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**THE VARIATION OF STRAIN RATIO IN DEFORMED NICKEL SINGLE  
CRYSTALS WITH NOMINALLY STABLE CRYSTALLOGRAPHIC  
ORIENTATION**

**ZMIANA STOSUNKU WYDŁUŻEŃ W ODKSZTAŁCONYCH  
MONOKRYSTAŁACH NIKLU O NOMINALNIE STABILNEJ  
ORIENTACJI KRYSZTAŁOGRAFICZNEJ**

The unstable behaviour of the nominally stable single crystal samples of f.c.c. metals and alloys subdued to uniaxial straining is due, first of all, to two factors: the deviation of the crystallographic orientation from the nominal [uvw] direction, and imperfection of the inner structure, being the source of 'texture in a single crystal'. It is evident that in deformed single crystals the only parameter, which has an essential effect on plastic anisotropy, is crystallographic orientation. Thus, it is possible to evaluate the variation of crystallographic orientation at the tensile test indirectly, basing on the change of the strain ratio. This statement explains the significance of the strain ratio  $r$  vs. strain  $\varepsilon$  function in the analysis of instability of crystallographic orientation in single crystals subdued to the tensile test.

In the previous papers the authors have revealed the change of the strain ratio with the strain in several low stacking fault energy copper alloys and silver, and in medium s.f.e. copper. The present paper constitutes, in a way, a supplement to these results, describing the instability of the strain ratio and crystallographic orientation in the high stacking fault energy nickel single crystals.

The present paper should be considered as an attempt to explain the different characteristics of instability in nominally stable [100], [111] and [110] f.c.c. single crystals.

Niestabilne zachowanie obserwowane w nominalnie stabilnych próbkach monokryształów metali o sieci A1, poddanych jednoosiowemu rozciąganiu ma za główną przyczynę dwa czynniki: odchylenie orientacji krystalograficznej od nominalnej [uvw], i niedoskonałości struktury wewnętrznej, będącej przyczyną „tekstury w monokryształach”. Jest wiadomym, że w odkształconych monokryształach metali jedynym czynnikiem mającym istotny wpływ na

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anizotropię plastyczną jest orientacja krystalograficzna. Dlatego możliwa jest ocena zmiany orientacji krystalograficznej z wydłużeniem  $\varepsilon$  na drodze pośredniej, opierając się na zmianie stosunku wydłużeń  $r$ . To stwierdzenie wyjaśnia znaczenie funkcji  $r(\varepsilon)$  w analizie niestabilności orientacji krystalograficznej w monokryształach poddanych próbie rozciągania.

W poprzednich pracach autorzy przedstawili zmianę charakterystyki anizotropii z wydłużeniem w licznych stopach miedzi i w srebrze, charakteryzujących się niską energią błędu ułożenia i w miedzi (średnia energia błędu ułożenia). Obecna praca jest uzupełnieniem uzyskanych wcześniej wyników, na przykładzie niklu – metalu o wysokiej energii błędu ułożenia.

Praca stanowi próbę wy tłumaczenia zmiennego stopnia niestabilności nominalnie stabilnych monokryształów [100], [111] i [110] metali i stopów o sieci A1 poddanych próbie rozciągania.

## 1. Introduction

It is evident that in pure metals and single-phase polycrystalline alloys the crystallographic texture is the only parameter which has an essential effect on plastic anisotropy. Thus, it is possible to evaluate the variation of crystallographic orientation at the tensile test basing on the change of the strain ratio, on condition, however, that both, texture and anisotropy are referred to the same level of strain. Similarly, the experimentally determined variation of the strain ratio in deformed single crystals can be applied to describe the change of the crystallographic orientation [1–3].

The study of mechanical instability in single crystals requires the investigation of a zone of large deformation, generally exceeding 30–40 pct strain. However, the examination of instability of crystallographic orientation demands studying the zone of small strains, even below 0.1 pct elongation. It has been shown in several authors' papers [1–3] that the procedure is then rather complex due to the large and asymmetric scatter around the  $r(\varepsilon)$  function in this zone ( $r$  is the strain ratio, coefficient of plastic anisotropy, and  $\varepsilon$  is the strain, elongation). To avoid this difficulty, Truszkowski and Kloch [1–3] elaborated the method of calculation of the  $r(\varepsilon)$  function, taking into account the known or assumed value of experimental error  $c_0$ , and the hyperbolic function  $r(\varepsilon, a) = a_1\varepsilon + a_2 + a_3(\varepsilon^2 + a_4\varepsilon + a_5)^{1/2}$ , while the error  $c$  is decreasing with the strain. In this procedure the values of  $a$ -parameters are mostly based on that zone of the function where the scatter is small. The essential feature of this method is, that it is not based on the mean, but on the maximal errors. The calculation of the fitting function gives then good results even when the experimental scatter at the onset of the tensile test is very large [4].

A perfect single crystal in the form of a cylindrical sample is a body in which all atoms are in lattice points and the [uvw] direction is identical with the orientation of the sample axis. In reality, such structure can never be attained and every single crystal contains orientation errors; their effect is the variation of  $r$  with  $\varepsilon$  even in nominally stable f.c.c. [100], [111] or [110] single crystals. In principle, this unstable behaviour is due to two physical factors:  $\alpha$  – the error in the orientation of the sample axis, and/or  $\rho$  – the microscatter of orientation in the single crystal [5]. In the case, however, when

a single crystal sample reveals marked macroinhomogeneity, this factor should additionally be taken into account. The  $\alpha$  parameter characterises the incorrect preparation of the sample, while the  $\varrho$  – the imperfection of the inner structure of the crystal in the whole volume of the sample;  $\varrho$  is described by the full width at half maximum (FWHM) of the rocking curve. The X-ray or neutron diffraction rocking curve is determined by measuring the diffraction intensity as the function of the rocking angle  $\omega$  by which the crystal is rotated through its reflection position. Concluding: a real single crystal sample can be appraised as revealing a very strong, one component texture [5,6]; this conclusion leads to the notion of the ‘texture in single crystal’. The diminishing of the sharpness of texture accompanies the appearance of mosaic structure and results in the broadening of the rocking curve [6,7].

The principal aim of the paper is the determination of the  $r(\varepsilon)$  relationship for nickel single crystals representing the high stacking fault energy ( $\gamma_{Ni} = 125 \text{ mJm}^{-2}$ ) f.c.c. metals. Since the straining of aluminium single crystals ( $\gamma_{Al} = 135 \text{ mJm}^{-2}$ ) [8] has revealed the variation of the deformation characteristics along the length of a sample, the experiments in nickel single crystals were performed in the three sections of each sample.

## 2. Variation of strain ratio in tensile tested nickel single crystals

### 2.1. Experimental method

The single crystals were prepared from 99.73% purity nickel by the Bridgman method. The chemical composition is presented in Table 1. Round section test pieces

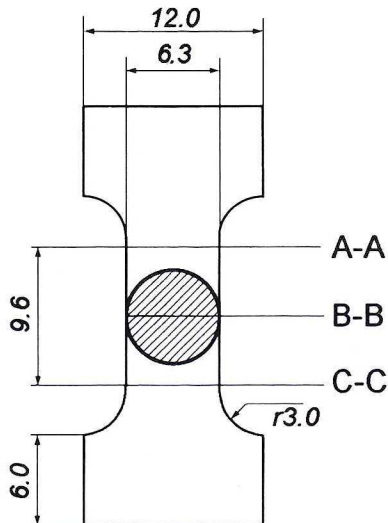


Fig.1. The scheme of the sample (the dimensions are in mm)

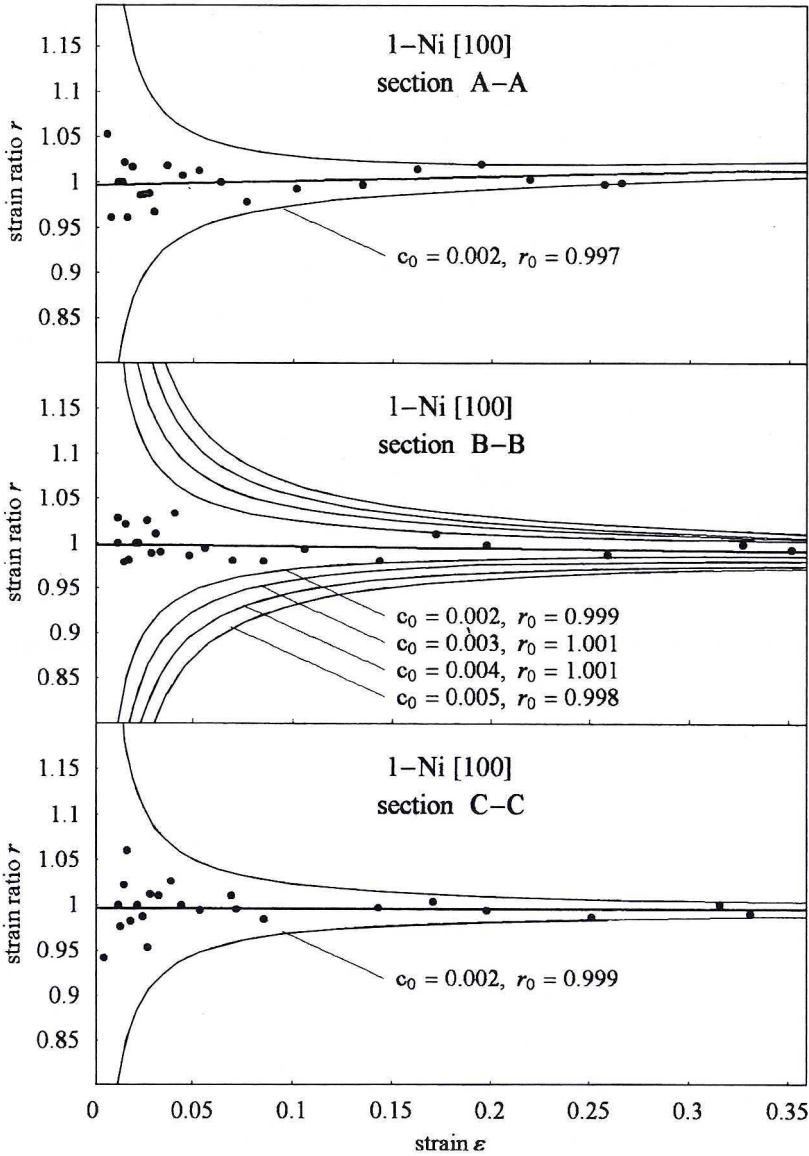


Fig.2. Variation of the strain ratio  $r$  with strain  $\epsilon$  in the 1-Ni[100] single crystal of nickel: sections A-A, B-B and C-C

TABLE 1

Chemical composition of the nickel sample

Ni	Co	C	Mn	Si	Fe	Mg	Cu
99.73	0.02	0.08	0.01	0.03	0.06	0.05	0.02



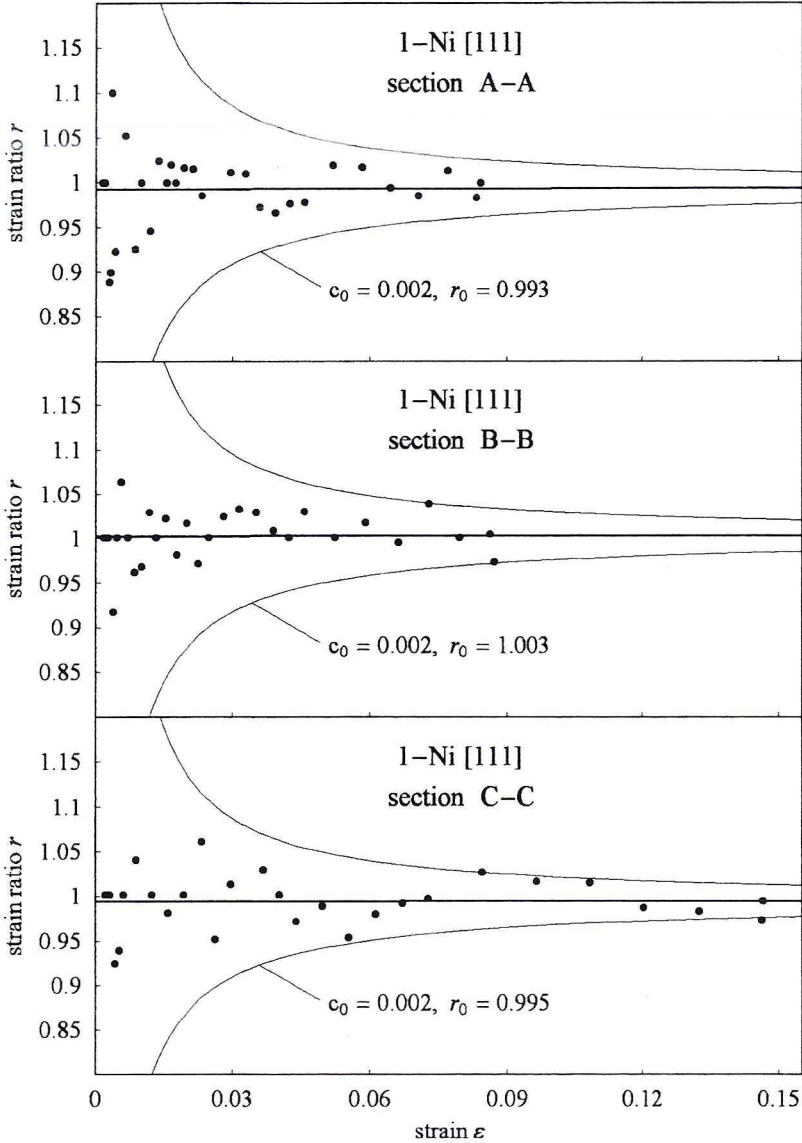


Fig. 3. Variation of the strain ratio  $r$  with strain  $\epsilon$  in the 1-Ni[111] single crystal of nickel: sections A-A, B-B and C-C

(Fig.1) with a diameter of ca 6 mm and ca 10 mm gauge lengths were used (accurate values are given in Tables 2, 3 and 4). After mechanical working they were chemically polished. The samples had the nominal [100], [111] and [110] orientations with accuracy of the  $\alpha$  angle estimated at  $30'$ . The tensile tests were carried out on an Instron testing machine at room temperature with initial strain rate  $3.5 \times 10^{-4} \text{ s}^{-1}$ . The single crystal specimens were fastened in special grips. The grip from the side of the A-A section was

stationary, while the grip from the side of the C-C section was fixed to the moving traverse of the testing machine. The measuring of the greater axis  $w$  and the smaller one  $t$  of the elliptical (after deformation) cross-section A-A, B-B and C-C (Fig.1) was executed with the accuracy  $10^{-3}$ mm.

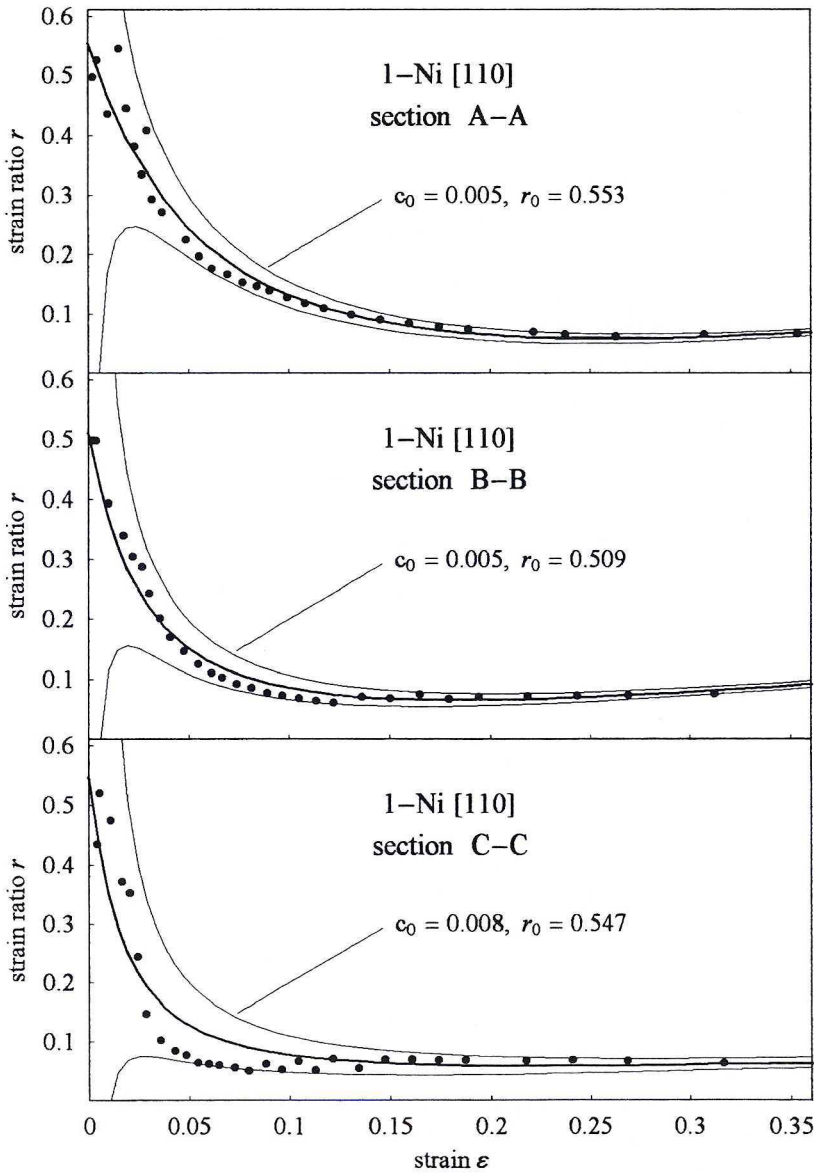


Fig.4. Variation of the strain ratio  $r$  with strain  $\epsilon$  in the 1-Ni[110] single crystal of nickel: sections A-A, B-B and C-C

## 2.2. Results

Tables 2, 3, 4 and Figures 2, 3 and 4 represent the  $r$  vs.  $\varepsilon$  relationships in the A-A, B-B and C-C sections of the 1-Ni [100], 1-Ni [111] and 1-Ni [110] samples, respectively. The proper values of  $c_0$  have been determined considering two criteria: (1) the zone between  $r^-(\varepsilon)$  and  $r^+(\varepsilon)$  functions [1,2] (denoted by thin lines in diagrams) contains all experimental points, and (2) the zone is at the same time the narrowest among those which possess this property. The procedure aiming at the determination of the proper  $c_0$  value is shown in Fig.2, section B-B.

The change of the strain ratio during uniaxial straining of the [100] nickel single crystal (Fig.2) is characterized by relatively small experimental scatter. The calculated fitting function is almost identical in all three analysed sections, and consequently the difference in the  $r_0$  value ( $r_0 = r(0)$ ) is insignificant (0.997, 0.999, 0.999), and very close to the theoretical one:  $r_{0[100]\text{theor.}} = 1.000$ .

Characteristic is the similar variation of the strain ratio in the [111] nickel single crystal (Fig.3); the difference in  $r_0$  values (0.993, 1.003, 0.995) is somewhat greater than in [100] crystal, but in the authors' opinion it may be also ascribed to the greater experimental error and/or some inhomogeneity at the length of a sample. However, the difference between the experimental  $r_0$  and the theoretical one ( $r_{0[111]\text{theor.}} = 1.000$ ) can be considered as negligible.

Contrary to the preceding cases, in the [110] nickel single crystal one observes inhomogeneous deformation along the length of a sample and the apparent instability of the strain ratio. The inhomogeneity of deformation manifests itself in different experimental scatter in A-A, B-B and C-C sections, while the varying instability of the strain ratio – in the change of the slope at the onset of the  $r(\varepsilon)$  function.

Even, when assuming that the central part of a sample is the most representative, the  $r_{0[110]\text{exper.}} = 0.509$  differs from the theoretical value  $r_{0[110]\text{theor.}} = 0.500$  [8], and this value rises much outside the centre:  $r_{0[110]\text{exper.}} \approx 0.55$ .

## 3. Discussion

On the basis of results of the previous research [5,6] one can expect the stable behaviour of the anisotropy characteristics in strained f.c.c. single crystal when  $\alpha$  and/or  $\varrho$  parameters are less than their critical values: (1)  $\alpha \leq \alpha_{\text{crit.}}$  and/or (2)  $\varrho \leq \varrho_{\text{crit.}}$ . The  $\alpha$  parameter describes the degree of accuracy of preparation of the sample, and depends much on the [uvw] orientation. In the research on the [100] copper single crystal, Takeuchi [9] as well as Truszkowski and Wierzbński [10] have found, when using different experimental methods that  $\alpha_{[100]\text{crit.}} \approx 2^\circ$ . One can suggest that in f.c.c. single crystal of the [111] orientation the  $\alpha_{[111]\text{crit.}}$  value is less than that of the [100] crystal, and even less at the [110] orientation.

The change of the anisotropy coefficient during uniaxial straining of the [100] nickel single crystal (Fig.2 and Table 2) is characterized by relatively small experimental scatter.

TABLE 2

Tensile test results of 1-Ni [100] single crystal. Section A-A:  $w_0 = 6.115$  mm,  $t_0 = 6.117$  mm. Section B-B:  $w_0 = 6.097$  mm,  $t_0 = 6.097$  mm. Section C-C:  $w_0 = 6.068$  mm,  $t_0 = 6.071$  mm

	A-A		B-B		C-C	
	$\epsilon$	$r$	$\epsilon$	$r$	$\epsilon$	$r$
1	0.0060	1.0530	0.0120	1.0278	0.0054	0.9415
2	0.0083	0.9617	0.0121	1.0000	0.0125	1.0004
3	0.0121	1.0003	0.0153	0.9786	0.0137	0.9765
4	0.0141	1.0003	0.0159	1.0209	0.0153	1.0223
5	0.0152	1.0221	0.0176	0.9814	0.0170	1.0607
6	0.0167	0.9616	0.0214	1.0000	0.0187	0.9828
7	0.0192	1.0176	0.0224	1.0000	0.0222	1.0004
8	0.0231	0.9861	0.0267	1.0251	0.0250	0.9872
9	0.0248	0.9870	0.0289	0.9885	0.0275	0.9530
10	0.0278	0.9884	0.0312	1.0107	0.0287	1.0122
11	0.0301	0.9678	0.0335	0.9901	0.0330	1.0106
12	0.0366	1.0186	0.0407	1.0334	0.0390	1.0266
13	0.0444	1.0078	0.0481	0.9861	0.0443	1.0004
14	0.0526	1.0131	0.0560	0.9939	0.0539	0.9942
15	0.0637	1.0003	0.0697	0.9807	0.0694	1.0103
16	0.0764	0.9783	0.0852	0.9801	0.0858	0.9845
17	0.1012	0.9935	0.1057	0.9934	0.0719	0.9957
18	0.1344	0.9977	0.1434	0.9805	0.1432	0.9980
19	0.1622	1.0157	0.1720	1.0104	0.1708	1.0047
20	0.1947	1.0209	0.1979	0.9981	0.1981	0.9950
21	0.2192	1.0036	0.2587	0.9885	0.2512	0.9872
22	0.2569	0.9989	0.3269	0.9988	0.3157	1.0017
23	0.2657	1.0003	0.3516	0.9933	0.3310	0.9911



TABLE 3

Tensile test results of 1-Ni [111] single crystal. Section A-A:  $w_0 = 6.115$  mm,  $t_0 = 6.114$  mm. Section B-B:  $w_0 = 6.101$  mm,  $t_0 = 6.107$  mm. Section C-C:  $w_0 = 6.062$  mm,  $t_0 = 6.075$  mm

	A-A		B-B		C-C	
	$\epsilon$	$r$	$\epsilon$	$r$	$\epsilon$	$r$
1	0.0013	0.9998	0.0016	1.0009	0.0019	1.0021
2	0.0019	0.9998	0.0026	1.0009	0.0296	1.0021
3	0.0031	0.8997	0.0037	0.9174	0.0041	0.9249
4	0.0034	1.0999	0.0045	1.0009	0.0051	0.9394
5	0.0027	0.8886	0.0054	1.0636	0.0059	1.0021
6	0.0162	1.0203	0.0068	1.0009	0.0087	1.0407
7	0.0040	0.9228	0.0083	0.9624	0.0122	1.0021
8	0.0063	1.0525	0.0100	0.9686	0.0157	0.9811
9	0.0085	0.9256	0.0116	1.0296	0.0192	1.0021
10	0.0098	0.9998	0.0131	1.0009	0.0232	1.0614
11	0.0118	0.9456	0.0152	1.0228	0.0262	0.9523
12	0.0136	1.0243	0.0176	0.9823	0.0297	1.0135
13	0.0154	0.9998	0.0199	1.0177	0.0367	1.0300
14	0.0192	1.0171	0.0224	0.9718	0.0402	1.0021
15	0.0212	1.0155	0.0247	1.0009	0.0439	0.9719
16	0.0231	0.9856	0.0280	1.0249	0.0497	0.9886
17	0.0358	0.9723	0.0315	1.0331	0.0554	0.9543
18	0.0393	0.9664	0.0352	1.0298	0.0612	0.9801
19	0.0424	0.9765	0.0388	1.0096	0.0670	0.9920
20	0.0458	0.9781	0.0423	1.0009	0.0726	0.9974
21	0.0518	1.0194	0.0457	1.0307	0.0844	1.0269
22	0.0582	1.0173	0.0524	1.0009	0.0965	1.0166
23	0.0643	0.9945	0.0590	1.0183	0.1083	1.0151
24	0.0704	0.9855	0.0661	0.9958	0.1203	0.9877
25	0.0768	1.0131	0.0727	1.0391	0.1326	0.9837
26	0.0831	0.9835	0.0795	1.0010	0.1462	0.9734
27	0.0841	0.9998	0.0862	1.0049	0.1464	0.9949
28			0.0872	0.9738		

TABLE 4

Tensile test results of 1-Ni [110] single crystal. Section A-A:  $w_0 = 6.151$  mm,  $t_0 = 6.117$  mm. Section B-B:  $w_0 = 6.091$  mm,  $t_0 = 6.074$  mm. Section C-C:  $w_0 = 6.096$  mm,  $t_0 = 6.068$  mm

	A-A		B-B		C-C	
	$\epsilon$	$r$	$\epsilon$	$r$	$\epsilon$	$r$
1	0.0020	0.4971	0.0020	0.4984	0.0038	0.4353
2	0.0042	0.5261	0.0034	0.4983	0.0052	0.5206
3	0.0096	0.4357	0.0098	0.3933	0.0107	0.4742
4	0.0152	0.5457	0.0175	0.3393	0.0163	0.3719
5	0.0190	0.4458	0.0218	0.3042	0.0202	0.3522
6	0.0230	0.3820	0.0265	0.2873	0.0242	0.2449
7	0.0266	0.3347	0.0302	0.2436	0.0281	0.1474
8	0.0316	0.2931	0.0353	0.2016	0.0354	0.1022
9	0.0366	0.2714	0.0406	0.1709	0.0426	0.0835
10	0.0293	0.4079	0.0476	0.1467	0.0484	0.0766
11	0.0488	0.2253	0.0546	0.1254	0.0545	0.0641
12	0.0554	0.1973	0.0612	0.1104	0.0597	0.0613
13	0.0618	0.1772	0.0666	0.1036	0.0649	0.0589
14	0.0695	0.1671	0.0740	0.0923	0.0726	0.0548
15	0.0772	0.1538	0.0814	0.0856	0.0797	0.0497
16	0.0840	0.1472	0.0891	0.0776	0.0882	0.0613
17	0.0905	0.1399	0.0966	0.0731	0.0961	0.0521
18	0.0994	0.1280	0.1051	0.0686	0.1045	0.0654
19	0.1081	0.1183	0.1134	0.0649	0.1130	0.0504
20	0.1175	0.1095	0.1221	0.0615	0.1215	0.0695
21	0.1312	0.0985	0.1361	0.0713	0.1343	0.0542
22	0.1455	0.0907	0.1502	0.0692	0.1473	0.0693
23	0.1597	0.0843	0.1646	0.0755	0.1605	0.0691
24	0.1744	0.0789	0.1793	0.0678	0.1737	0.0679
25	0.1890	0.0743	0.1939	0.0711	0.1872	0.0677
26	0.2214	0.0695	0.2185	0.0722	0.2175	0.0672
27	0.2375	0.0661	0.2434	0.0729	0.2409	0.0673
28	0.2628	0.0629	0.2692	0.0740	0.2687	0.0669
29	0.3073	0.0660	0.3122	0.0767	0.3168	0.0621
30	0.3532	0.0670			0.3677	0.0591

The calculated fitting function is very similar in all three analysed sections, and consequently the difference in extrapolated  $r_{0[100]_{\text{exper.}}}$  values is insignificant ( $r_{0[100]_{\text{exper.}}}$ : 0,997; 0,999; 0,999). The calculated  $r_{0_{\text{exper.}}}$  values are very close to the theoretical one:  $r_{0[100]_{\text{theor.}}} = 1.000$ .

Characteristic is that the analogical variations in [111] nickel single crystals (Fig.3 and Table 3) are more distinct than in [100] crystal and greater are differences in calculated in different A-A, B-B and C-C sections the  $r_0$  values ( $r_{0[111]_{\text{exper.}}} = 0.993$ ; 1.003 and 0.995). The theoretical value is  $r_{0[111]_{\text{theor.}}} = 1.000$ .

Therefore, the observed in the [100] and [111] nickel crystal:  $r(\varepsilon) = \text{const} = 1.000$ , shows that both above mentioned criteria for  $\alpha$  and  $\varrho$  are fulfilled: (1) the samples have been prepared with sufficient accuracy, and (2) the texture in both crystals is sharp enough.

On the other hand the instability of the strain ratio in the [110] nickel single crystal is evident (Fig.4 and Table 4). It is described by the rapid slope of the  $r(\varepsilon)$  function in the first range of strain; the different slope in A-A, B-B and C-C sections indicates the inhomogeneity of the material's behaviour along the length of the sample. If the middle part of a sample was assumed as the most representative for the tested material, the  $r_0$  value for the B-B section ( $r_{0[110]_{\text{exp.}}} = 0.509$ ) would be considered to be less differing from the theoretical one ( $r_{0[110]_{\text{theor.}}} = 0.500$ ). A much greater experimental scatter outside the centre of the sample is responsible for the greater difference in  $r_0$  values in A-A and C-C sections.

The general conclusion of the research on nickel single crystals is that in spite of a great experimental scatter the fitting function calculated on the basis of the Truszkowski-Kloch maximal error method describes well the variation of the strain ratio with strain. The best agreement between the experimental results and the theoretical  $r_0$  values occurs in case of a stable behaviour of the [100] crystal, being less good in the case of a greater scatter in the [111] crystal, and the worst in the case of instable behaviour of the [110] single crystal. And, as the condition  $\alpha \leq 30^\circ$  was secured in all tested single crystals, one can suppose that the criterion ( $\varrho \leq \varrho_{\text{crit.}}$ ) was not fulfilled only in case of the [110] crystal.

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