Materialia 23 (2022) 101460

Contents lists available at ScienceDirect

Materialia

journal homepage: www.elsevier.com/locate/mtla

Cryogenic impact toughness of a work hardened austenitic stainless steel

M.V. Odnobokova [a], A. Belyakov [b,*], N.A. Enikeev [a], R.O. Kaibyshev [c], R.Z. Valiev [a]

[a] Institute for Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa 450008, Russia
[b] Belgorod State University, Belgorod 308015, Russia
[c] Russian State Agrarian University - Moscow Timiryazev Agricultural Academy, Moscow 127550, Russia

ARTICLE INFO

Keywords:
Rolling
Stainless steels
Martensitic phase transformation
Impact behavior
Ductile fracture

ABSTRACT

Warm rolling at 473 K with total reductions up to 65% significantly strengthened a 304 L austenitic stainless steel. The tensile strength at room temperature increased from 600 MPa to 1040 MPa. Outstanding mechanical properties of the warm rolled steel were observed at 77 K, when the ultimate tensile strength of 1830 MPa and a fracture elongation of 39% were achieved. Moreover, the warm rolled steel exhibited high impact toughness, especially, at cryogenic temperature that was attributed to the crack branching crosswise to the principal crack direction.

Currently, there is a need for structural materials that can be used in cryogenic applications. In addition to strength and ductility, such materials must have sufficient toughness at cryogenic temperatures. Austenitic steels are an attractive material for cryogenic applications because they are commonly characterized by high impact toughness, excellent plasticity and good corrosion resistance [1]. Moreover, austenitic stainless steels are very susceptible to strain hardening. Therefore, desired level of strength in such materials can be easily obtained by cold to warm working. On the other hand impact toughness and plasticity of the work hardened steels may significantly degrade at very low temperatures [2,3]. The deformation and impact behavior at cryogenic temperatures of austenitic stainless steels subjected to cold to warm working have not been studied in sufficient detail. An outstanding toughness and plasticity of austenitic steels with low stacking fault energy (SFE) at room temperature is commonly attributed to the twinning-induced plasticity (TWIP) and transformation-induced plasticity (TRIP) effects [3]. The enhanced strength-ductility properties of a 304 L stainless steel at liquid nitrogen temperature have been attributed to pronounced Luders-type deformation leading to local dislocation accumulation and strengthening [4]. However, the multiple deformation twinning may lead to twinning-assisted cracking and, hence, impair plasticity at low temperatures [5]. Regarding the TRIP effect on plasticity and toughness of low-SFE metastable austenitic steels at cryogenic temperatures, this interesting phenomenon deserves further clarification. Commonly, the strain-induced martensitic transformation and corresponding strengthening are promoted by a decrease in deformation temperature [6,7]. The grain refinement has been discussed to increase the austenite stability against the strain-induced martensitic transformation, although opposite effect has been reportedly occurred in the ultrafine grained range [6]. The deformation-induced martensitic transformation has been experimentally shown to decrease the low-temperature toughness [8]. In contrast, studying by means of theoretical modeling [9] or model materials [10], a positive effect of the martensitic transformation on the impact behavior owing to expanding the plastic zone ahead of the crack has been suggested. Such debates are consequence of a lack of adequate experimental results. The present study is, therefore, aimed to experimentally clarify the work-hardening effect on the tensile behavior and the impact toughness of an austenitic stainless steel at 77 K.

A 304 L austenitic stainless steel with the chemical composition of Fe-0.05%C-18.2%Cr-8.8%Ni-1.65%Mn-0.43%Si-0.05%P-0.04%S (all in wt%) was investigated. The ingot was forged at 1373 K to square bar of 30×30 mm^2 cross section. The forged samples were subjected to plate rolling at 473 K to reduction in thickness of 40% or 65%. The tensile specimens with a cross-section of 3×1.5 mm^2 and a gauge length of 12 mm were tensioned at 293 K and 77 K using an Instron 5882 testing machine. The impact toughness was evaluated on standard Charpy V-notch specimens using an Instron 450 J impact machine with an Instron Dynatup Impulse data acquisition system at 293 K and 77 K. The impact and tensile directions were parallel to the normal (ND) and rolling (RD) directions, respectively. The microstructures were observed using Quanta 600 FEG scanning electron microscope (SEM) by means of the electron backscatter diffraction (EBSD) method close to the fracture surface. The fractured surfaces of the Charpy impact specimens were observed using Nova Nanosem 450 SEM. Besides EBSD, the fraction of strain-induced martensite was measured by magnetic induction method with Feritscope FMP30.

Hot forging resulted in the uniform microstructure with an average grain size of 24 μm. The microstructure evolution during rolling at a

* Corresponding author.
E-mail address: belyakov@bsu.edu.ru (A. Belyakov).

https://doi.org/10.1016/j.mtla.2022.101460
Received 22 March 2022; Accepted 15 May 2022
Available online 16 May 2022

Table 1
The impact toughness (KCV), the yield strength ($\sigma_{0.2}$), the ultimate tensile strength (UTS), total elongation (δ) and the martensite fraction (F_M) of a 304 L steel in initial state and subjected to rolling at 473 K to different reductions and tested at the indicated temperatures.

	Initial state		Rolling reduction 40%		Rolling reduction 65%	
	T_{test} = 293 K	T_{test} = 77 K	T_{test} = 293 K	T_{test} = 77 K	T_{test} = 293 K	T_{test} = 77 K
KCV (J/cm^2)	380	240	210	195	125	130
$\sigma_{0.2}$ (MPa)	220	385	770	800	970	1070
UTS (MPa)	600	1540	800	1680	1040	1830
δ (%)	100	53	35	46	12	39
F_M in Charpy specimens (near fracture)	undefined	0.67	0.06	0.57	0.12	0.73
F_M in tensile specimens (neck zone)	0.19	0.36	0.25	0.45	0.28	0.47

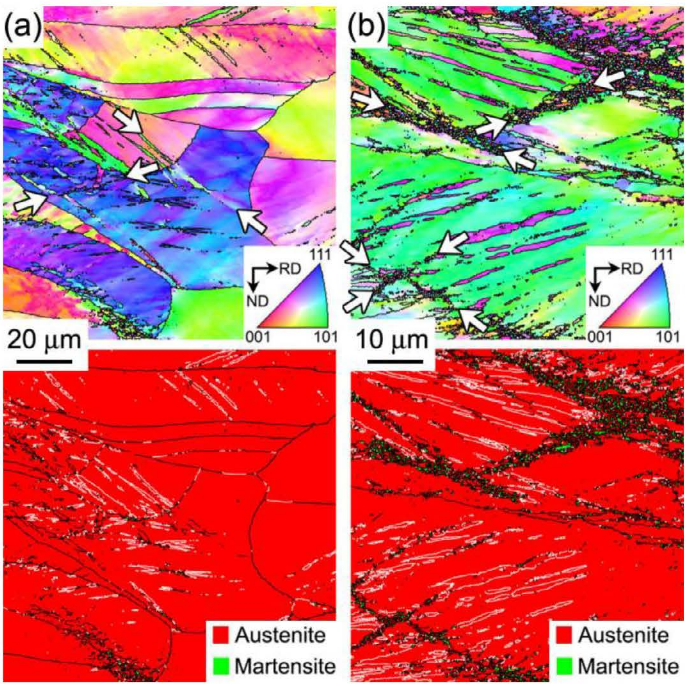

Fig. 1. Microstructures (upper images) and phase distribution (down images) in a 304 L steel subjected to warm rolling at 473 K with 40% (a) and 65% (b) of rolling reduction. The black and white lines indicate ordinary high-angle boundaries and Σ3 CSL boundaries, respectively. The inverse pole figures are shown for the normal direction (ND).

temperature of 473 K was detailed elsewhere [11]. It can be stressed here that the warm worked microstructures were characterized by numerous deformation twins and microshear bands running at acute angles to the rolling plane (Fig. 1). The spacing of high-angle grain boundaries substantially decreased with rolling reduction owing to increasing the number density of microshear bands (indicated by white arrows in Fig. 1). The mean transverse/longitudinal grain sizes were 1.4/3.0 μm and 0.5/1.0 μm after rolling reductions of 40% and 65%, respectively [11]. The martensitic transformation hardly developed in the steel during rolling at 473 K to total reduction of 65%; the martensite fraction did not exceed 3% [11]. The fine martensite crystallites occurred only in microshear bands, which contained high dislocation density.

The steel samples are characterized by outstanding strain hardening upon tensile tests in the initial hot forged conditions (Fig. 2a). A gradual decrease in the strain hardening during the tensile test at 293 K results in pronounced strengthening from 220 MPa (the yield strength, $\sigma_{0.2}$) to 600 MPa (the ultimate tensile strength, UTS) over a large uniform elongation of approx. 100% (Table 1). On the other hand, following the yield at 385 MPa, a sharp increase in the strain hardening in the range of 10–15% elongation leads to almost threefold increase in the stress during the tensile test at 77 K, although a uniform elongation decreases to about 50%. Nevertheless, pronounced strain hardening at 10–30% elongation results in UTS of 1540 MPa. Such behavior has been attributed to a rapid strain-induced martensitic transformation at lowered temperatures [4,6]. In contrast, the warm rolled steel samples do not possess any remarkable strain hardening upon tensile tests at 293 K (Fig. 2b and c); thus, $\sigma_{0.2}$ and UTS are quite close to each other (Table 1). An increase in the warm rolling strain to 65% increases the tensile strength and decreases plasticity, especially, uniform elongation, which drops down to few percent at 293 K. It is interesting that the warm rolled steel samples exhibit exceptional strain hardening in the range of 15–20% elongation upon tensile tests at 77 K that improves both the strength and plasticity (Fig. 2b and c). UTS increases from 800 MPa to 1680 MPa and from 1040 MPa to 1830 MPa in the samples after 40% and 65% rolling reductions, respectively, with a decrease in test temperature from 293 K to 77 K; corresponding total elongation (δ) increases from 35% to 46% and from 12% to 39% (Table 1). As a result, the product of UTS × δ of about 80 GPa% in the initial state remains above 70 GPa% after warm rolling. Such pronounced flexure on the stress-elongation curves at 77 K was observed in steels exhibiting TWIP/TRIP effect [12]. The fraction of strain-induced martensite in the specimens after tensile tests at 77 K is well above 0.4 (Table 1). The microstructural mechanisms providing this strength-ductility enhancement deserve more comprehensive investigation.

The load - displacement curves obtained during the impact tests are shown in Fig. 2d and e, and the impact toughness is listed in Table 1. The load gradually increases to its maximum (P_m) followed by slow decrease till complete fracture at a large displacement during the impact test at 293 K of initial hot forged steel, leading to high impact toughness of 380 J/cm^2. It is worth noting that the displacement corresponding to P_m does not depend on test temperature. Significant strengthening of hot forged steel is observed during impact test at 77 K, when the maximal load is almost twice as the general yield, although the impact toughness decreases to 240 J/cm^2 due to rapid fracture at relatively small displacements. In contrast, almost the same values of impact toughness are recorded in the warm rolled steels during impact tests at 293 K and 77 K. P_m is attained at displacements of about 3.5 mm and 2.5 mm during impact tests of steel subjected to 40% and 65% rolling reduction, respectively, resulting in lower impact toughness of larger rolled steel. The same impact toughness at 293 K and 77 K of the warm rolled steel samples is associated with a specific flexure on the load-displacement curves after P_m during the tests at 77 K. This flexure suggests an apparent crack arrest behavior that expands the region of stable crack propagation, increasing the impact toughness. The stress at the notch root can be expressed as follows [13].

$$\sigma = \beta LP(2CB)^{-1}W^{-2} \quad (1)$$

where $\beta = 2$ for the Tresca criterion, L is the span of 40 mm, P is the load, C is constraint factor of 1.363 for ASTM tip, B is the specimen thickness of 10 mm and W of 8 mm is the rest of the specimen width ahead of the notch. Assuming that maximal stresses at front of crack during the crack propagation are constant and can be roughly evaluated by Eq. (1), the

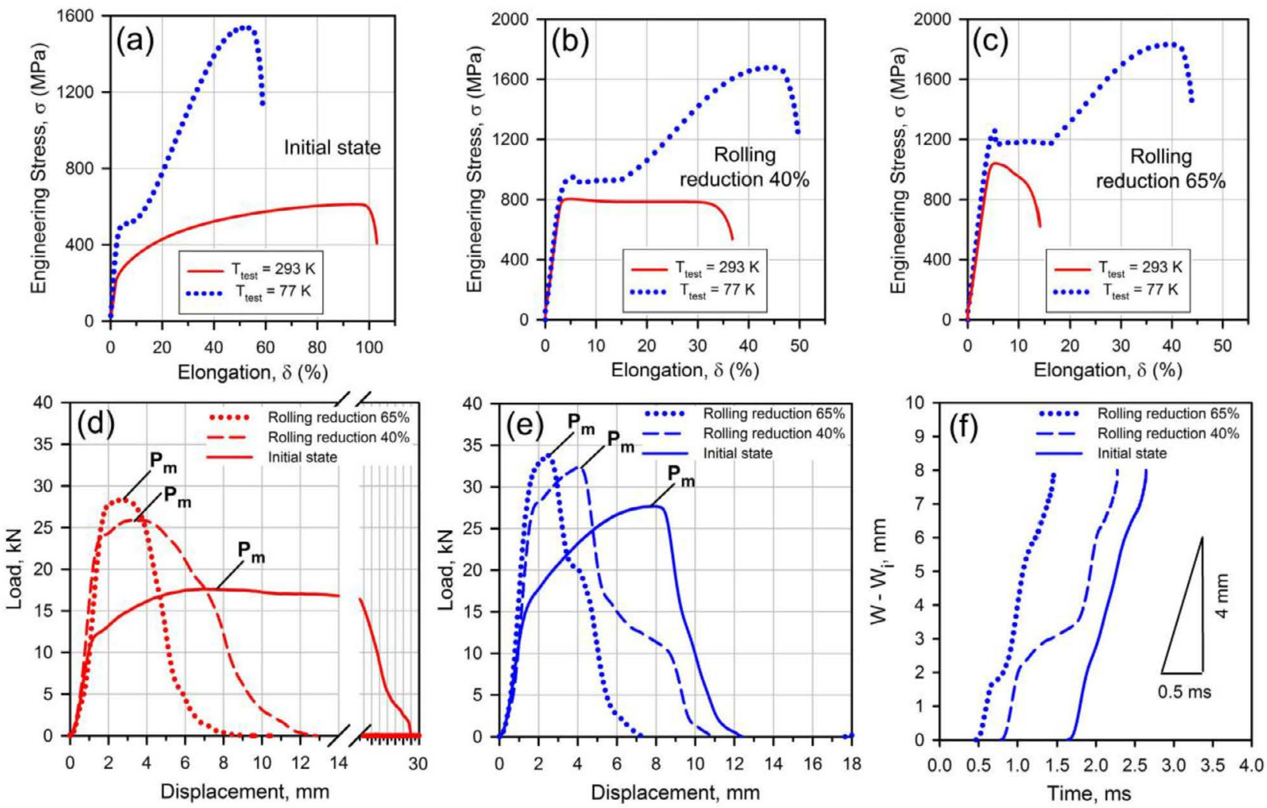

Fig. 2. Tensile stress - elongation curves (a, b, c), impact load - displacement curves at 293 K (d) and 77 K (e), and crack propagation during impact tests at 77 K (f) for a 304 L steel.

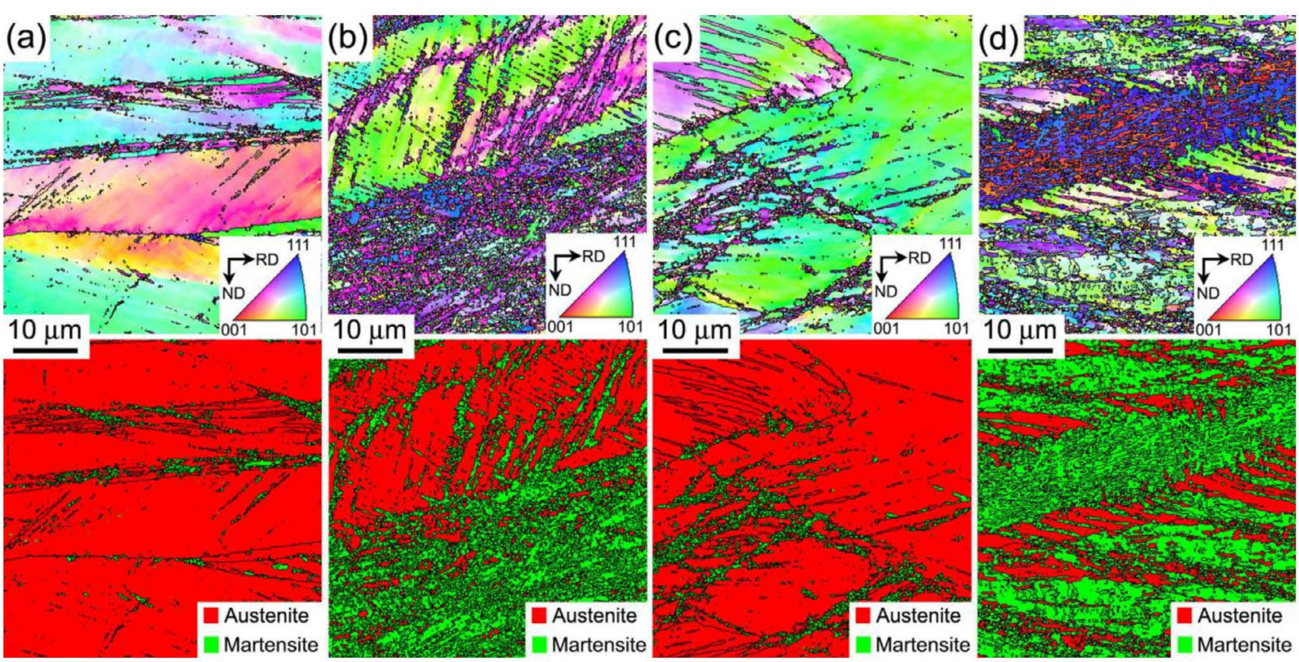

Fig. 3. Microstructures (upper images) and phase distributions (down images) in the Charpy specimens of a 304 L steel subjected to rolling at 473 K to reductions of 40% (a, b) or 65% (c, d), and tested at 293 K (a, c) or 77 K (b, d). High-angle boundaries are indicated by black lines. The inverse pole figures are shown for the normal direction (ND).

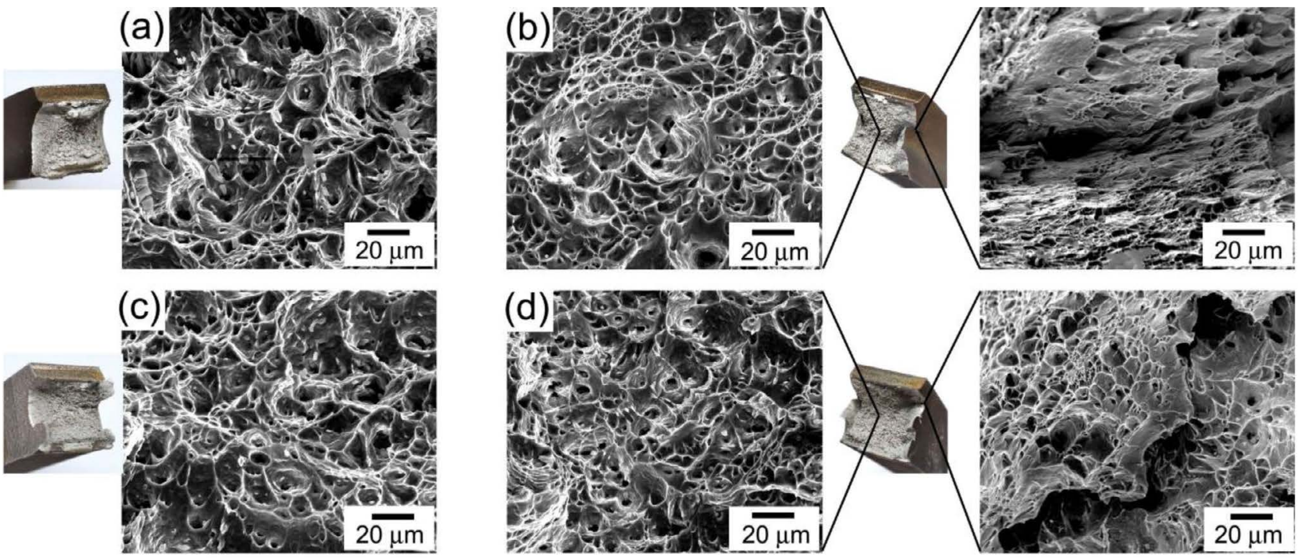

Fig. 4. Ductile fracture surfaces in the Charpy V-notch specimens of a 304 L steel subjected to rolling at 473 K to reductions of 40% (a, b) or 65% (c, d), and tested at 293 K (a, c) or 77 K (b, d).

rest of the specimen thickness ahead of crack during the impact tests can be estimated as follows.

$$W_i = (\beta L P_i)^{0.5}(2\sigma CB)^{-0.5} \quad (2)$$

where P_i is the current load behind P_m. The distance of crack propagation along the specimen thickness, i.e., $W - W_i$, is represented in Fig. 2f for the impact tests at 77 K. Almost the same rate of crack propagation of about 8 m/s, except for the flexures associated with the crack arrest in Fig. 2f, is indicative of the same fracture mechanisms in the specimens tested at 77 K irrespective of previous work hardening.

The microstructures that evolve near the fracture surface of the Charpy specimens are shown in Fig. 3. The microstructural changes are characterized by the development of deformation microbands, number of which remarkably increases with a decrease in test temperature. The strain-induced martensite readily develops along the deformation microbands at 77 K. The fraction of strain-induced martensite is quite small in the specimens tested at 293 K, although warm rolling promotes the transformation (Table 1). On the other hand, the martensite fraction is well above 0.5 in the specimens tested at 77 K (Table 1). The fracture surfaces after impact tests are shown in Fig. 4. All fractures are characterized by fine-dimpled surfaces, i.e., the fracture occurs in a ductile manner. The small cracks running along the impact specimens are clearly seen in the warm rolled samples after tests at 77 K (right sides in Fig. 4b and d). These cracks are observed at a quarter of the specimen thickness from the notch and are evidently responsible for the crack arrest and the corresponding flexure at a level of approx. half of P_m on the load - displacement curves in Fig. 2e. The similar fracture behavior, namely, delamination along the impact specimens that improves the impact toughness, was observed in carbon steels subjected to tempforming [14]. However, in contrast to tempforming, where delamination was assisted by brittle fracture [15], the present samples exhibit ductile fracture irrespective of the direction of crack propagation. The crack arrest associated with the crack branching crosswise to the principal crack propagation in the warm rolled samples can be attributed to the microshear bands (Fig. 1) that contain high density of both dislocations and grain/subgrain boundaries as well as fine martensite crystallites favoring the micro-crack/void formation and crack propagation at low temperatures [8,16,17].

In conclusion, warm rolling significantly increases the yield strength at expense of remarkable degradation of plasticity and impact toughness of a 304 L stainless steel at room temperature. On the other hand, the work hardening by warm rolling is not accompanied by a decrease in plasticity and impact toughness at cryogenic temperature. The value of impact toughness at 77 K remains at a level of KCV values at 293 K. This improvement of the impact toughness at low temperature is associated with the crack branching, when the deviation of the main crack propagation from the impact direction may reach 90°, i.e., crack arrest behavior owing to specific pan-caked grain microstructure with microshear bands involving the ultrafine austenite and martensite crystallites with high dislocation densities in the warm rolled samples.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The study was funded by RFBR, project number 19–38–60047.

References

[1] K.H. Lo, C.H. Shek, J.K.L. Lai, Resent developments in stainless steels, Mater. Sci. Eng. R 65 (2009) 39–104.
[2] H. Ding, Y. Wu, Q. Lu, P. Xu, J. Zheng, L. Wei, Tensile properties and impact toughness of S30408 stainless steel and its welded joints at cryogenic temperatures, Cryogenics 92 (2018) 50–59 (Guildf).
[3] S.S. Sohn, S. Hong, J. Lee, B.C. Suh, S.K. Kim, B.J. Lee, N.J. Kim, S. Lee, Effects of Mn and Al contents on cryogenic-temperature tensile and Charpy impact properties in four austenitic high-Mn steels, Acta Mater. 100 (2015) 39–52.
[4] J. Li, Z. Zhou, S. Wang, Q. Mao, C. Fang, Y. Li, G. Wang, J. Kang, X. Zhu, Deformation mechanisms and enhanced mechanical properties of 304L stainless steel at liquid nitrogen temperature, Mater. Sci. Eng. A 798 (2020) 140133.
[5] T. Ye, F. Zhao, L. Chen, K. Jiang, Q. Deng, Y. Chen, Q. Wang, T. Suo, Effect of strain rate and low temperature on mechanical behaviour and microstructure evolution in twinning-induced plasticity steel, Mater. Sci. Eng. A 823 (2021) 141734.
[6] M.J. Sohrabi, M. Naghizadeh, H. Mirzadeh, Deformation-induced martensite in austenitic stainless steels: A review, Arch. Civ. Mech. Eng. 20 (2020) 124.
[7] M. Soleimani, A. Kalhor, H. Mirzadeh, Transformation-induced plasticity (TRIP) in advanced steels: A review, Mater. Sci. Eng. A 795 (2020) 140023.
[8] B. Hwang, T.H. Lee, S.J. Park, C.S. Oh, S.J. Kim, Correlation of austenite stability and ductile-to-brittle transition behavior of high-nitrogen 18Cr-10Mn austenitic steels, Mater. Sci. Eng. A 528 (2011) 7257–7266.
[9] H. Murakawa, H. Serizawa, T. Tsujimoto, S. Tomiyama, Mechanism and effect of stress-induced transformation on improvement of fracture toughness, Trans. JWRI 38 (2009) 71–78.
[10] X. Wang, O. Sanchez-Mata, S.E. Atabay, J.A. Muniz-Lerma, M.A. Shandiz, M. Brochu, Crystallographic orientation dependence of Charpy impact behaviours in stainless steel 316L fabricated by laser powder bed fusion, Addit. Manuf. 46 (2021) 102104.

[11] M. Odnobokova, A. Belyakov, N. Enikeev, R. Kaibyshev, R.Z. Valiev, Microstructural changes and strengthening of austenitic stainless steels during rolling at 473 K, Metals 10 (2020) 1614 (Basel).
[12] V. Torganchuk, A. Belyakov, R. Kaibyshev, On the transformation-induced plasticity of a medium-manganese steel, Mater. Lett. 304 (2021) 130599.
[13] R. Chaouadi, A. Fabry, On the utilization of the instrumented Charpy impact test for characterizing the flow and fracture behavior of reactor pressure vessel steels, in: From Charpy to Present Impact Testing, Elsevier, 2002, pp. 103–117.
[14] Y. Kimura, T. Inoue, F. Yin, K. Tsuzaki, Inverse temperature dependence of toughness in an ultrafine grain-structure steel, Science 320 (2008) 1057–1060.
[15] A. Dolzhenko, R. Kaibyshev, A. Belyakov, Tempforming as an advanced processing method for carbon steels, Metals 10 (2020) 1566 (Basel).
[16] R.L. Tobler, D. Meyn, Cleavage-like fracture along slip planes in Fe-18Cr-3Ni-13Mn-0.37N austenitic stainless steel at liquid helium temperature, Metall. Trans. A 19A (1988) 1626–1631.
[17] Y. Tomota, Y. Xia, K. Inoue, Mechanism of low temperature brittle fracture in high nitrogen bearing austenitic steels, Acta Mater. 46 (1998) 1577–1587.