling of ultra-fine grained pure titanium. *Metall. Trans. B*, V.45B, December 2014, pp.2315-2326.
16. Stepanov N.D., Kuznetsov A.V., Salischev G.A., Raab G.I., Valiev R.Z. Effect of cold rolling on microstructure and mechanical properties of copper subjected to ECAP with various number of passes. *Mat. Sci. and Eng. A* 554 (2012), pp. 105-115.
17. Murashkin M.Yu., Enikeev N.A., Kazykhanov V.U., Sabirov I., Valiev R.Z. Physical simulation of cold rolling of ultra-fine grained Al 5083 alloy to study microstructure evolution. *Rev.Adv.Mater.Sci.* 35 (2013), pp.75-85.
18. Sabbaghianrad S., Langdon T.G. Microstructural saturation, hardness stability and superplasticity in ultrafine-grained metals processed by a combination of severe plastic deformation techniques. *Letters of materials* 5 (3), 2015, pp.335-340.
19. Vorhauer A., Kleber S., Pippan R. Influence of processing temperature on microstructure and mechanical properties of high-alloyed single-phase steels subjected to severe plastic deformation. *Mat.,Sci.,Eng.A* 410-411 (2005), pp.281-284.
20. Dobatkin S.V., Rybal'chenko O.V., Raab G.I. Structure formation, phase transformations and properties in Cr-Ni austenitic steel after equal-channel angular pressing and heating. *Mat.Sci.and Eng/A* 463 (2007) pp.41-45.
21. J.C.Pang, M.X.Yang, G.Yang, S.D.Wu, S.X.Li, Z.F.Zhang Tensile and fatigue properties of ultrafine-grained low-carbon steel processed by equal channel angular pressing. *Mat.Sci.Eng. A* 553 (2012), pp.157-163.
22. *Nanostructured metals and alloys. Processing, microstructure, mechanical properties and applications.* Edited by Sung H. Whang. Woodhead Publishing Limited, 2011.
23. Yu. Ivanisenko, W. Lojowski, R.Z. Valiev, H.-J. Fecht. The mechanism of formation of nanostructure and dissolution of cementite in a pearlitic steel during high pressure torsion. *Acta Mat.* 51 (2003), pp.5555-5570.
24. M.V. Karavaeva, S.K. Nurieva, N.G. Zaripov, A.V. Ganeev, R.Z. Valiev, Microstructure and mechanical properties of medium-carbon steel subjected to severe plastic deformation. *Metal Science and Heat Treatment*, 2012, pp.1-5.
25. Ganeev A.V., Karavaeva M.V., Sauvage X., Ivanisenko Yu., Valiev R.Z. The grain-boundary precipitates in ultrafine-grained carbon steels produced by HPT. In: XV Int.conf. on Intergranular and interphase boundaries in materials. Moscow, MISiS, 2016, p.28.
26. Kositsyna I.I., Sagaradze V.V. Phase transformations and mechanical properties of stainless steel in the nanostructural state. *Bulletin of the Russian Academy of Sciences: Physics*, 2007, Vol.71, No.2, pp.293-296.
27. Chen X.H., Lu J., Lu L., Lu K. Tensile properties of a nanocrystalline 316L austenitic stainless steel. *Scripta Materialia* 52 (2005), pp.1039-1044.
28. Greger M., Vodárek V., Dobrzański L.A., Kander L., Kocich R., Kuřetová B. The structure of austenitic steel AISI 316 after ECAP and low-cycle fatigue. *J. of Achievements in Materials and manufacturing Engineering*. V.28, issue 2, 2008.

29. Huang C.X., Yang G., Gao Y.L., Wu S.D., Zhang Z.F. Influence of processing temperature on the microstructures and tensile properties of 304L stainless steel by ECAP. *Mat.Sci. and Eng.A* 485 (2008), pp.643-650.
30. Kashyap B., Tangri K. On the Hall-Petch relationship and substructural evolution in type 316L stainless steel. *Acta Mater.* 1995, v. 43, pp.3971–3981.
31. Üçok İ., Ando T., Grant N. Property enhancement in type 316L stainless steel by spray forming. *Mater. Sci. Eng. A*, 1991, v.133, pp. 284–287.
32. Pakieła Z., Garbacz H., Lewandowska A., Suś-Ryszkowska M., Zieliński W, et al. Structure and properties of nanomaterials produced by severe plastic deformation. *Nukleonika* 2006, v.51, pp.19–25.
33. Wang H., Shuro I., Umemoto M., Kuo H.-H., Todaka Y. Annealing behavior of nano-crystalline austenitic SUS316L produced by HPT. *Mat., Sci., and Eng. A* 556 (2012), pp.906-910.



© 2016 by the authors; licensee *Preprints*, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons by Attribution (CC-BY) license (<http://creativecommons.org/licenses/by/4.0/>).