ISSN 1318-0010
KZLTET 32(6)463(1998)

INVESTIGATION ON YIELD STRESS AND STRAIN AGEING OF STRUCTURAL STEELS

RAZISKAVE NAPETOSTI TEČENJA IN DEFORMACIJSKEGA STARANJA KONSTRUKCIJSKIH JEKEL

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Prejem rokopisa - received: 1998-10-01; sprejem za objavo - accepted for publication: 1998-12-19

Five steels with yield stress 265 to 1003 MPa and the microstructure of polygonal ferrite and pearlite, distorted ferrite as well as tempered martensite were investigated. By all steels a good agreement was found between the empirical and the calculated yield stress. By steels with a microstructure of polygonal ferrite and pearlite mechanical properties are strongly changed by cold deformation and hardly affected by the following ageing annealing. Heat treated steels are wirtually non propensive to strain ageing.

Key words: structural steels, microstructure, mechanical properties, empirical yield stress, calculated yield stress, strain ageing

Raziskano je bilo 5 jekel z mejo plastičnosti med 265 in 1003 MPa ter mikrostrukturo iz poligonalnih ferita in perlita, spačenega ferita in popuščnega martenzita. Ujemanje med empirično in izračunano napetost tečenja je dobro pri vseh jeklih. Hladna deformacija močno spremeni lastnosti jekel z mikrostrukturo iz poligonalnih ferita in perlita, med tem ko se lastnosti komaj spremenijo pri žarjenju za staranje. Toplotno obdelana jekla so odporna proti deformacijskemu staranju.

Ključne besede: konstrukcijska jekla, mikrostruktura, mehanske lastnosti, empirična napetost tečenja, izračunana napetost tečenja, deformacijsko staranje

1 INTRODUCTION

Weldable structural steels are manufactured in a wide range of yield stress. By yield stress up to appr. 500 MPa the microstructure consists of polygonal ferrite and pearlite and four mechanisms contribute to the strengthening: solid solution, second phase, grain size and precipitation of AlN and NbC in austenite. In steels with yield stress up to appr. 1000 MPa the microstructure consists of distorted and acicular ferrite or tempered martensite. In the last case an additional increase in yield stress is obtained also by ferrite hardening through cementite precipitates, and precipitation of VC and NbC in ferrite. Properties, especially notch toughness, transition temperature, and propensity to strain ageing, depend on the microstructure and differ strongly between different steels. In this article ferrite pearlite and tempered martensite steels will be discussed with special accent on yield stress and propensity to strain ageing. Two ferrite-pearlite steels two tempered martensite steels and one distorted ferrite steels will be examined on the base of experimental data in **ref. 1**.

2 COMPOSITION OF STEELS

The composition of steels is given in **table 1**. Steels 2,3,4, and 5 were molten in electric arc furnace, and steel 1 in oxygen convertor, continous cast to over 200 mm slabs and hot rolled to plates of thickness 20 to 30 mm (steels 1,2,3, and 4) and 50 mm (steel 5). Steels 1 and 2 were air cooled from the finishing rolling temperature, while steels 3,4 and 5 were quenched and tempered. The microstructure of steels 1 and 2 consists of polygonal ferrite and pearlite **(figure 1 and 2)**. The microstructure of steel 3 consists of fine grained distorted and acicular ferrite, rare cementite precipitates and small colonies of distorted pearlite **(figure 3 and 4)**. The microstructure of steels 4 and 5 consists of tempered martensite, thus of acicular ferrite conserving the habitus of martensite, and cementite precipitates **(figures 5,6,7, and 8)**. Chromium, nickel and molybdenum are added to steels 3,4, and 5 with the aim to increase the hardenability, especially by plates with greater thickness. The carbon equivalent is found in a relatively wide range. It warrants a good weldability of all steels after appropriate preheating for steels 2,4, and 5, while steels 1 and 3, are welded without preheating. It was established that steel 3 is particularly resistant to hydrogen induced cracking (2,3) and very suited for great vessels for hydrocarbons.

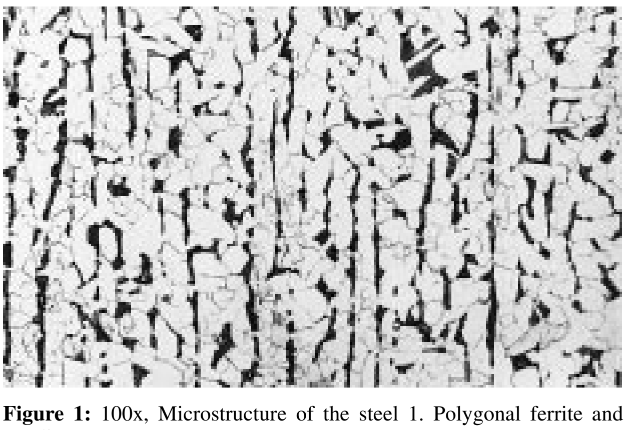

Figure 1: 100x, Microstructure of the steel 1. Polygonal ferrite and pearlite
Slika 1: 100x, Mikrostruktura jekla 1. Poligonalna ferit in perlit

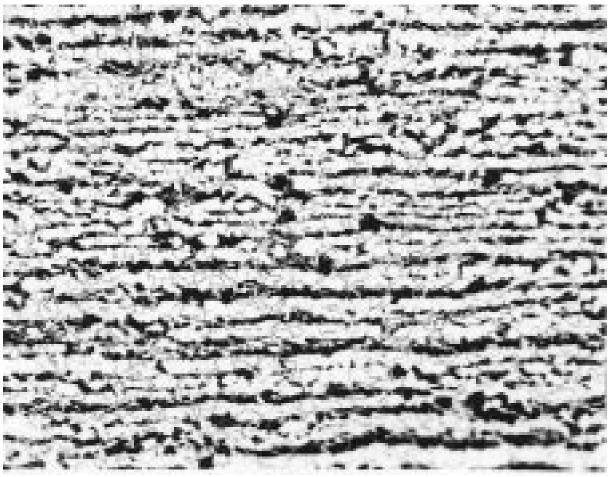

Figure 2: 100x, Microstructure of the steel 2. Polygonal ferrite and pearlite
Slika 2: 100x, Mikrostruktura jekla 2. Poligonalna ferit in perlit

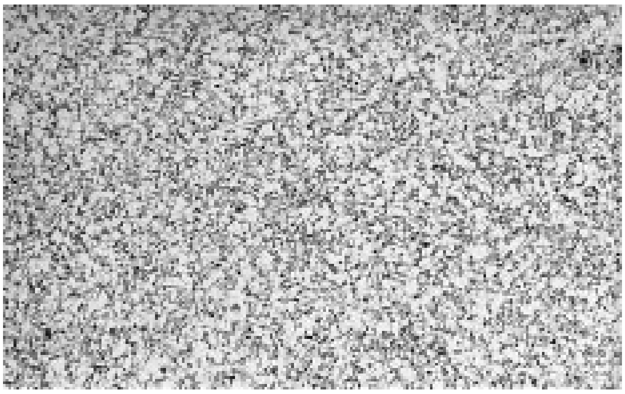

Figure 3: 100x, Microstructure of the steel 3. Distorted ferrite and pearlite
Slika 3: 100x, Mikrostruktura jekla 3. Spačeni ferit in perlit

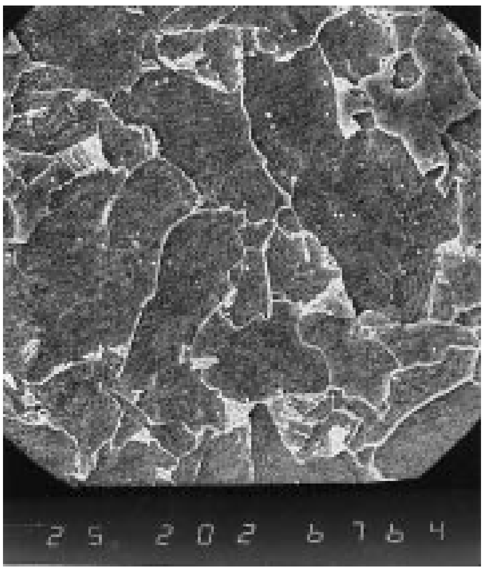

Figure 4: 2000x, Microstructure of the steel 3. Distorted ferrite, pearlite, cementite precipitates
Slika 4: 2000x, Mikrostruktura jekla 3. Spačeni ferit, perlit in cementitni izločki

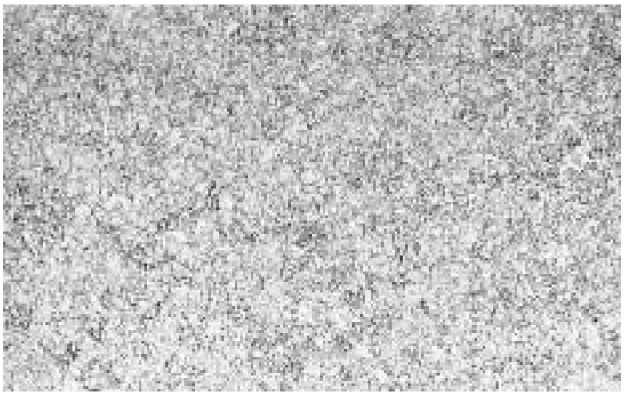

Figure 5: 100x, Microstructure of the steel 4. Tempered martensite, acicular ferrite and cementite precipitates
Slika 5: 100x, Mikrostruktura jekla 4. Popuščeni martensit, acikularni ferit in cementitni izločki

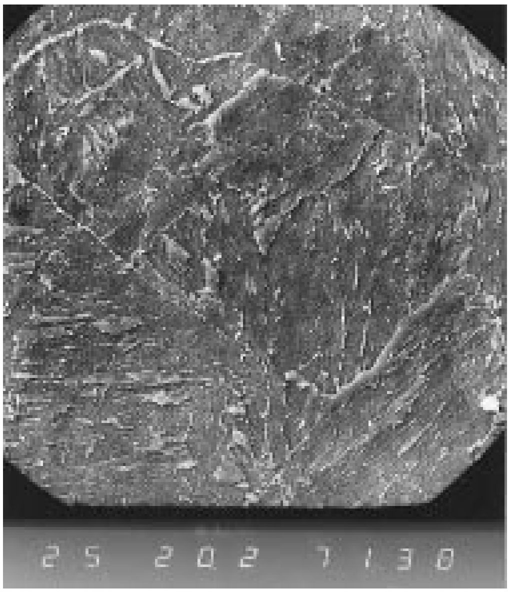

Figure 6: 2000x, Microstructure of the steel 4. Tempered martensite acicular ferrite and cementite precipitates
Slika 6: 2000x, Mikrostruktura jekla 4. Popuščeni martenzit, acikularni ferit in cementitni izločki

Figure 7: 100x, Microstructure of the steel 5. Tempered martensite, acicular ferrite and cementite precipitates
Slika 7: 100x, Mikrostruktura jekla 5. Popuščeni martenzit, acikularni ferit in cementitni izločki

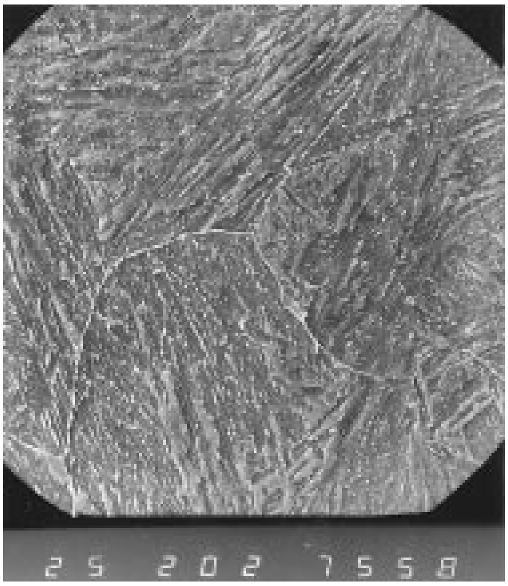

Figure 8: 2000x, Microstructure of the steel 5. Tempered martensite, acicular ferrite and cementite precipitates

Slika 8: 2000x, Mikrostruktura jekla 5. Popuščeni martenzit, acikularni ferit in cementitni izločki

Tabela 1: Chemical composition of steels

Steel	Element in wt %							
	C	Si	Mn	P	S	Cr	Ni	Nb
1	0.21	0.25	0.51	0.01	0.02	0.02	0.04	-
2	0.17	0.32	1.28	0.02	0.01	0.21	0.13	-
3	0.08	0.34	0.36	0.01	0.004	0.54	0.27	0.058
4	0.11	0.28	0.27	0.01	0.007	1.07	2.80	-
5	0.14	0.29	0.51	0.012	0.009	1.64	2.76	
	Mo	Cu	V	Al	N			
1	-	0.10	-	0.027	0.008			
2	0.35	-	0.045	0.009				
3	0.27	0.36	-	0.052	0.007			
4	0.26	0.20	0.06	0.043	0.007			
5	0.42	0.21	0.01	0.054	0.007			

3 TENSILE PROPERTIES

Tensile properties of as delivered and strain aged steels are shown in **tables 2 and 3**. By increasing yield stress of as delivered steels uniform elongation is strongly decreased, while reduction of area remains very high. The ratio yield stress over tensile strenght increases from 0.58 by steel 1 to 0.94 by steel 4 and it remains

Table 2: Tensile properties

Steel	Plate Mm	Yield stress MPa	Tensile strength MPa	Uniform elongation %	Reduction of area %	Yield stress	Tensile strength	Uniform elongation	Reduction of area
Absolute values						Relative values			
As delivered steels									
1	30	265	458	21.0	56.1	1	1	1	1
2	25	377	553	14.3	69.7	1	1	1	1
3	25	522	604	10.9	78.8	1	1	1	1
4	20	737	787	7.5	72.5	1	1	1	1
5	50	1003	1070	3.8	63.4	1	1	1	1
Strain aged steels									
1	30	484	596	6.9	49.5	1.82	1.3	0.33	0.88
2	25	586	656	4.8	64.0	1.55	1.17	0.33	0.92
3	25	684	713	2.7	73.5	1.31	1.18	0.25	0.93
4	20	883	895	2.8	70.8	1.20	1.12	0.37	0.97
5	50	1149	1200	1.6	59.6	1.14	1.12	0.42	0.94

Table 3: Notch toughness properties

Steel	Plate	Upper shelf NT[1] J	Transition[2] temperature °C	Nil ductility temperature °C	Upper shelf NT	Transition temperature	Nil ductility temperature
Absolute values					Difference[3]		
As delivered steels							
1	30	111	0				
2	25	147	-20	-115			
3	25	245	-75	-133			
4	20	160	-100	-124			
5	50	102	-50	-115			
Strain aged steels							
1	30	9					
2	25	119	0	-120	-28	+30	-5
3	25	245	-67	-145	0	+10	-12
4	20	156	-95	-135	-4	+15	-9
5	50	82	-45	-125	-20	+5	-10

[1] NT Notch toughness, [2] At 50% upper shelf notch toughness, [3] Difference between as delivered and strain aged steels

Table 4: Tensile properties of as delivered, cold deformed and strain aged steels

Steel	Yield stress MPa	Tensile strength MPa	Uniform elongation %	Yield stress MPa	Tensile strength MPa	Uniform elongation %	Yield stress MPa	Tensile strength MPa	Uniform elongation %
	As delivered steels			Cold deformed steels			Strain aged steels		
	Absolute values								
2	366	553	14.3	595	624	4.1	586	656	4.8
3	522	604	10.9	694	724	2.5	694	713	2.7
4	737	787	7.5	863	866	2.0	883	895	2.2
	Relative values								
2	1	1	1	1.62	1.13	0.28	1.60	1.18	0.33
3	1	1	1	1.33	1.20	0.23	1.33	1.18	0.25
4	1	1	1	1.11	1.10	0.26	1.20	1.14	0.29

unchanged by the further very significant increase of yield stress to 1003 MPa by steel 5.

The notch toughness transition temperature is lower for all three steels with greater yield stress and particularly low by steel 3 and 4, the first with a microstructure of distorted ferrite and the second with a microstructure of tempered martensite. The nil ductility temperature is similar by steels 2,3,4, and 5 and it is significantly higher by steel 1.

After strain ageing the yield stress is stronger increased by lower as delivered value. For the increase of yield stress after strain ageing $\Delta\sigma_y$ the following relationship was derived (4):

$$\Delta\sigma_y = 1647.5\ (\sigma_y - 200)^{-0,674} \qquad (1)$$

Tensile strength is increased after strain ageing to a smaller extent then yield stress. The increase is greater by steel 1 (30%) and smaller by steels 2,3,4, and 5.

After strain ageing the reduction of area is smaller by 3 to 12%, while uniform elongation is diminished much stronger, by 58 to 75% of the value by as delivered steels. It is interesting that the smallest relative lowering of uniform elongation is found by steel 5 with the highest yield stress and lowest as delivered uniform elongation.

Notch toughness transition temperature is increased after strain ageing the must by the two steels with a microstructure of polygonal ferrite and pearlite and the lower yield stress. It is lowered also by steels 3,4, and 5, however very little, only by 5 to 8°C by an initial value of -50°C or lower.

After strain ageing the nil ductility temperature (NDT) is somewhat surprisingly lowered by all steels. In **ref. 5** the decrease of NDT is explained in terms of increased yield stress. For a group of steels with yield stress 265 to 1003 MPa the following empirical relation connecting fracture toughness (K_{IC}), yield stress (σ_y) and notch toughness (CVN) was developed (5):

$$K_{IC} = 0.776\ \sigma_y^{0.60}\ (CVN)^{0.19} \qquad (2)$$

By testing of 10 steels in the given yield stress range a regression coefficient of 0.92 was established confirming the great reliability of the equation (5).

Also the mechanical properties in the range T_{NDT} ±20°C were investigated. Since these properties are discussed in **ref. 6**, only a summary will be given here.

When compared to room temperature the yield stress and particularly tensile strength are significantly increased in the range T_{NDT} ±20°C, while uniform elongation and reduction of area remain virtually unchanged. Notch and fracture toughness are also decreased, generally, the stronger the greater is the room temperature value. In the interval T_{NDT} -20°C to T_{NDT} +20°C notch and fracture toughness increase much more than yield stress and tensile strength. Some properties change virtually linearly from room temperature to NDT.

In strain ageing two processes are involved, a 10% cold deformation by rolling and a 30 min. annealing at 250°C. On steels 2,3, and 4 with a basically different microstructure tests were performed in as delivered, cold deformed and strain aged state. The results are given in **table 4** as absolute and relative values. All properties are strongly affected by cold deformation. Uniform elongation is diminished by all three steels for appr. 75%. Yield stress is increased the more, the lower was the initial value, by more than 60% by steel 2 with a microstructure of polygonal ferrite and pearlite, and by 17%, by steel 4 with a microstructure of tempered martensite. Tensile strenght is increased after cold deformation in all three steels to a similar extent, between 10 and 20%. After the following annealing tensile properties are virtually unchanged when compared to the properties after cold deformation, while uniform elongation is even slightly increased. It is therefore evident that the tensile properties of steels with the three different types of microstructure were affected mostly by cold deformation and that the following annealing had a very limited effect.

4 EMPIRICAL AND THEORETICAL YIELD STRESS

Yield stress is calculated as the summa of the contribution of several strengthening mechanisms and its value for α iron, which according to **ref. 7** amounts to 30 MPa. Carbon and nitrogen are found in solid solution in ferrite. According to the solubility product log (Al) x (N) =

-7500/T + 1.48 in **ref. 8** in a 0.04% Al and 0.01% N steel appr. 0.002% N remains in solid solution in austenite by normalising annealing. No data were found on the solid solubility of aluminium nitride in ferrite. It is supposed that because of the great dilution and the slow diffusion of aluminium also after cooling 0.002% N remains in solid solution in ferrite.

In **ref. 9** the equilibrium solid solubility of carbon in ferrite is given as

$$N_C/N_{Fe} = 0.12 \exp^{-4850/T} \quad (3)$$

With N_C and N_{Fe} the respective numbers of carbon and iron atoms and T the temperature in K. Considering the average tempering temperatures of 615°C, 605°C, and 480°C the following solid solution are calculated: 0.011% C for the steel 3, 0.01% C for the steel 4, and 0.004% C for the steel 5.

The austenite to ferrite transformation temperature ranges 850 to 700°C for steel 1 and 780 to 660°C for steel 2 were taken from **ref. 10**. According to the binary Fe-C diagram an average solubility of 0.014% C for steel 1 and 0.016% C for steel 2 corresponds to the transformation temperature range.

However, the solubility of carbon in ferrite decreases strongly by decreasing temperature. From 0.0218% at the eutectoide temperature by 727°C, it drops to appr. 0.002% at 500°C. The diffusion path of carbon by 1 min. isothermal holding at 500°C is of the magnitude of ferrite grain size. Thus, carbon diffusion is virtually no obstacle for the precipitation of residual carbon, either to cementite lamellae in pearlite grains, or to independent precipitates. By observation in SEM at sufficient magnification in interior of ferrite grains cementite precipitates were found **(figure 9)**. The size of these precipitates was on average near 0.10 μm. Supposing that by continous transformation of steel 1 - 0.014% carbon was left in solution in ferrite and that by cooling to 500°C 0.014 - 0.002 = 0.012% C precipitated producing $2.6.10^{-3}$ mol of cementite. By an average size of precipitates of 10^{-4} cm, the yield stress increase would amount according to equation 5 to appr. 2.6 MPa. The strengthening effect of carbon and nitrogen in interstitial solution in ferrite is very strong (11,12,13) and amounts to 4.5 MPa per 10^{-3}% C and 3.4 MPa per 10^{-3}% N in solid solution. Since, and as explained allready, probably even less than 0.002% of carbon remains in solid solution, the interstitial strengthening is estimated to 10 MPa for carbon and 7 MPa for nitrogen for all steels investigated.

Figure 9: 10000x, Cementite precipitates in ferrite grains in steel 1
Slika 9: 10000x, Cementitni izločki v feritu v jeklu 1

Other alloying elements, such as manganese, silicon, copper etc are in substitutional solid solution in ferrite.

According to **ref. 11,12, and 13** the solid solution strenghtening of α phase is proportional to the content of elements, thus $\Delta R_E = \alpha.C$ each having a different strengthening constant α. It was assumed also that carbide forming elements, such as chromium and molybdenum remain in solid solution.

Yield stress is increased by low carbon content also proportionally to the share of lamellar pearlite in the microstructure (14). Yield stress is increased also proportionally to the inverse of the square root of grain size (d), thus

$\Delta R_E = K \,.\, D^{-1/2}$ according to **ref. 15**

$$\Delta R_E = 17.4.D^{-1/2} \quad (4)$$

with D the grain size in mm.

Ferrite grain size was assessed by manual measuring of the size of grains in two directions.

In **ref. 7** the following equation is given for the increase of yield stress due to a volume f of precipitates of diametre d

$$\Delta R_E = \alpha\, G.b.f^{1/3}\, d^{-1} \quad (5)$$

with α - a constant (α≈1), G - shear modulus of ferrite ($G = 75.8.10^5$ MPa) and b - the Burgers vector ($2.5.10^{-10}$ m).

Three types of precipitation strengthening occur in the investigated steels:

- strengthening due to the tempering carbide precipitates of size of appr. 10^{-7} m, which is calculated according to equation 5 assuming that all carbon is bound to cementite;
- strengthening due to the precipitates of aluminium nitride and niobium carbonitride formed according to the solubility products by austenitising annealing and having a size of $2\text{-}3.10^{-8}$ m. The resulting strengthening is calculated using the equation (16):

$$\Delta E = 157.5/d\ (\%Nb^{1/3} - 0.12) \quad (6)$$

ΔE - increase of d yield stress in kp/mm^2,
d - precipitate size in mm,
%Nb - weight Nb content.

Since the quantity of precipitates produced depends on the number of atoms and not on their weight, the con-

Table 5: Share of strengthening mechanisms in the yield stress (YS)

Steel	1	2	3	4	5	1	2	3	4	5
Strength.mechan.	Absolute values, MPa					Relative values, % Theor.YS				
1. α iron YS	30	30	30	30	30	11.6	8.1	5.9	4.0	3.1
2. Pearlite	50	61	15	-	-	19.4	16.4	3.0	-	-
3. Interstitial sol.	17	17	17	17	17	6.6	4.6	3.4	2.2	1.7
4. Substit.sol.	56	104	136	245	300	21.7	27.9	27.0	32.3	30.8
5. Grain size	85	135	254	348	503	32.9	36.3	50.4	45.8	51.7
6. Dispersion	-	-	9	42	89	-		1.8	5.5	9.2
7. Prec.γ phase	20	25	43	25	27	8.0	6.7	8.5	3.3	2.8
8. Prec.α phase	-	-	-	52	6	-		-	6.8	0.6
A Theor. YS	258	372	504	759	972	100	100	100	100	100
B Emp. YS	265	377	522	737	1003	-		-	-	
B - A	7	5	18	22	29	2.7	1.3	3.4	3.0	2.9

tent of aluminium is multiplied with the ratio of atomic weights of niobium and aluminium. According to **ref. 17**

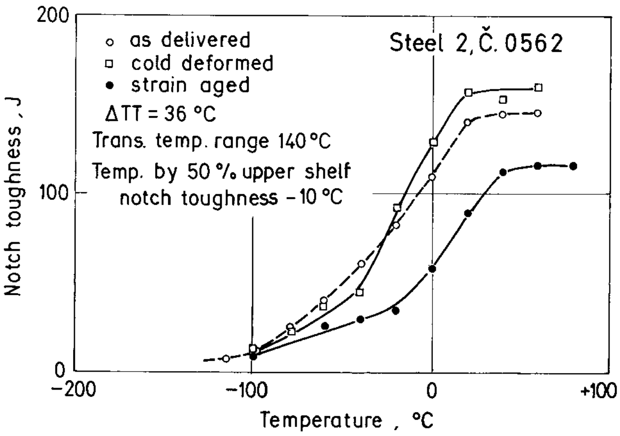

Figure 10: Steel 2. Influence of testing temperature on notch toughness
Slika 10: Jeklo 2. Vpliv temperature preizkušanja na zarezno žilavost

in the calculation an average size of precipitates d = 2,6.10^{-8} m was considered.

The third precipitation strengthening results from the precipitation of vanadium carbide in ferrite. No data were found on the size of particles which would precipitate by the tempering of steel 4 containing 0.06% of vanadium. For that reason, this strengthening was determined by interpolation of data in **ref. 18**, in which the increase of hardness was established after 6 hr of annealing of a 0.15% C, 1,43% Mn, and 0.07% V steel at 600 and 650°C. Assuming a ratio yield stress versus tensile strength the increase of yield stress due to precipitation hardening of ferrite by vanadium carbide amounts to appr. 6 MPa.

In **table 5** the share of different strengthening mechanisms in absolute values and relative share, the theoretical and the empirical yield stress are shown.

In all steels the grain size strengthening is predominant, since it contributes to 32.9 to 51.7% of the yield stress. Second by magnitude is the contribution of solid solution strengthening which amounts to 21.7 to 32.3%

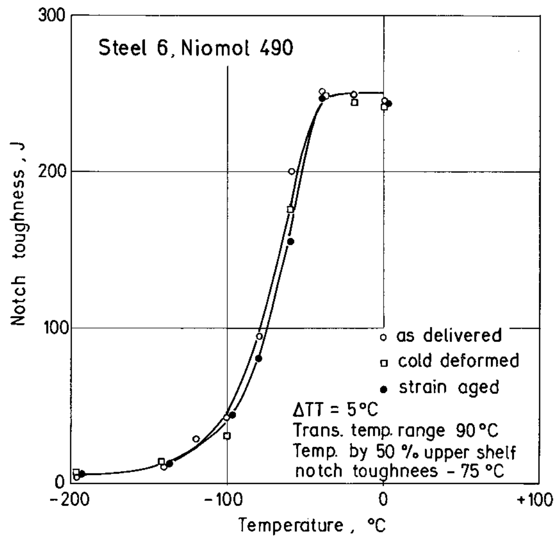

Figure 11: Steel 3. Influence of testing temperature on notch toughness
Slika 11: Jeklo 3. Vpliv temperature preizkušanja na zarezno žilavost

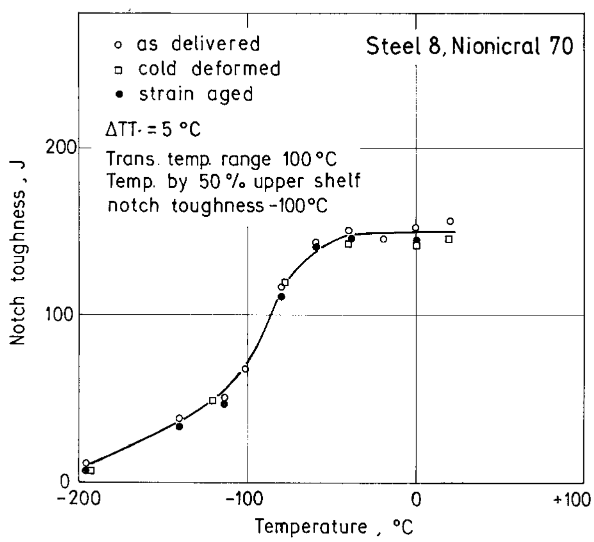

Figure 12: Steel 4. Influence of testing temperature on notch toughness
Slika 12: Jeklo 4. Vpliv temperature preizkušanja na zarezno žilavost

of the yield stress. The contribution of lamellar pearlite amounts to appr. 18% in both steels with a microstructure of polygonal ferrite and pearlite. The contribution of precipitate strengthening is small, also in both steels with a microstructure of tempered martensite. It is, thus confirmed that higher alloying of these steels is necessary to obtain a sufficient hardenability. It is also evident that in these steels a higher yield stress can be obtained only through finer martensite, f.i. the addition of niobium which would help to obtain a smaller austenite grain size by austenitizing before the quenching.

The contribution of α iron yield stress is the greater by steel 1 with 11.6%. It is very low by steels 4 and 5.

The difference between the empirical and the theoretical value of yield stress is small by steels.

5 NOTCH TOUGHNESS AND STRAIN AGEING

Some data on notch toughness and the change of properties due to strain ageing were allready described and discussed. The accent will be given in this place to the analysis of the effect of microstructure on strain ageing propensity. In **figures 10, 11, and 12** the effect of testing temperature in transition range on notch toughness is shown for as delivered and strain aged steels 2,3, and 4 as well as for the cold deformed steel 2. For the dependence of the temperature by 50% of upper shelf toughness (T_{50}) after strain ageing and the yield stress of as delivered steels (R_E) the following equation was derived from empirical values (19):

$$T_{50} = 27 \text{ Arc sh } (-0.016 (R_E - 410)) - 35 \qquad (7)$$

with Arc sh(t) = ln $(R_E + \sqrt{R_E^2 + 1})$

It is clear from **figures 10, 11, and 12** that quenched and tempered steels are virtually nonpropensive to strain ageing, while steel 2 is very propensive to strain ageing. Therefore, this steel transition temperature and upper shelf toughness are strongly modified by ageing. It is useful to remind on this point that after cold deformation yield stress and tensile strength are increased and uniform elongation strongly, while reduction of area is only slightly diminished, and that after the following ageing annealing tensile properties are only slightly modified. Two conclusions are derived from empirical data:

- to strain ageing are propensive steels with a microstructure of polygonal ferrite and pearlite
- the deteriorating effect of ageing annealing is felt after appearance of brittle fracture as cleavage between (001) planes in ferrite.

It is concluded, finally, that the deteriorating effect of ageing annealing could be, as proposed in **ref. 4**, an interplanar segregation or precipitation of interstitial atoms.

6 CONCLUSIONS

Five structural steels with a microstructure of polygonal ferrite and pearlite, distorted ferrite as well as tempered martensite and yield stress 265 to 1003 MPa were investigated in terms of strengthening mechanisms and strain ageing propensity. The following strengthening mechanisms were considered: pearlite share, interstitial and substitutional solid solution, grain size and precipitation in austenite as well as in ferrite. The following conclusions are proposed:

- in all investigated steels grain size strengthening is predominant with 33 to 51% of the yield stress value. The higher share of grain size strengthening is found by quenched and tempered steels. The second by magnitude is the share of substitutional solid solution strengthening with a share up to 32% of the yield stress. In both ferrite pearlite steels the contribution of pearlite is significant and represents appr. 18% in steel 1. The share of other strengthening mechanisms is lower.
- It is evident from this investigation that the optimal chemical composition of quenched and tempered steels should warrant a small austenite grain size and a fine martensite microstructure after quenching;
- To the contrary of steels with a microstructure of polygonal ferrite and pearlite quenched and tempered steels are virtually no propensive to strain ageing in terms of lower notch toughness and higher transition temperature. Most of the properties are modified by 10% of cold deformation, yield stress and tensile strength increased, uniform elongation strongly and reduction of area slightly decreased. The effect of the following ageing annealing is minor;
- It is proposed that the strain ageing mechanism involves a platelike precipitation or interlayer segregation, which would promote the brittle crack propagation by cleavage between dense populated (001) lattice planes.

7 REFERENCES

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