Structural strengthening of an austenitic stainless steel subjected to warm-to-hot working

Z. Yanushkevich, A. Mogucheva, M. Tikhonova, A. Belyakov *, R. Kaibyshev

Belgorod State University, Pobeda 85, Belgorod 308015, Russia

ABSTRACT

The effect of multiple rolling in the temperature interval of 500-1000 °C on the microstructure and the tensile behaviour of an austenitic stainless steel was studied. The structural changes during warm-to-hot working were characterized by the elongation of original grains towards the rolling axis and the development of new fine grains. The fraction of fine grains and the average grain size increased with increasing the rolling temperature. The multiple hot rolling resulted in significant strengthening. The offset yield strength approached 1030 MPa in the specimen processed at 500 °C, while a strength of 480 MPa was obtained after rolling at 1000 °C. The relationship between the deformation microstructures and the tensile behaviour is discussed.

Keywords:
Austenitic stainless steel
Warm rolling
Dynamic recrystallization
Strengthening
Tensile behaviour

1. Introduction

Austenitic stainless steels are one of most frequently used structural materials [1]. Such a widespread acceptance in various applications is associated with favourable combinations of their properties, i.e. ductility, toughness, formability, weldability, and corrosion resistance. However, engineering applications of Cr-Ni austenitic stainless steel are fairly limited by a relatively low yield strength of such materials [2]. Commonly, an increase in the strength of metals and alloys can be achieved by a structural strengthening, which include a grain size strengthening and a substructural (dislocation) strengthening. A remarkable grain refinement in stainless steels takes place during warm to hot working accompanied by the development of dynamic recrystallization [3–5].

The occurrence of dynamic recrystallization results in the formation of uniform microstructure with the final grain sizes being dependent on processing conditions; namely the dynamic grain size decreases with the decrease in deformation temperature and/or increase in strain rate. Processing at temperatures above about 0.7 of melting point is accompanied by the development of discontinuous dynamic recrystallization [3,4]. The new grains nucleate as a result of local bulging of grain boundaries; then, the recrystallization nuclei grow out consuming the work hardened surroundings. The sequential cycles of nucleation and growth lead to the dynamically stable average grain size that evolves after sufficiently large strains. Generally, the dynamically recrystallized microstructures consist of recrystallized grains with relatively low dislocation densities and work hardened grains having high density dislocations.

A decrease in deformation temperature slows down diffusion processes and, therefore, impedes the nucleation and growth of new grains by the discontinuous mechanism of dynamic recrystallization. Recent studies on dynamic recrystallization behaviour suggest the gradual change in the operating recrystallization mechanisms from discontinuous to continuous ones with a decrease of deformation temperature from about 0.7 to 0.5 of melting point [5,6]. Under conditions of warm working at about half of melting temperature, the new grains result from a kind of continuous reactions that are associated with progressive evolution of deformation subgrains. A spatial network of low-angle dislocation subboundaries develop at relatively small

---

* *Corresponding author.* Tel.: +7 4722 585457; fax: +7 4722 585417.
E-mail address: belyakov@bsu.edu.ru (A. Belyakov).

strains. Angular misorientations between subgrains gradually increase to typical values of high-angle grain boundaries upon further deformation, finally leading to the development of new grained microstructure at large strains. Since the new grains during continuous dynamic recrystallization result from the evolution of deformation substructure, they are characterized by rather high dislocation density in their interiors.

It should be noted that static restoration structural mechanisms commonly follow the dynamic recrystallization under conditions of conventional industrial processes involving multiple deformations. So-called metadynamic (or post-dynamic) recrystallization takes place during and after the hot working accompanied by the discontinuous dynamic recrystallization, while static recovery processes assist the development of continuously recrystallized microstructure in warm working [7,8]. The aim of this paper is to study the features of deformation microstructures developed in an 18%Cr-8%Ni-type austenitic stainless steel subjected to multiple rolling at temperatures of 500-1000 °C (0.44-0.73 of melting point) and the relationship between the developed microstructures and some mechanical properties. The total rolling strain of 2 was selected as one of the frequently used in metal forming processes.

2. Experimental

An austenitic stainless steel, Fe-0.1C-0.1Si-0.95Mn-0.01P-0.006S-18.4Cr-7.85Ni-0.5Nb-2.24Cu-0.12N-0.005B (wt. %), was hot forged at 1150-1200 °C and solution treated at 1100 °C. The rod specimens with the initial cross area of 20×20 mm^2 were heated in a muffle furnace to different temperatures ranging from 500 °C to 1000 °C and then rolled with a rolling rate of 7 m/min. The multiple rolling was carried out with a pass strain of 0.25 to a total cumulative strain of 2.0 followed by water quenching. The steel rods were reheated in about 5 minutes to the processing temperature after each 0.5 strain increment.

Structural investigations were performed on the sections parallel to the rolling axis, using a Quanta 600 FEG scanning electron microscope equipped with an electron back scattering diffraction pattern analyzer incorporating an orientation imaging microscopy system and a Jeol JEM-2100 transmission electron microscope (TEM). The orientation imaging microscopy micrographs were subjected to a cleanup procedure setting a minimal confidence index of 0.1. The grain/subgrain sizes were evaluated perpendicular to the rolling axis. The grain sizes were measured on the orientation imaging microscopy micrographs as an average distance between boundaries with misorientations above 15°. The transverse subgrain sizes were measured on the TEM micrographs by a linear intercept method, counting all clear defined (sub) boundaries. Total of about 200 grains and 100 subgrains were counted for each data point. To evaluate the size of dispersed precipitates in the initial state, 23 individual particles were measured. The dislocation densities were estimated by counting individual dislocations in (sub)grain interiors on at least six arbitrary selected typical TEM images for each specimen. The mechanical properties of processed samples were evaluated by means of tensile tests at an ambient temperature by using flat specimens with a gauge length of 12 mm and cross section of 3.0×1.5 mm^2. Three tensile specimens were used for each processing condition.

3. Results and Discussion

3.1. *Deformation Microstructures*

The initial microstructure consisted of austenitic grains containing homogeneous distribution of dispersed particles. The grain and particle sizes were about 7 μm and 50 nm, respectively. Typical microstructures that develop after bar rolling at different temperatures are shown in Fig. 1. The bar rolling leads to the elongation of original grains along the rolling axis and the development of new fine grains, the size and volume fraction of which depend on processing temperature. The fine grains are also somewhat elongated towards the metal flow direction. The average spacing between high-angle grain boundaries in the transverse section of the rolled bars increases from about 0.4 to 1 μm with increasing the rolling temperature. It should be noted that the reduction in the thickness of original grains due to a rolling strain of 2 should result in the boundary spacing of 2.7 μm. Therefore, the grain refinement is apparent under the all studied conditions.

The microstructure of a lamella-type with a few of submicron size grains located at longitudinal grain boundaries develops during the rolling at relatively low temperatures of 500-700 °C (Fig. 1a). The number of fine grains increases remarkably by processing at 800 °C (Fig. 1b). This deformation structure consists of highly elongated grains, which are interleaved with chains of the fine grains. It is clearly seen in Fig. 1b that the new grains develop at frequently corrugated original grain boundaries. At a higher temperature of 900 °C, the evolved microstructure is strongly affected by the strain localization in deformation microbands (Fig. 1c). The latter ones are set at about 20 degrees to the rolling axis. The new grains appear in the vicinities of original grain boundaries and, especially, at the triple junctions as well as at the deformation microbands. The most uniform and equiaxed grain structure develops during the rolling at 1000 °C (Fig. 1d). It should also be noted that the bar rolling does not lead to any specific texture development. The fiber textures of <111> and <001>, which are inherent in face centered cubic metals, are alternated with various orientations including <011> in Fig. 1.

The deformation substructures in the rolled steel bars are characterized by the evolution of high density dislocations and the development of sharp subboundaries subdividing original grains (Fig. 2). Majority of the well developed subboundaries are arranged along the rolling axis. Similar to the grain boundary spacing, the transverse subgrain size increases from about 100 to 200 nm, when the deformation temperature rises from 500 °C to 1000 °C. In spite of elevated processing temperature, the deformation substructures involve high dislocation densities within subgrains. The dislocation densities comprise about 20×10^{14} and 7×10^{14} m^{-2} in the steel subjected to multiple rolling at 500 °C and 1000 °C, respectively. The main structural parameters indicated with 95% confidence intervals for the steel rods processed at different temperatures are collected in Table 1.

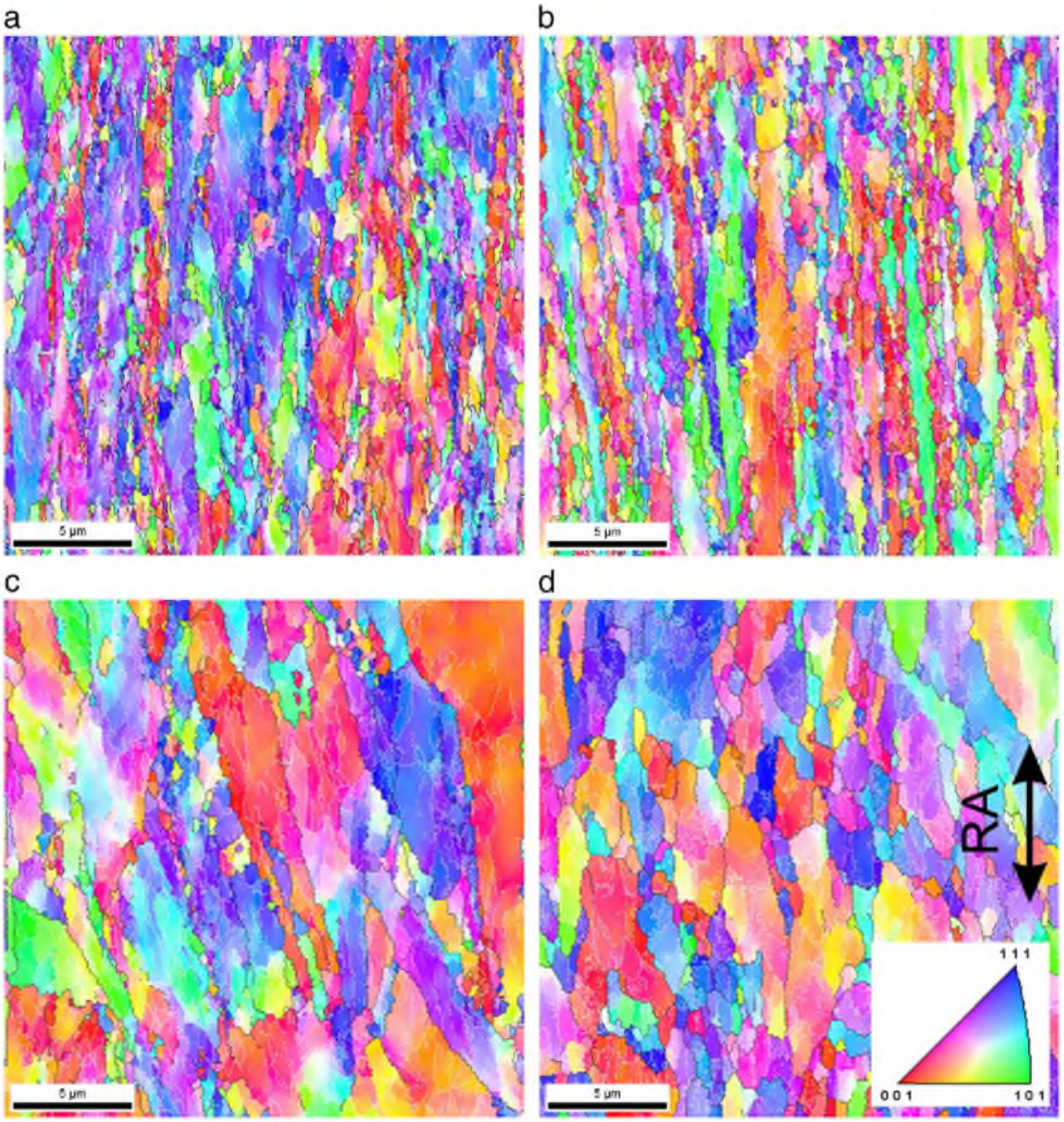

Fig. 1 – Orientation imaging microscopy micrographs of an 18%Cr-8%Ni-type stainless steel processed by multiple rolling at 600 °C (a), 800 °C (b), 900 °C (c), and 1000 °C (d). The inverse pole figure is shown for the rolling axis (RA). The black and white lines indicate high- and low-angle boundaries, respectively.

Somewhat elongated shape of the fine grains/subgrains along with the high dislocation density testify that the deformation microstructures are evolved during the warm-to-hot rolling, while static recrystallization hardly took place after the final rolling pass and did not affect the developed microstructures. Generally, the both discontinuous and continuous mechanisms of dynamic recrystallization concurrently operate under the studied conditions. The general features of the developed microstructures suggest that the discontinuous recrystallization assisted by a partial metadynamic recrystallization is the main structural mechanism responsible for the new grain formation at 1000 °C that leads to the uniform microstructure. The discontinuous recrystallization mechanism consists of nucleation of recrystallizing grains by local boundary migration followed by enlargement of newly formed grains that grow out consuming work hardened surroundings. The new grain evolution can be assisted by the metadynamic recrystallization that should operate during the interpass holding time. The high dislocation density, however, indicates that the contribution of metadynamic recrystallization in the new grain development is negligible after the final rolling pass.

On the other hand, a decrease of the deformation temperature retards the kinetics of discontinuous recrystallization. The incomplete discontinuous recrystallization results in the development of the necklace microstructure consisting of the chains of new fine grains along original grain boundaries [9]. The grain refinement at low temperatures below 800 °C should be assisted by the continuous recrystallization mechanism, the contribution of which into dynamic grain development increases with decreasing the deformation temperature. Continuously recrystallized grains result from a progressive evolution of subgrains into the new grains due to the development of strain-induced subboundaries, angular misorientation of which gradually

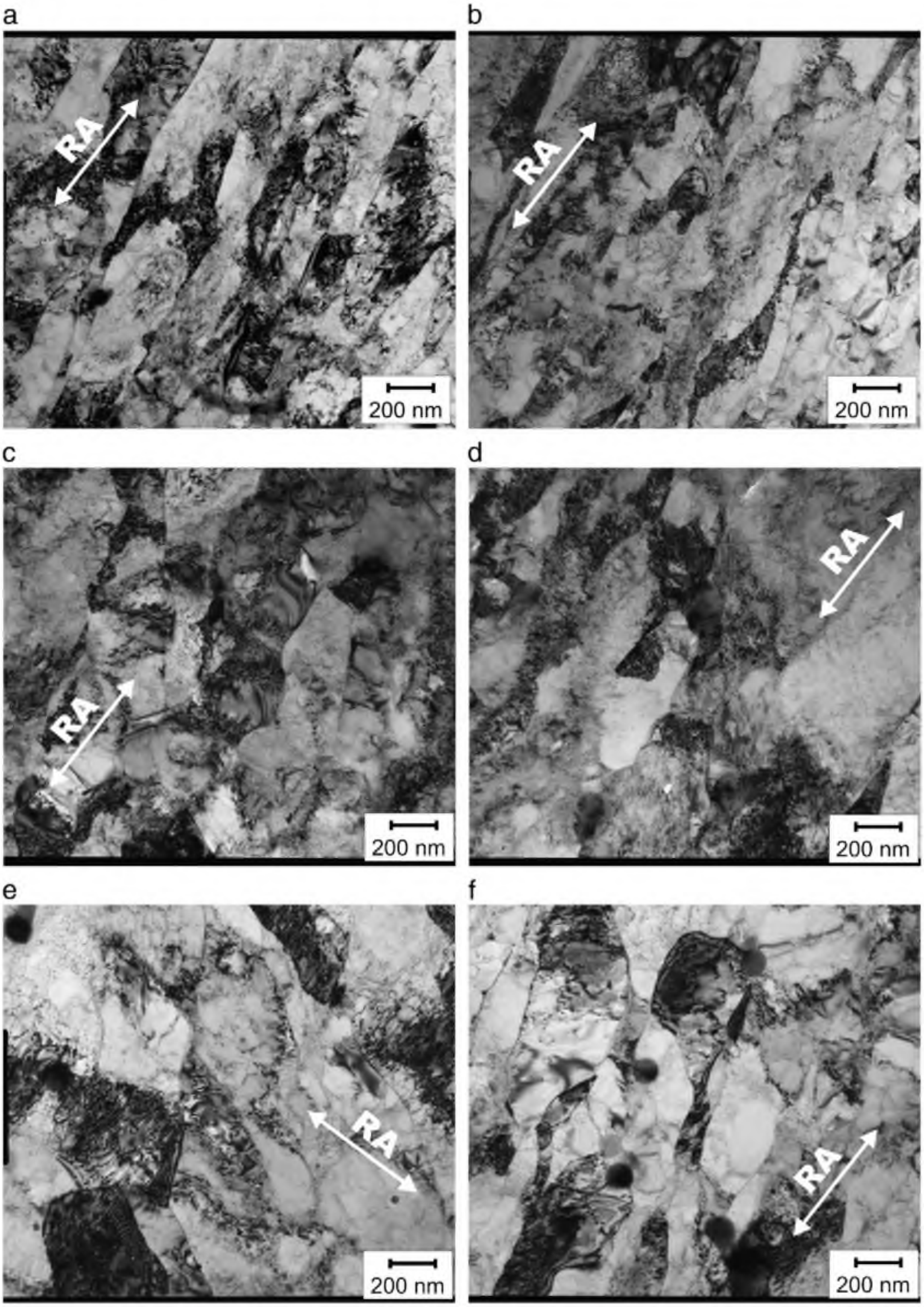

Fig. 2 – TEM micrographs of an 18%Cr-8%Ni-type stainless steel processed by multiple rolling at 500 °C (a), 600 °C (b), 700 °C (c), 800 °C (d), 900 °C (e), and 1000 °C (f). The RA indicates the direction of rolling axis.

increases with straining. The deformation subboundaries that evolved near the original grain boundaries have been reported to have higher misorientations compared to those in the grain interiors and could rapidly transform to high-angle boundaries [10,11]. Therefore, the new fine grains developed during warm rolling at relatively low temperatures of 500-700 °C can be observed at original grain boundaries. The change in the operative recrystallization mechanisms revealed in the present

Table 1 – The grain and subgrain sizes, and the dislocation density in an 18%Cr-8%Ni-type stainless steel subjected to multiple rolling at temperatures of 500 to 1000 °C.

Temperature, °C	Grain size, nm	Subgrain size, nm	Dislocation density, $\times 10^{14}$ m^{-2}
500	420±60	115±10	20.2±5.7
600	390±55	120±10	19.6±2.3
700	470±65	150±10	14.5±4.1
800	430±60	165±25	12.6±3.1
900	850±120	170±10	11.4±2.0
1000	950±130	195±30	6.6±1.5

study at temperatures of 600-800 °C is in consistence with previous studies on dynamic recrystallization [4,6,12].

3.2. *Tensile Behaviour*

A representative series of stress-strain curves obtained by tensile tests at an ambient temperature is shown in Fig. 3. Decrease in the rolling temperature results in significant strengthening of the steel. The mean offset yield strength of 1030 MPa in specimens after rolling at 500 °C is more than twice as high as that for specimens processed at 1000 °C. The strengthening by warm rolling is accompanied by a degradation of plasticity. The uniform elongation gradually decreases from about 17% to 7% with decreasing the rolling temperature from 1000 °C to 800 °C and then drops to almost zero in the specimens processed at temperatures below 700 °C. The specimens rolled at 500-600 °C are characterized by a negligible short stage of strain hardening after the onset of plastic flow during the tensile test that makes the ultimate tensile strength almost the same with the yield strength. The changes in mechanical behaviour correlate with the variations of recrystallization mechanisms. The high dislocation densities in the specimens processed at relatively low temperatures restrict further strain hardening and impair plasticity.

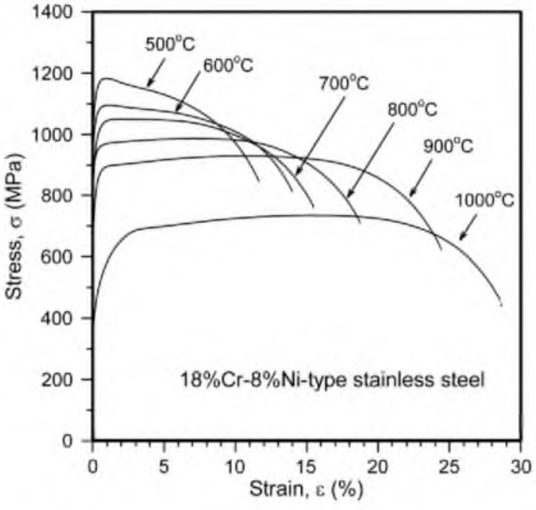

Fig. 3 – Tensile behaviour of an 18%Cr-8%Ni-type stainless steel subjected to multiple rolling at temperatures of 500 to 1000 °C.

Generally, offset yield strength ($\sigma_{0.2}$) can be related to the grain size (D) through the Hall-Petch relationship [13], which is valid for various recrystallized metals and alloys. However, the original Hall-Petch relation does not count any substructural strengthening, which can be significant in the studied steel samples. It is clearly seen in Fig. 4 that a unique power law relationship between subgrain size (d) and dislocation density (ρ), i.e. $d \sim \rho^{-0.5}$ [14,15], is generally held for the studied steel subjected to warm-to-hot rolling at various temperatures, in spite of different operative mechanisms for microstructure evolution. Therefore, the substructural strengthening can be estimated through either subgrain size or dislocation density. The strengthening increment ($\Delta\sigma$) due to increased dislocation density (ρ) can be expressed as follows [16]:

$$\Delta\sigma = \alpha G\, b\, \rho^{0.5} \tag{1}$$

Here, G and b are shear modulus and Burgers vector, respectively, and the factor of α falls in the range 0.05 to 1. Therefore, the modified expression for the offset yield strength includes three terms:

$$\sigma^{*}_{0.2} = \sigma_0 + \alpha G\, b\, \rho^{0.5} + K\, D^{-0.5} \tag{2}$$

The best fit of experimental results is obtained when $\sigma_0 = 160$ MPa, $\alpha = 0.7$, and $K = 0.12$ MPa m$^{0.5}$. Note here that the value of $\sigma_0 = 160$ MPa is close to that of 200 MPa obtained by Young and Sherby [17] for stainless steels. The strength properties of solution treated and processed steel samples are represented in Table 2 along with the values of offset yield strength calculated by Eq. (2).

The steel strengthening by multiple rolling in the temperature range of 800-1000 °C is mainly affected by a decrease in the average grain size. Remarkable contribution of discontinuous recrystallization under hot deformation conditions improves the plasticity of the hot rolled samples. In contrast, the rise of yield strength with the decrease in the rolling temperature from 800 to 700 °C is attributed to the substructural strengthening due

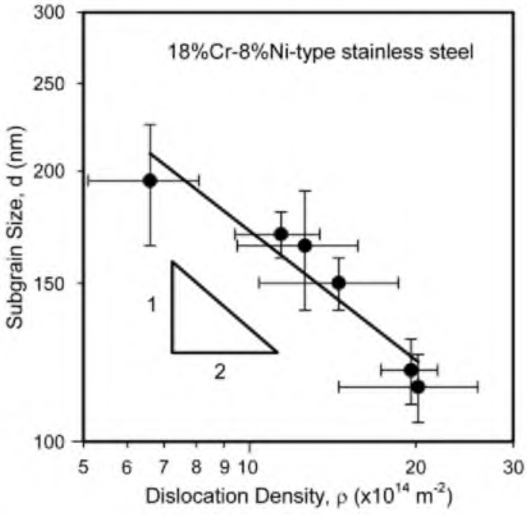

Fig. 4 – Relationship between the subgrain size and the dislocation density evolved in an 18%Cr-8%Ni-type stainless steel subjected to multiple rolling at temperatures of 500 to 1000 °C.

Table 2 – The offset yield strength and the ultimate tensile strength of an 18%Cr-8%Ni-type stainless steel subjected to multiple rolling at temperatures of 500 to 1000 °C.

Temperature, °C	Offset yield strength, MPa	Ultimate tensile strength, MPa	Offset yield strength calculated by Eq. (2), MPa
Initial	330±20	670±10	251
500	1030±75	1130±25	1002
600	1000±25	1070±15	1000
700	955±20	1055±10	893
800	860±40	970±10	862
900	780±30	920±10	784
1000	480±80	795±50	659

to high dislocation density. The grain refinement accompanied by the evolution of high density dislocations under warm working conditions may provide a remarkable strengthening, although plasticity is deteriorated.

4. Conclusions

The deformation microstructures and their effect on the tensile properties were studied in an 18%Cr-8%Ni-type stainless steel subjected to multiple rolling at 500-1000 °C. The main results can be summarized as follows.

1. The multiple warm-to-hot rolling was accompanied by concurrent development of discontinuous and continuous dynamic recrystallization. The discontinuous recrystallization was the main mechanism responsible for microstructure evolution at 1000 °C. The contribution of discontinuous recrystallization to the new grain development gradually decreased, while that of continuous recrystallization increased with a decrease of processing temperature. At 500-700 °C the new grain boundaries were developed as a result of continuous strain-induced reactions.
2. The multiple rolling resulted in significant strengthening of processed steel; and the strength increment built up with decrease in processing temperature. The offset yield strength increased from 480 MPa to 1030 MPa when the rolling temperature was changed from 1000 °C to 500 °C.
3. Both the grain size and substructural strengthening contributed to the mechanical properties. The strength of processed steel can be roughly evaluated by a modified Hall-Petch relation taking into account the strength increment caused by high density dislocations.

Acknowledgements

This study was supported by Ministry of Education and Science, Russia, under Grant No. 14.740.11.0333. The authors are grateful to the staff of the Joint Research Centre, Belgorod State University, for their assistance with instrumental analysis.

REFERENCES

[1] Lo KH, Shek CH, Lai JKL. Recent developments in stainless steel. Mater Sci Eng, R 2009;65:39–104.
[2] Martienssen W, Warlimont H. Springer Handbook of Condensed Matter and Materials Data. Berlin: Springer; 2005.
[3] McQueen HJ, Jonas JJ. Recovery and recrystallization during high temperature deformation. In: Arsenault RJ, editor. Treatise on Materials Science and Technology, Vol. 6. New York: Academic Press; 1975. p. 394–490.
[4] Belyakov A, Sakai T, Miura H, Kaibyshev R. Grain refinement under multiple warm deformation in 304 type austenitic stainless steel. Iron Steel Inst Jpn Int 1999;39:592–9.
[5] Belyakov A. Changes in the grain structure of metallic materials upon plastic treatment. Phys Met Metall 2009;108: 390–400.
[6] Dudova N, Belyakov A, Sakai T, Kaibyshev R. Dynamic recrystallization mechanisms operating in a Ni-20%Cr alloy under hot-to-warm working. Acta Mater 2010;58:3624–32.
[7] Dehghan-Manshadi A, Barnett MR, Hodgson PD. Recrystallization in AISI 304 austenitic stainless steel during and after hot deformation. Mater Sci Eng, A 2008;485:664–72.
[8] Beladi H, Cizek P, Hodgson PD. The mechanism of metadynamic softening in austenite after complete dynamic recrystallization. Scr Mater 2010;62:191–4.
[9] Jafari M, Najafizadeh A. Correlation between Zener-Hollomon parameter and necklace DRX during hot deformation of 316 stainless steel. Mater Sci Eng, A 2009;501:16–25.
[10] Belyakov A, Gao W, Miura H, Sakai T. Strain induced grain evolution in polycrystalline copper during warm deformation. Metall Mater Trans A 1998;29A:2957–65.
[11] Salishchev G, Mironov S, Zherebtsov S, Belyakov A. Changes in misorientations of grain boundaries in titanium during deformation. Mater Charact 2010;61:732–9.
[12] Tan S, Wang Z, Cheng S, Liu Z, Han J, Fu W. Processing maps and hot workability of super304H austenitic heat-resistant stainless steel. Mater Sci Eng, A 2009;517:312–5.
[13] Hall EO. The deformation and ageing of mild steel: III Discussion of results. Proc R Soc London, Ser B 1951;64:747–53.
[14] Takeuchi S, Argon AS. Steady-state creep of single-phase crystalline matter at high temperature. J Mater Sci 1976;11: 1542–66.
[15] Castro-Fernandez FR, Sellars CM, Whiteman JA. Changes of flow stress and microstructure during hot deformation of Al-1Mg-1Mn. Mater Sci Technol 1990;6:453–60.
[16] Hull D, Bacon DJ. Introduction to Dislocations. Oxford: Butterworth-Heinemann; 1984.
[17] Young CM, Sherby OD. Subgrain formation and subgrain-boundary strengthening in iron-based materials. J Iron Steel Inst 1973;211:640–7.