

# Forming of the texture, structure and mechanical properties of cold-rolled AISI 304 steel

## Introduction

Stainless austenitic steels are commonly used for their possible unique mechanical and plastic properties combined with corrosion resistance. From a structural point of view these steels can be divided into steels with a stable austenite structure, steels with an unstable austenite structure and steels with an austenitic-ferritic structure [1-4].

Steels 18-8, i.e. those with metastable austenite, may undergo transformations induced both by plastic strain and by quenching. Depending on the steel's chemical composition, stacking fault energy, the size and shape of grains as well as plastic working conditions (degree, rate and temperature of strain), phase change in such steels may proceed as follows:  $\gamma \rightarrow \varepsilon$ ,  $\gamma \rightarrow \varepsilon \rightarrow \alpha'$  or  $\gamma \rightarrow \alpha'$  [5-6]. The obtained volume fraction of individual phases affects the mechanical properties and corrosion resistance of these steels [7]. During plastic strain, metastable austenitic steels undergo the development of an austenite texture as well as the development of a martensite texture, formed as a result of the transformation [8]. The texture plays a significant role in the process of product formation and in finished products. The texture obtained in the post-transformation material is closely connected with the texture of the material at the initial state. Metals and their alloys with a face-centred cubic lattice (A1) after plastic strain may have one of the two types of texture deformation, namely, a copper-type texture (high value of stacking fault energy) or an alloy-type texture (low value of stack-

ing fault energy). The crystallographic dependences between the austenite texture ( $\gamma$ ) and the martensite texture ( $\alpha'$ ) are described by a range of relationships such as the Bain relationship, the Kurdjumov-Sachs (K-S) relationship and the Nishiyama-Wassermann (N-W) relationship [9]. The purpose of this research work was to determine the impact of cold plastic strain during rolling, on shaping the texture, structure and mechanical properties of steel AISI 304.

## Materials used and research methodology

The research involved austenitic steel AISI 304 with the chemical composition presented in Table 1. The starting material in the form of a sheet (2 mm × 40 mm × 700 mm) underwent hyperquenching at 1100°C for 1 hour and cold rolling until it reached 70% strain. The rolling was carried out at room temperature, maintaining the same direction and side of the band being rolled.

Table 1: The chemical composition of steel AISI 304 [% by weight]

C	Cr	Ni	Mn	Si
0,033	18,08	9,03	1,32	0,41
Mo	P	S	N	Fe
0,23	0,026	0,002	0,026	70,84

Based on empirical formulas [10] the following parameters were calculated for the austenitic steel tested – stacking fault energy (SFE), the temperature at the beginning of a martensite transformation ( $M_s$ ) and

the temperature of a martensite transformation induced by a plastic strain (Md30), which were  $SFE = 32.1 \text{ mJ/m}^2$ ,  $M_s = -63.11^\circ\text{C}$  and  $M_{d30} = 22.7^\circ\text{C}$  respectively.

Metallographic tests were carried out on mechanically ground and polished longitudinal metallographic specimens. In order to reveal their structure, the specimens were etched in the so-called “aqua regia” heated up to approximately  $40^\circ\text{C}$ . The observations of the steel were carried out with a light microscope GX71 produced by OLYMPUS, using magnification from 100 to 1000x.

X-ray tests included the phase analysis of the surface and of the middle layer of the bands as well as the measurements of material textures at the initial state well as after hyperquenching and plastic strain. The x-ray phase analysis was conducted with a diffractometer D500, using a lamp with a copper anode  $\text{CuK}_\alpha$  ( $\lambda_{\text{K}\alpha} = 0.154 \text{ nm}$ ). Diffraction lines were registered in the range of angle  $2\Theta$  from  $40^\circ$  to  $92^\circ$ , by means of a stepping method, with a step of angle  $2\Theta$  equalling  $0.02^\circ$  and a pulse-counting time of 5 seconds in one measurement position.

for martensite, the orientation distribution function (ODF) and orientation fibres were calculated. The calculations of the quantitative fraction of martensite  $\alpha'$  in the structure of the steel involved the use of the magnetic method.

The tests of mechanical properties were carried out with a universal testing machine ZWICK 100N5A, using a static tensile test according to standard PN-EN ISO 6892-1:2010 [11]. The samples for tests were cut out of a sheet in parallel to the direction of rolling.

The hardness measurements of steel AISI 304 were carried out by means of the Vickers hardness tests, on metallographic specimens under a load of 50g, using a hardness testing machine PMT-3.

### Test results

Based on the metallographic tests, it was possible to establish that steel AISI 304 at the initial state is characterised by a structure composed of equiaxial austenite grains with an average diameter of approximately  $22 \mu\text{m}$ , containing annealing twins and few spot non-metallic inclusions (Fig. 1a).

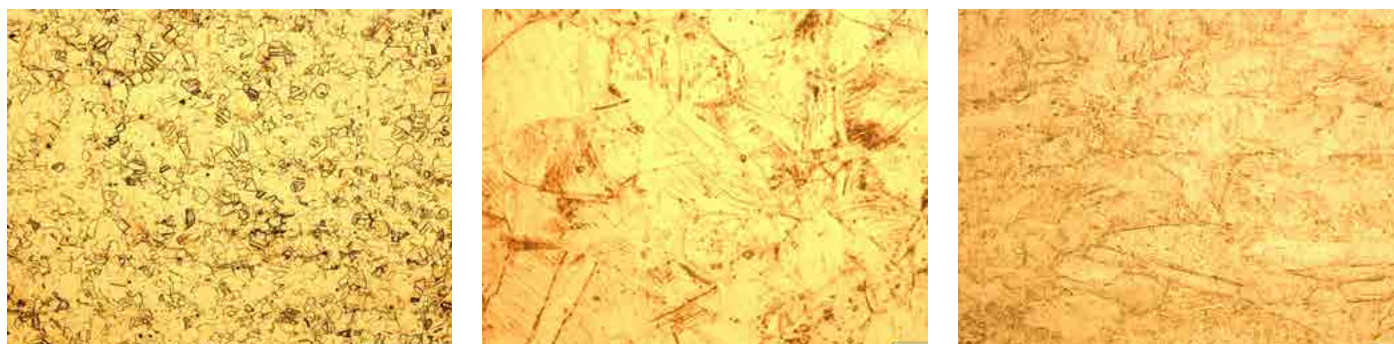


Fig. 1. Structure of steel AISI 304 at the initial state (a) and after 30% (b) and 70% (c) plastic strain, respectively; etchant – aqua regia

The tests of the textures were carried out using a diffractometer D8 Advance manufactured by the Bruker company and equipped with the Euler’s wheel. The source of radiation was a lamp with a cobalt anode  $\text{CoK}_\alpha$  ( $\lambda_{\text{K}\alpha} = 0.179 \text{ nm}$ ). On the basis of three incomplete polar figures of planes  $\{111\}$ ,  $\{200\}$ ,  $\{220\}$  for austenite and  $\{110\}$ ,  $\{200\}$ ,  $\{211\}$

The steel was characterized by a similar structure after hyperquenching.

After cold plastic strain within a  $10\% \div 20\%$  strain range, the steel revealed a structure composed of elongated grains  $\gamma$  with slip bands, deformed twins and non-metallic inclusions. The elongated character of austenite grains corresponds to the crushed condition of

the steel, where austenite grains undergo elongation in the direction of rolling. The structure of the steel after over 30% strain also revealed, in addition to elongated austenite grains with deformed twins and non-metallic inclusions, few areas of parallel lamellas characteristic of martensite  $\alpha'$  (Fig. 1b).

During cold rolling of steel AISI 304, an increase in strain is accompanied by the formation of new phase  $\alpha'$  dividing elongated austenite grains, which results in the so-called “refinement” of the steel structure and hardening of the steel (Fig. 1c). The metallographic observations revealed that the amount of the phase  $\alpha'$  in the structure of the steel tested increases along with the steel strain degree.

The results of the diffraction phase qualitative analysis of steel AISI 304 at the initial state (SD), hyperquenched (PP), and plastic-strained within a 10% ÷ 70% range are presented in Figure 2.

The diffraction phase analysis of steel AISI 304 at the as-delivered state revealed diffraction lines originating both from the phase  $\gamma$  and  $\alpha'$  (Fig. 2a, b). The diffraction patterns prepared for the surface of the steel at the initial state contain four diffraction lines originating from the phase  $\gamma$ , corresponding to the planes  $\{111\}\gamma$ ,  $\{200\}\gamma$ ,  $\{220\}\gamma$  and  $\{311\}\gamma$ . There is also one peak  $(110)\alpha'$  originating from the martensite phase (Fig. 2a). Identical diffraction lines can be observed in the diffraction patterns prepared for the middle layer (Fig. 2b). In addition, the middle layer of steel AISI 304 at the initial state revealed weak peaks  $(200)\alpha'$  and  $(211)\alpha'$ , originating from the martensite phase (Fig. 2b).

The diffraction patterns prepared for the surface and middle layers of the steel at the hyperquenched state did not reveal any significant changes as to the intensity of the individual diffraction lines originating from the phase  $\gamma$  when compared with the diffraction patterns of the steel at the initial state (Fig. 2a,b). The phase  $\alpha'$  at the hyperquenched state was not revealed.

The presence of the diffraction lines originating from the martensite phase on the diffraction patterns of steel AISI 304 at the initial state reveals the process of the phase change  $\gamma \rightarrow \alpha'$ , where the martensite revealed in the steel at the initial state might have been formed during the initial treatment of the material.

The diffraction image of the surface of steel AISI 304 after cold plastic strain within a 10% ÷ 40% range contains peaks  $(111)\gamma$ ,  $(200)\gamma$ ,  $(220)\gamma$  and  $(311)\gamma$  originating from austenite ( $\gamma$ ) as well as peaks  $(110)\alpha'$ ,  $(200)\alpha'$  and  $(211)\alpha'$  originating from martensite  $\alpha'$  (Fig. 2a). After further straining of the steel (over 50%), in the diffraction pattern one can observe the disappearance of diffraction lines  $(200)\gamma$  and  $(311)\gamma$  originating from austenite and the intensification of diffraction lines  $(200)\alpha'$  and  $(211)\alpha'$  originating from

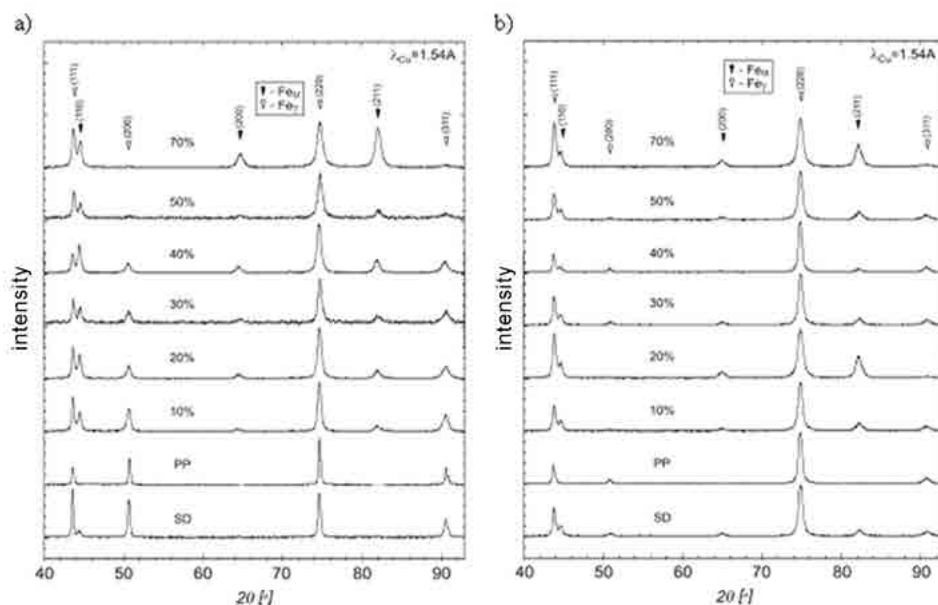


Fig. 2. Diffraction patterns of steel AISI 304 at the initial state (SD), hyperquenched (PP), and plastic-strained within 10% ÷ 70% range: a) surface, b) middle

martensite. In the whole range of strains, the strongest peak originating from austenite was  $(220)\gamma$ . In turn, the intensity of peak  $(111)\gamma$  underwent changes. After the maximum, i.e. 70% degree of strain, the strongest peak originating from martensite was peak  $(211)\alpha'$ . It was also possible to observe a widening of the diffraction lines originating both from phases  $\gamma$  and  $\alpha'$ , which was associated with an increase in structural defects formed during the plastic strain (Fig. 2a). In the diffraction patterns prepared for the middle layers of steel AISI 304, no significant changes in the intensity of individual diffraction lines were observed (Fig. 2b).

The conducted diffraction tests revealed that diffraction lines  $(111)\gamma$ ,  $(110)\alpha'$ ;  $(200)\alpha'$ ,  $(220)\gamma$ ,  $(211)\alpha'$  of  $(311)\gamma$  of the tested phases of steel AISI 304 after cold rolling with 40% strain reveal distinct steel texturing (Fig. 2a, b). The diffraction phase analysis of the steel deformed within a 10% ÷ 70% range did not reveal diffraction lines originating from the phase  $\epsilon$ , which is consistent with information found in reference publications [1-10]. A phase change proceeds directly according to the sequence  $\gamma \rightarrow \alpha'$ .

The texture of the austenite of steel AISI 304 both at the initial and hyperquenched state was relatively weak (Fig. 3a, b). Yet, it should be mentioned that the austenite of the tested steel is a metastable phase and that the development of the steel texture is complex. During the plastic strain of austenite the following processes take place at the same time: austenite texturing, phase change  $\gamma \rightarrow \alpha'$  and the change in orientation of the martensite formed during the strain. The texture of the steel strain is therefore described by the texture constituents as both of austenite and martensite (Fig. 4 and 5).

The main constituent of the austenite texture of the steel at the initial and hyperquenched state was a confined fibre  $\alpha = \langle 110 \rangle \parallel \text{ND}$  (ND – normal direction), in

which the strongest orientation was close to the orientation  $\{110\}\langle 112 \rangle$  of the alloy type. The maximum value of the orientation distribution function for this orientation was  $\text{ODF} = 3.9$  for the initial state and  $\text{ODF} = 3.3$  for the hyperquenched austenite respectively (Fig. 3a, b).

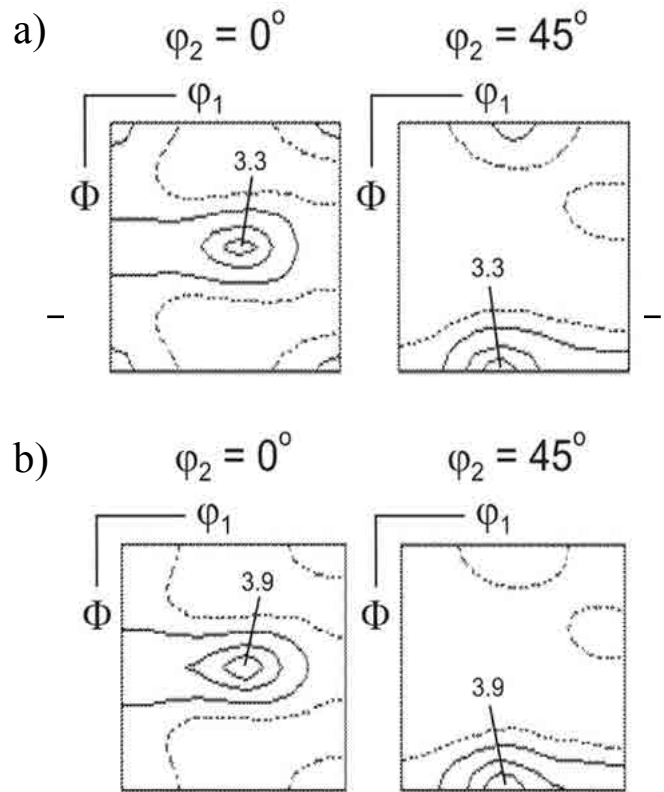


Fig. 3. Austenite texture at the initial state (a), at the hyperquenched state (b) on ODF cross sections  $\phi_2=0^\circ$ ,  $\phi_2=45^\circ$

In the texture of the austenite after the strain within a 10% ÷ 70% draft, it was possible to observe orientations described by the fibre  $\alpha = \langle 110 \rangle \parallel \text{ND}$ ,  $\tau = \langle 110 \rangle \parallel \text{TD}$  (TD – transverse direction),  $\beta = \{110\}\langle 112 \rangle$  by  $\{123\}\langle 634 \rangle$  to  $\{112\}\langle 111 \rangle$ . The strongest constituent of the austenite texture of steel AISI 304 was the orientation  $\{110\}\langle 113 \rangle$  of the fibre  $\alpha = \langle 110 \rangle \parallel \text{ND}$ , which is close to the constituent of the alloy type  $\{110\}\langle 112 \rangle$ . It is also possible to observe the Goss orientation  $\{110\}\langle 001 \rangle$  of the fibre  $\alpha = \langle 110 \rangle \parallel \text{ND}$  (Fig. 4a and 5a). The increase in the strain was accompanied by the reinforcement of the austenite texture. During the conducted tests it was possible to observe that an increase in the strain degree is accompanied first by the

elongation, and next by the contraction of the austenite fibre. The texture of the austenite deformation is typical of materials with low and medium value of SFE (Stacking Fault Energy).

During the plastic strain martensite is formed in the structure of the steel tested. The presence of the martensite and an increase in its content is the result of the phase change  $\gamma \rightarrow \alpha'$  induced by the strain. An increase in the strain is accompanied by a change in the martensite texture, caused by the texturing of the initial phase of the austenite, from which the phase  $\alpha'$  is formed. The texture of martensite after the strain within a 10% ÷ 70% range is described by the fibres of the orientation  $\alpha_1 = \langle 110 \rangle \parallel \text{RD}$  (RD – rolling direction),  $\gamma = \{111\} \parallel \text{ND}$  and  $\epsilon = \langle 001 \rangle \parallel \text{ND}$ . The dominant orientation of the martensite texture of steel AISI 304 was the orientation  $\{111\} \langle 112 \rangle$  of the fibre  $\gamma = \{111\} \parallel \text{ND}$  (Fig. 4b and 5b). After 30% strain, in the martensite texture, one can observe a rotated cubic orientation  $\{001\} \langle 110 \rangle$  (Fig. 4b). In turn, after 70% strain, the texture of strained martensite is dominated by the orientation  $\{112\} \langle 110 \rangle$

composing the confined fibre  $\alpha_1$  and the orientation  $\{111\} \langle 112 \rangle$  being the main constituent of the homogenous fibre  $\gamma$ . Rolling the steel with a 70% deformation degree causes that in the martensite texture the fibre

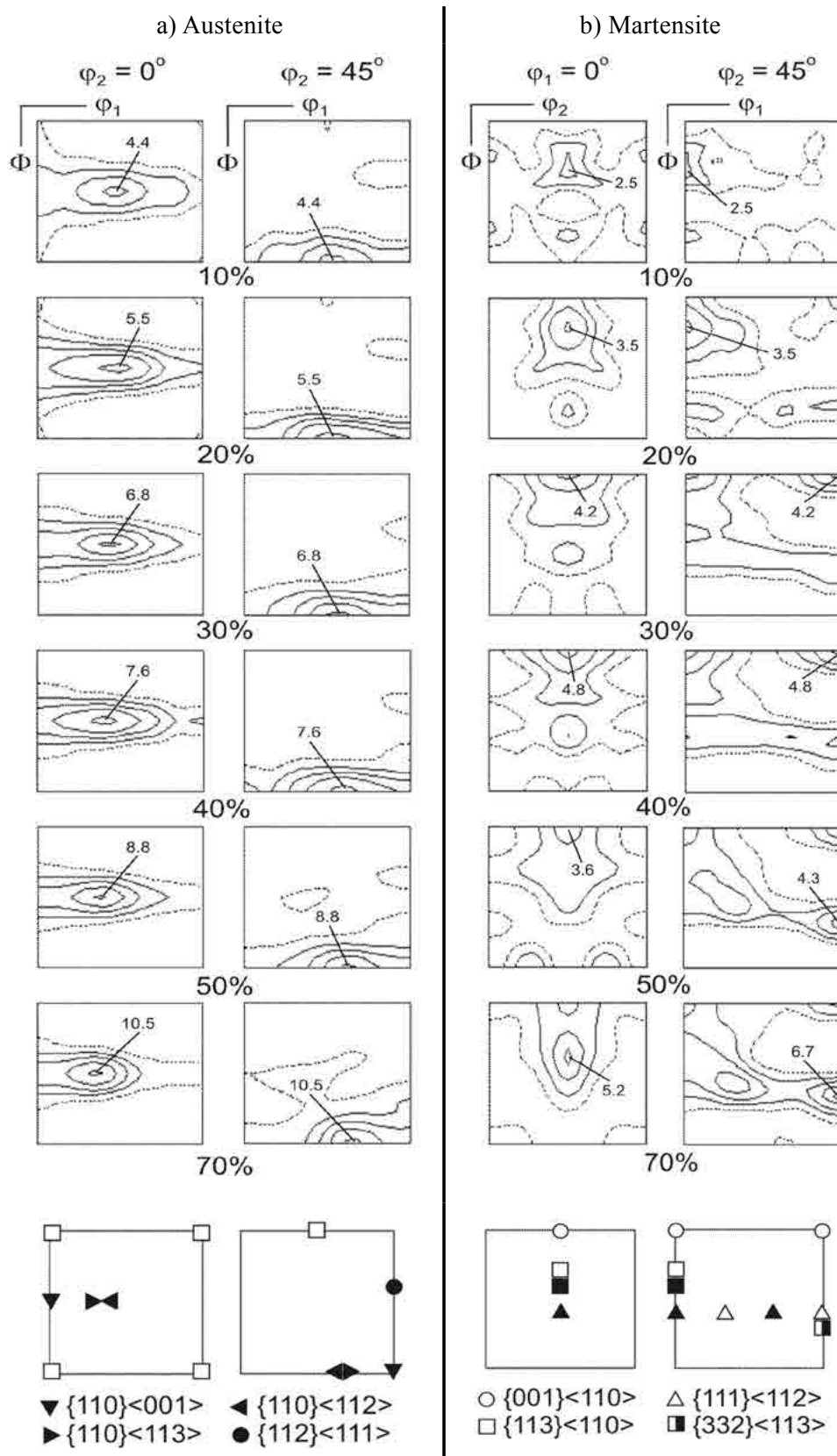


Fig. 4. Orientation distribution function for steel AISI 304 after various degrees of restraint presented in cross sections  $\phi_2 = 0^\circ$ ,  $\phi_2 = 45^\circ$  for austenite (a) and  $\phi_1 = 0^\circ$ ,  $\phi_2 = 45^\circ$  for martensite (b)

$\gamma = \{111\} \parallel \text{ND}$  is stronger than the fibre  $\alpha_1 = \langle 110 \rangle \parallel \text{RD}$  (Fig. 4b and 5b). The martensite texture remained weak within the whole range of restraint.

The crystallographic relationships between the texture of austenite and of martensite formed during the strain are best described by the Kurdjumow-Sachs (K-S) and Nishiyama-Wassermann (N-W) relationships.

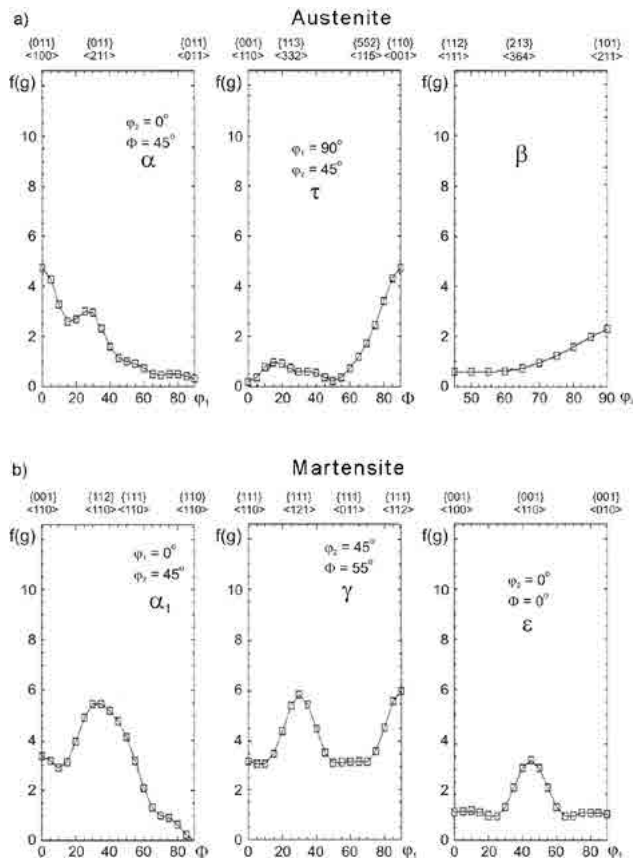


Fig. 5. Values of orientation distribution function  $f(g)$  along fibres  $\alpha$ ,  $\tau$ ,  $\beta$  for austenite (a) and fibres  $\alpha_1$ ,  $\gamma$ ,  $\epsilon$  for martensite (b) of steel AISI 304 after 70% strain

The conducted tests of mechanical properties revealed that the values of hardness HV0.05, tensile strength  $R_m$  and conventional yield point  $R_{p0.2}$  of steel AISI 304 increase along with an increase in the strain degree, whereas the value of elongation  $A$  decreases (Fig. 6).

At the initial state, the conventional yield point for steel AISI 304 is approximately 330 MPa, tensile strength approximately 647 MPa, hardness approximately 162 HV0.05, and elongation approximately 52%. An increase in the strain degree within a 10% ÷

50% range in the steel is accompanied by a change in its tensile strength from approximately 784 MPa to approximately 1257 MPa, conventional yield point from approximately 586 MPa to approximately 960 MPa, and elongation from approximately 32% to approximately 2%. The maximum, i.e. 70% strain of the steel, causes a significant increase in its values of  $R_m$ ,  $R_{p0.2}$  and HV0.05. Tensile strength increases to approximately 1496 MPa, yield point to approximately 1161 MPa, and hardness to approximately 400 HV0.05. It is also possible to observe a significant decrease of elongation i.e. to approximately 1% (Fig. 6).

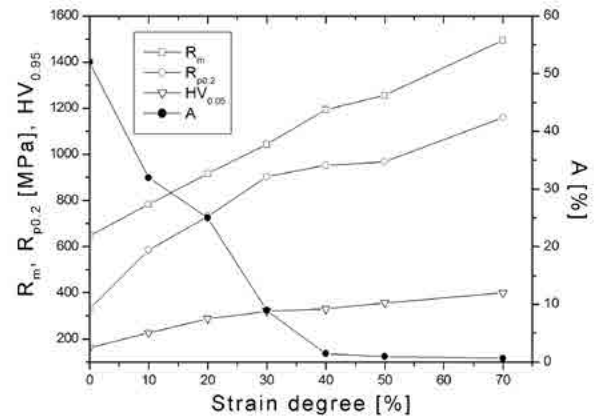


Fig. 6. Changes in mechanical properties of steel AISI 304 in the function of plastic strain

The tests of mechanical properties confirmed the analytical dependence of the yield point of austenitic steel AISI 304 on the strain degree in the process of rolling.

Based on the analysis of the magnetic tests, it was established that the amount of martensite phase in the structure of steel AISI 304 increases along with the strain degree in the process of rolling. After 70% strain, the steel contains approximately 28% of martensite  $\alpha'$ .

### Conclusions

The analysis of the test results for austenitic steel AISI 304 leads to the following conclusions:

1. The plastic strain induces the martensite transformation  $\gamma \rightarrow \alpha'$  in the whole range of applied strains.

2. At the initial state, the steel structure is composed of equiaxial grains  $\gamma$  with an average diameter of approximately 22  $\mu\text{m}$  with annealing twins and non-metallic inclusions, whereas after the plastic strain of the steel with a draft of approximately 30% - the structure of elongated austenite grains with areas of parallel lamellas characteristic of the martensite  $\alpha'$ .

3. The texture of the strained austenite is described by orientation fibres  $\alpha = \langle 110 \rangle \parallel \text{ND}$ ,  $\tau = \langle 110 \rangle \parallel \text{TD}$ ,  $\beta (\{110\} \langle 112 \rangle$  by  $\{123\} \langle 634 \rangle$  to  $\{112\} \langle 111 \rangle$ ); this texture is typical of materials with a low value of SFE.

4. An increase in the strain degree is accompanied by the development of the martensite texture; its main constituents are the orientations of the fibre  $\alpha_1 = \langle 110 \rangle \parallel \text{RD}$ ,  $\gamma = \{111\} \parallel \text{ND}$  and  $\varepsilon = \langle 001 \rangle \parallel \text{ND}$ .

5. The fraction of the martensite phase  $\alpha'$  in the steel structure increases along with an increase in the steel strain degree. After the maximum, i.e. 70% strain, the steel contains approximately 28% of the phase  $\alpha'$ .

6. The changes in the volume fractions of the phases  $\gamma$  and  $\alpha'$  during the cold strain of steel AISI 304 and the texture development in these phases, affect the mechanical properties.

***The research was conducted within the confines of research project no. 2632/B/T02/2011/40 funded by the National Science Centre (Narodowe Centrum Nauki).***

## References

1. Donadille C., Valle R., Penelle R.: *Development of texture and microstructure during cold-rolling and annealing of fcc alloys: Examples of an austenitic stainless steel*. Acta Metallurgica, 1989, vol. 37, s. 1547.
2. Abreu H., Carvalho S., Neto P., Santos R.: *Deformation induced martensite in an AISI 301LN stainless steels*. Materials Research, 2007, Vol. 10, s.359.
3. Angel T.: *Formation of martensite in austenitic stainless steels: Effects of deformation, temperature and composition*. Journal of the Iron and Steel Institute, 1954, s.165.
4. Ozgowicz W., Kurc A.: *The effect of the cold rolling on the structure and mechanical properties in austenitic stainless steels type 18-8*. Archives of Materials Science and Engineering, 2009, vol. 38, s.26.
5. Reed R.: *The spontaneous martensitic transformations in 18%Cr, 8%Ni steels*. Acta Metallurgica, 1962, vol. 10, s.865.
6. Kurc-Lisiecka A., Kalinowska-Ozgowicz E.: *Structure and mechanical properties of austenitic steel after cold rolling*. Archives of Materials Science and Engineering, 2011, vol. 44, s.148.
7. Kowalska J., Ratuszek W., Chruściel K.: *Crystallographic relations between deformation and annealing texture in austenitic steels*. Archives of Metallurgy and Materials, 2008, vol. 53, s.131.
8. Singh C. D., Ramaswamy V.: *Development of rolling texture in an austenitic stainless steel*. Textures and Microstructures, 1964, vol. 12, s.145.
9. Łuksza J., Rumiński M., Ratuszek W., Blicharski M.: *Texture evolution and variations of  $\alpha'$ -phase volume fraction in cold rolled AISI 301 steel strip*. Journal of Materials Processing Technology, 2006, vol. 177, s.555.
10. Padilha A.F., Pault R.L., Rios P.R.: *Annealing of cold-worked austenitic stainless steels*. ISIJ International, 2003, vol. 43, s.135.
11. Norma PN-EN ISO 6892-1:2010 *Metale. Próba rozciągania. Część 1: Metoda badania w temperaturze pokojowej*.