

# Modeling of Nitrogen Solubility in Stainless Steel

Saeed Ghali, Hoda El-Faramawy, Mamdouh Eissa, Michael Mishreky

**Abstract**—Scale-resistant austenitic stainless steel, X45CrNiW 18-9, has been developed, and modified steels produced through partial and total nickel replacement by nitrogen. These modified steels were produced in a 10 kg induction furnace under different nitrogen pressures and were cast into ingots. The produced modified stainless steels were forged, followed by air cooling. The phases of modified stainless steels have been investigated using the Schaeffler diagram, dilatometer, and microstructure observations. Both partial and total replacements of nickel using 0.33-0.50% nitrogen are effective in producing fully austenitic stainless steels. The nitrogen contents were determined and compared with those calculated using the Institute of Metal Science (IMS) equation. The results showed great deviations between the actual nitrogen contents and predicted values through IMS equation. So, an equation has been derived based on chemical composition, pressure, and temperature at 1600 °C:  $[N\%] = 0.0078 + 0.0406 * X$ , where X is a function of chemical composition and nitrogen pressure. The derived equation has been used to calculate the nitrogen content of different steels using published data. The results reveal the difficulty of deriving a general equation for the prediction of nitrogen content covering different steel compositions. So, it is necessary to use a narrow composition range.

**Keywords**—Solubility, nitrogen, stainless steel, Schaeffler.

## I. INTRODUCTION

IN last few decades, nitrogen is added to stainless steels instead of nickel to improve mechanical, physical and corrosion behavior. In addition, nickel is an expensive alloying element and it is interesting and economical to replace it with another cheaper element. The main problem of nitrogen steel is how to introduce nitrogen in molten metal and how to retain it during solidification. The solubility of nitrogen is controlled by types and amounts of alloying elements, temperature of melting as well as nitrogen pressure during casting. Alloying is the easier way of enhancing the nitrogen solubility. By adding elements with a negative interaction parameter and avoiding those of positive sign, the low nitrogen content of an iron melt may be raised by more than two orders of magnitude. In the order of  $Cu < Co < Ni < Al < Si < C < N$  alloying have increasing detrimental effect on nitrogen solubility, while in the order of  $W < Mo < Mn < Cr < Nb < V < Ti$  nitrogen is more and more attracted to the melt. Thus, its activity is lowered and its solubility is raised. Reference [1] found that, Ti and V quite considerably increase the solubility of nitrogen, even with small additions. Cr and Mn promote solubility but not quite as much. Ni acts neutrally while C & Si drastically reduce nitrogen solubility in pure iron.

Nitrogen was found to improve to large extent the properties of steel, especially austenitic stainless steels. References [2]-[4] have revealed that the nitrogen causes an increase in yield

strength. Reference [5] found that ultimate tensile strength of steels was also improved by nitrogen alloying.

Although the basic solid solution hardening mechanism by nitrogen in austenitic stainless steels is still in dispute, some concepts are based on the classical Fleischer theory of solid solution hardening in Face Centered Cube (fcc) materials as illustrated by [6] – [10] according to which the yield strength depends on the nitrogen concentration as shown by:

$$R_o = 1.77 * G * \sqrt{c} * f_o^{3/2} \quad (1)$$

where, C is the concentration of the solute nitrogen atoms (atomic %),  $f_o$  is a parameter to describe the interaction between dislocations and solute atoms as shown by [10], G is shear modulus ( $G = \frac{E}{2(1+\nu)}$ ).

In addition, nitrogen has great effect on grain size of steel which influences the mechanical properties considerably. The yield strength of high nitrogen austenitic stainless steels as a function of grain size is usually expressed by the Hall-Petch equation,  $RP_{0.2} = R_0 + k_y * d^{-0.5}$ .

Reference [11] illustrated that nitrogen and carbon atoms occupy octahedral interstitial sites in the crystal lattice of austenite. The size of octahedral site for fcc lattice has a higher volume than in Body Centered Cube (bcc), while the size of tetrahedral site for bcc lattice has higher volume than in fcc.

Nitrogen dissolution reaction is endothermic in the bcc iron and exothermic in the fcc iron with the values of the solution heat (in J) 34947, -8038 and 25468 in  $\alpha$ ,  $\gamma$  and  $\delta$  phases, respectively. The reaction of dissolution of nitrogen in iron is described as given by [12]:

$$1/2N_2 = N_{Fe} \quad \Delta G = 48400 \text{ J at } 1600 \text{ }^\circ\text{C} \quad [13] \quad (2)$$

$$\log K = - \Delta G^\circ / RT = -1.35 \quad (3)$$

$$K = 0.045$$

$$K = a_N / P_{N_2}^{1/2} = \%N * f_N / P_{N_2}^{1/2} = 0.045 \quad (4)$$

For dilute solution  $a_N = C_N$  &  $f_N = 1$ , i.e. the solubility of nitrogen is equal to about 0.045 % at  $P_{N_2} = 1$  bar

$$\log f_N = e_N^N * \%N + r_N^N * (\%N)^2 + \dots \quad (5)$$

At a concentration range of about 0.045 %, the second term in (5) can be neglected as compared to the first one. The value

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$e_N^N$  is about 0.1 &  $e_N^N \cdot \%N < 0.01$  i.e., the correction for  $f_N$  is less than 1% nitrogen solubility.

$$\%N = 0.045 P_{N_2}^{1/2} \quad (6)$$

The previous relation (6) is known as Sieverts' law. The equilibrium content of nitrogen in steel is dependent on three variables of state: temperature, nitrogen pressure and alloy concentration of the steel. Reference [13] shows that due to the interatomic spacing, the nitrogen solubility is lowest in ferrite as compared to austenite and the melt.

Nitrogen solubility in different phases of pure iron at  $P_{N_2} = 1$  bar is temperature dependence as illustrated by [14] and [15] as given in Fig. 1, the solubility of nitrogen in  $\gamma$ -iron is expected to be decreased with increasing temperature in accordance with Le Chatelier's principle which postulates that any process taking a system out of the equilibrium is prevented. Vice versa, the solubility of nitrogen in  $\alpha$ - and  $\delta$ -iron and that of carbon in all the iron phases increases with increasing temperature because the system stays in equilibrium that way.

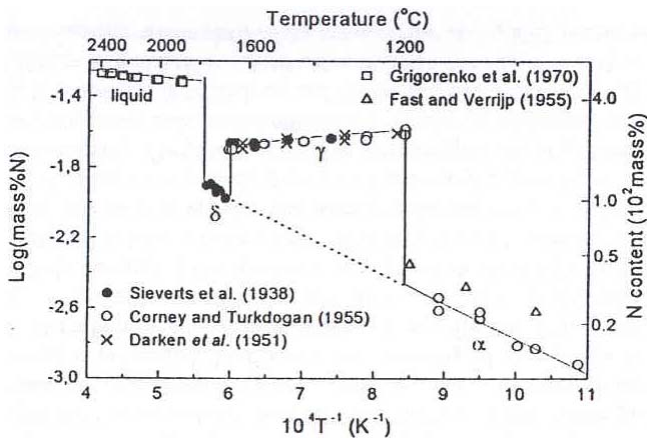


Fig. 1 Solubility of nitrogen in pure iron at  $P_{N_2} = 1$  bar

Sievert's law  $[N\%]_{Fe} = P_{N_2}^{1/2}$  for dissolution of nitrogen in pure liquid iron was confirmed in numerous studies, e.g. [16], [17]. It is not confirmed for multicomponent iron alloys. Reference [18] shows that chromium causes deviations from the linear relation when its content is above 10%.

Reference [15] proposed empirical formula for nitrogen solubility with temperature and pressure.

$$[N\%]_{Fe-Cr} = [\%N]_{Fe} \cdot C_{Fe} \cdot P_{N_2}^{1/2} + [\%N]_{Cr} \cdot C_{Cr}^2 \cdot P_{N_2}^\alpha \quad (7)$$

where  $\alpha = 0.4-0.26C_{Cr}$ ,  $C_i$  is the mass fraction of the  $i$ -th element &  $C_i = [\%i]/100$ .

Sievert's law does not hold if an alloying element has a strong interaction with nitrogen. For the Fe-Cr-Ni-N system with Cr above 10 wt. %, the pressure dependence of nitrogen significantly deviates from Sievert's law as illustrated by [19].

Rashev has derived an equation to predict the nitrogen content in high nitrogen steels [20].

$$\log \frac{[N\%]}{(5.1 - [N\%])} = \frac{-188}{T} - 1.95757 - \left[ \frac{3280}{T} - 0.75 \right] \log F_N \Sigma + \log (P_{N_2})^{1/2} \quad (8)$$

where:  $[N\%]$  is equilibrium nitrogen concentration in mass percent,  $T$  is temperature of the melt, (K);  $P_{N_2}$  is nitrogen partial pressure (bar);  $\log F_N \Sigma = \sum e_N^i [i\%] + \sum r_N^i [i\%]$ ;  $[i\%]$ : concentration of the element ( $i$ ) in the molten stainless steel.

$$\log F_N \Sigma = e_N^C [C\%] + e_N^{Si} [Si\%] + e_N^{Cr} [Cr\%] + r_N^{Cr} [Cr\%]^2 + e_N^{Mn} [Mn\%] + e_N^{Mo} [Mo\%] + e_N^V [V\%] + e_N^W [W\%] + e_N^{Ni} [Ni\%].$$

where:  $e_N^C = 0.13$ ,  $e_N^{Si} = 0.049$ ,  $e_N^{Mn} = -0.02$ ,  $e_N^{Cr} = -0.048$ ,  $r_N^{Cr} = 0.0005$ ,  $e_N^V = -0.1$ ,  $e_N^{Mo} = -0.011$ ,  $e_N^W = -0.0015$ ,  $e_N^{Ni} = 0.01$ .

The present work is a part of complex research study aiming to develop a modified scale resistance austenitic stainless steel through partial and total replacement of nickel by nitrogen.

The present study concerns with investigation of the solubility behavior of nitrogen in the austenitic stainless steels which contains carbon from 0.3% to 0.4%, and the validity of the published models in prediction of nitrogen content in these modified steels.

## II. EXPERIMENTAL

Eight stainless steels with different nickel and nitrogen contents were melted in 10 kg induction furnace under different nitrogen pressures as given in Table I. The reference steel was melted in induction furnace in open air. The ferrochromium nitrogen alloy was the source of nitrogen. It was added after complete melting under nitrogen pressure. Molten metal was cast into refractory sand mold at 1600 °C under nitrogen pressure. The ingots produced were recharged into reheating furnace. The forging process was started at 1150 °C by using load of one-ton free forging (dieing). The finishing temperature was adjusted to 900 °C. The forging process was followed by air cooling. The Kjeldahl method was used for determination of total nitrogen content in the produced stainless steels. The dilatometer 805A/D by Boher. Thermo-analyze was used to estimate the phase transformation in the samples. Dilatometric specimen was cylindrical in shape with 5 mm diameter and 10 mm length. The specimens were heated to 1200 °C. The heating rate from room temperature to 425 °C was 10 °C/min, from 425 °C to 950 °C was 60 °C/min to prevent carbide formation and finally from 950 °C up to 1200 °C was 1 °C/min to allow structure to be homogenized. Samples were prepared to carry out microscopic examination.

## III. RESULTS AND DISCUSSIONS

The aim of this work was to investigate the behavior of nitrogen in stainless steels, and illustrate the influence of partial & total replacement of nickel by nitrogen on the microstructure. Eight stainless steels were designed to cover partial and complete replacement of nickel by nitrogen. The chemical compositions of stainless steels produced are given in Table I.

TABLE I  
CHEMICAL COMPOSITION OF FORGED STAINLESS STEELS

Steel No.	C%	Si%	Mn%	Cr%	W%	Ni%	N%	Pressure, bar
1*	0.42	3.20	1.14	18.10	1.20	8.56	0.007	Open air
2	0.30	3.07	1.61	19.33	1.17	0.17	0.430	18
3	0.35	2.74	1.27	19.39	1.20	5.13	0.330	15
4	0.40	2.45	1.16	17.80	1.14	0.13	0.501	26
5	0.41	2.42	1.22	20.33	1.22	7.06	0.250	10
6	0.39	2.77	1.16	18.60	1.23	3.71	0.402	20
7	0.33	2.63	1.21	19.33	1.14	7.09	0.360	17
8	0.35	2.69	1.30	16.33	1.13	4.13	0.490	34

As it is well known, at constant melting temperature, nitrogen solubility in steel is affected by the alloying elements and their concentration as well as the nitrogen pressure at which the heat is carried out. According to IMS in Bulgaria, the nitrogen content in alloy steel melted at high nitrogen pressure can be calculated by using (8).

The nitrogen content of the seven investigated nitrogen contained stainless steel samples were determined and compared with those calculated, using the IMS equation (8), in Fig. 2. It is clear from the figure that there are great differences between the actual and predicted nitrogen contents through using IMS equation. Mathematical treatment of the practical results at 1600 °C and (8) has led to deduction of (9) for prediction of nitrogen content based on chemical composition of stainless steels and nitrogen pressure:

$$[N\%] = 0.0078 + 0.0406 * X \quad (9)$$

where

$$X = [(200 * [Mn\%] + 110 * [Mo\%] + 480 * [Cr\%] + 15 * [W\%]) / (1300 * [C\%] + 490 * [Si\%] + 100 * [Ni\%] + 5 * [Cr\%]^2)] * (P_{N_2})^{1/2}$$

Fig. 3 illustrates the difference between the actual and calculated nitrogen contents based on author's equation (9). Predicted nitrogen content mainly depends on chemical composition of investigated stainless steels, nitrogen pressure of melting and casting processes as given in (9). Figs. 2 and 3 show that the applicability of (9) is more agreement with the experimental results (nitrogen content) than that of (8). It is clear that the IMS formula was rather less accurate over the chemical composition and nitrogen pressure investigated.

By applying (8) and (9) on the results obtained by [21] on stainless steel (0.04%C, 22.35%Cr, 0.9%Mn and 0.36%Si) at 1600 °C and 1800 °C, it was found that there is a deviation of both equations as shown in Figs. 4 and 5. The deviation is more pronounced at 1600 °C (Fig. 4).

Equation (9) has been derived using experimental results of high carbon stainless steels ( $\approx 0.4\%C$ ) which may explain the lower nitrogen content predicted using this equation compared to the actual nitrogen content of low carbon stainless steel (0.04%C). Carbon reduces the nitrogen solubility and it is expected that the nitrogen content of high carbon steel is less than that of lower carbon steel.

However, Fig. 5 shows less deviation of the predicted nitrogen content compared to the actual one. This may be

explained by the fact that increasing the temperature increases the diffusion of nitrogen to the outer atmosphere (desorption). For higher rate of diffusion, the nitrogen content at 1800 °C is lower than that at 1600 °C. This is the cause of smaller deviation between the actual and predicted nitrogen content calculated by IMS and the authors' equations at 1800 °C. Further comparison of the predicted nitrogen contents by using (8), (9) and experimental results of [22] on stainless steel containing 0.003%C, 3%Mo, 5%Mn, 10%Ni and 18%Cr is given in Fig. 6, which illustrates that there is a great deviation of both equations from the experimental data which could be attributed to the very low carbon content (0.003%C) of the used stainless steel.

From the above-mentioned results, it could be concluded that it is very difficult to derive general equation for the prediction of nitrogen content covering different steel compositions. So, to overcome this difficulty, it has been necessary to use narrow composition range.

The phases of stainless steels can be estimated using Schaeffler diagram. Phases of stainless steels can be predicted by using chromium and nickel equivalents of stainless steels by using Schaeffler diagram [23]. The chromium and nickel equivalents of designed stainless steels can be calculated according to (10) and (11):

$$Cr_{\text{equivalent}} = Cr + 1.5 * Mo + 1.5 * W + 0.48 * Si + 2.3 * V + 1.7 * Nb + 2.5 * Al \quad (10)$$

$$Ni_{\text{equivalent}} = Ni + Co + 0.1 * Mn - 0.01 * Mn^2 + 18 * N + 30 * C \quad (11)$$

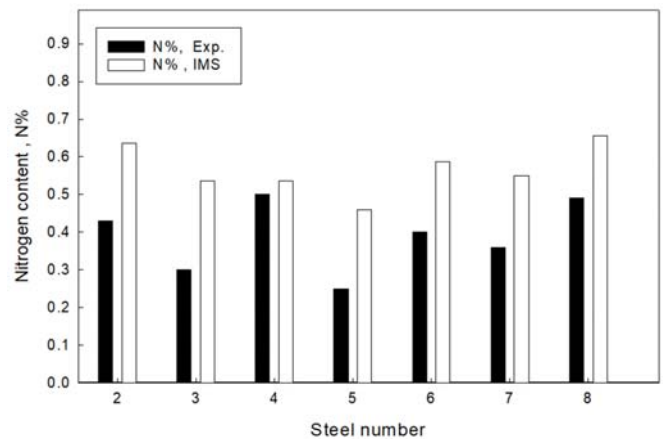


Fig. 2 Experimental nitrogen contents of investigated steels compared to the predicted nitrogen using IMS equation

The estimated chromium and nickel equivalents of the investigated stainless steels are given in Table II.

TABLE II  
CHROMIUM AND NICKEL EQUIVALENTS FOR INVESTIGATED STAINLESS STEELS

Steel number	1	2	3	4	5	6	7	8
Cr-equivalent	21.44	22.45	22.51	20.67	23.32	21.77	22.30	19.32
Ni-equivalent	21.27	17.07	21.55	21.25	23.98	22.70	23.47	23.43

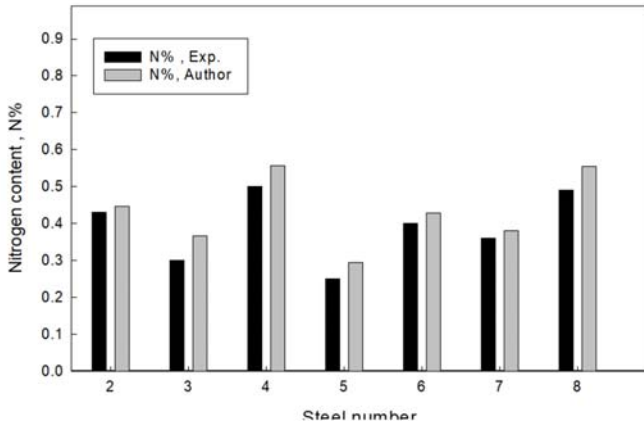


Fig. 3 Extent of nitrogen content deviation from the predicted one through author equation

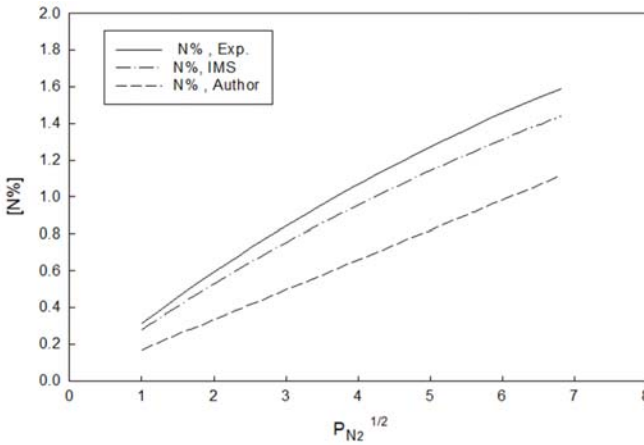


Fig. 4 Actual nitrogen contents of steel (0.04%C, 22.35%Cr, 0.09%Mn, 0.36%Si) compared to the predicted nitrogen using IMS and the author equations at 1600 °C

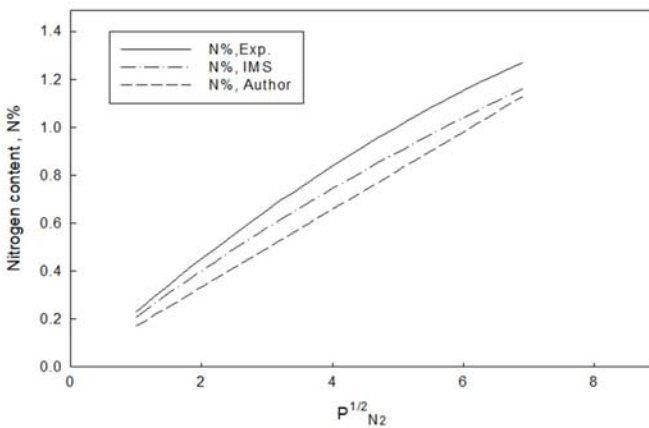


Fig. 5 Actual nitrogen contents of steel (0.04%C, 22.35%Cr, 0.09%Mn, 0.36%Si) compared to the predicted nitrogen using IMS and the author equations at 1800 °C

The investigated stainless steels belong to austenitic region as shown in Fig. 7 according to Schaeffler diagram. Microstructures of forged stainless steels in quenched state are given in Fig. 8. The microstructures of produced stainless steels

show mainly austenitic phase with different grain size depending on the nitrogen content. This indicates that nitrogen has more positive effect than nickel as austenitic stabilizer.

To investigate the phase transformation of developed stainless steels, specimens were heated with specific rate through temperature intervals up to 1200 °C as mentioned in the experimental section. The samples were held for 60 min at 1200 °C, then normally air cooled. Dilatograms were plotted illustrating the change in specimen length as a function of temperature for different investigated stainless steels. Fig. 9 shows the dilatograms of produced stainless steels. It is clear that through heating and cooling cycles, there is no definite transformation that can be identified. Mean thermal expansion coefficient can be calculated by using equation:

$$\text{Mean Thermal Expansion Coefficient} = (dL/dT) * L \quad (12)$$

dL: change in length; L: length of the sample; dT: change in temperature.

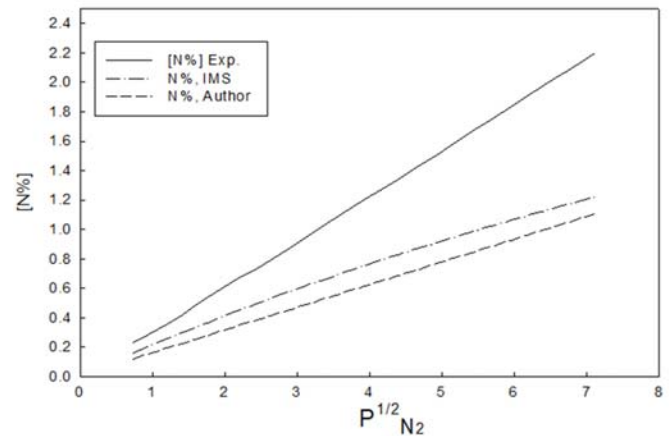


Fig. 6 The extent of deviation of nitrogen for 0.003%C, 3%Mo, 5%Mn, 10%Ni & 18%Cr steel from nitrogen predicted by IMS & author equation at 1600 °C

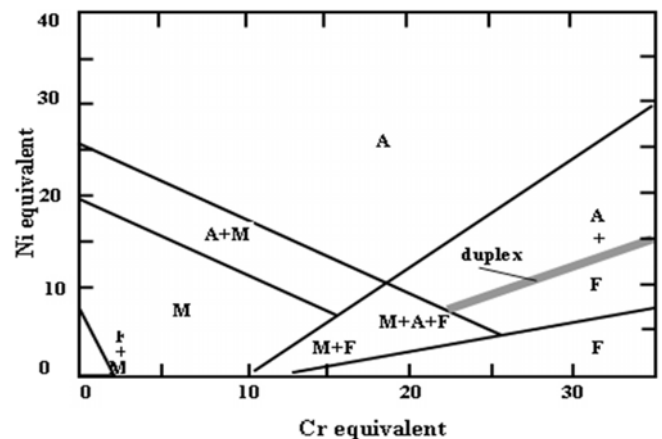


Fig. 7 Schaeffler diagram, with (mass %), A = austenite, M = martensite, F = ferrite

The results of the mean thermal expansion coefficient are given in Table III. The results show that there is regularity in

thermal expansion coefficients. The mean thermal expansion coefficient is mainly affected by chemical composition of stainless steels.

From the results, it is noted that the mean thermal expansion coefficient increases by increasing nickel content in steel, while it decreases by increasing nitrogen content in steels. Every couple of steels (2, 4), (5, 7) and (6, 8) nearly have the same mean thermal expansion coefficient. This may be attributed to that every couple of stainless steels nearly have the same nickel

content and summation of carbon and nitrogen contents. The change in mean thermal expansion coefficient from (20-100) to (20-700) does not exceed 2.03 in any nitrogen stainless steels, while reference steel (X45CrNiW 18-9) is about 3.1. This means that the produced stainless steels with high nitrogen content have positive effect on mean thermal expansion coefficient. This means that as nitrogen content increases the resistance to phase transformation increases i.e. nitrogen is more austenitic stabilizer than nickel.

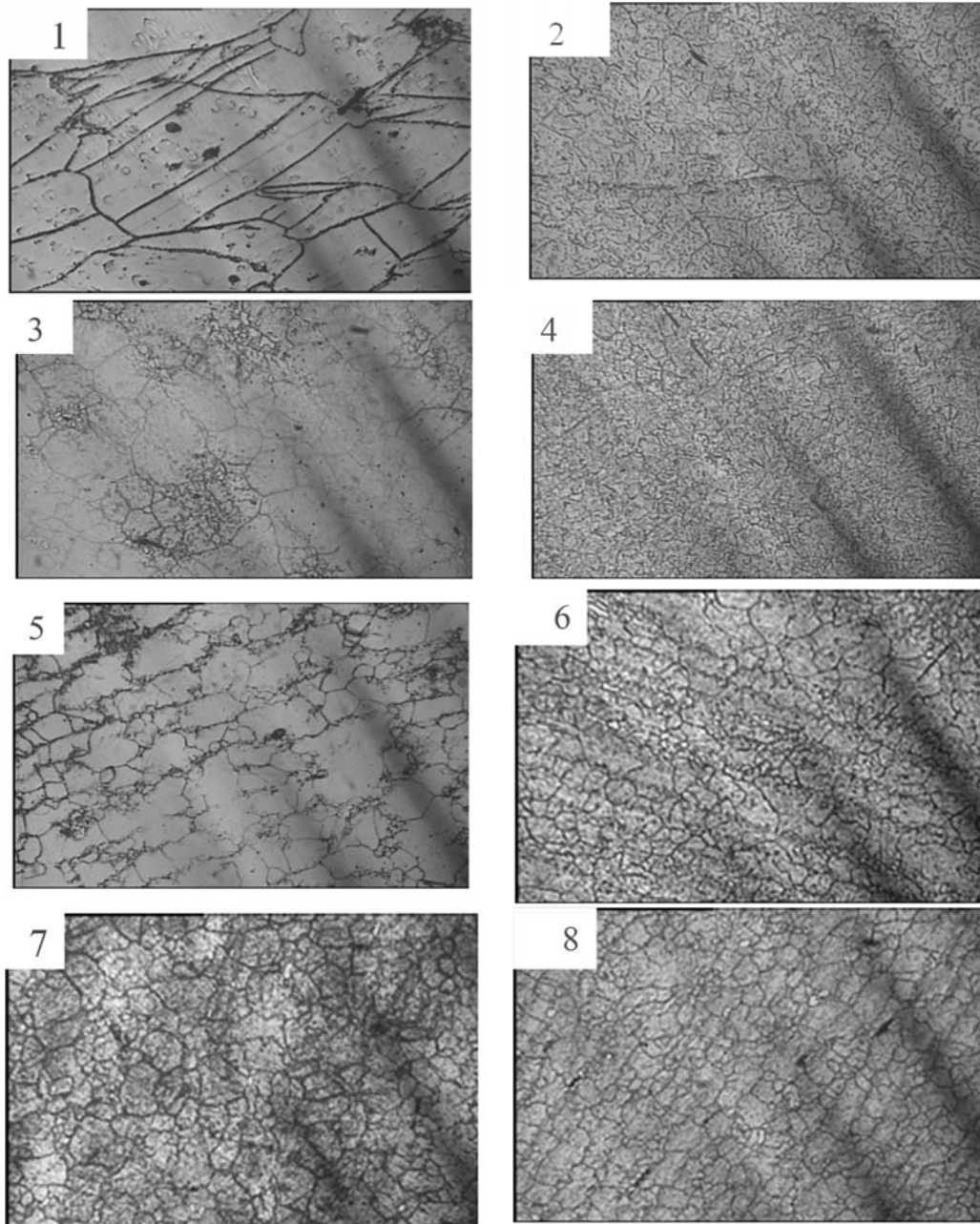


Fig. 8 Microstructures of forged stainless steels as – quenched, 300X

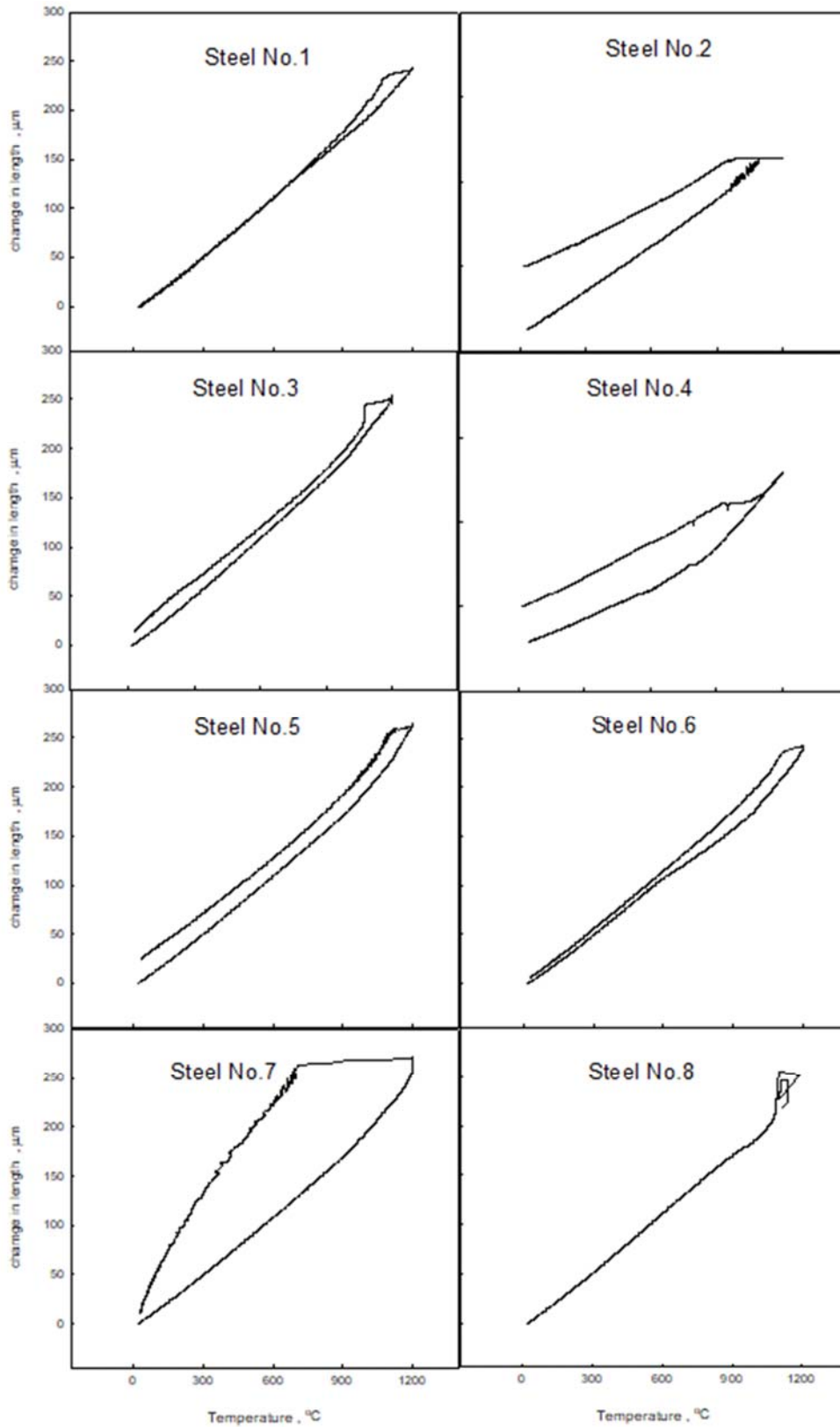


Fig. 9 Dilatograms for investigated stainless steels

TABLE III  
THERMAL EXPANSION COEFFICIENT  $\alpha \times 10^{-6} \text{ (K}^{-1}\text{)}$  AT DIFFERENT TEMPERATURE RANGES, AND CHANGE IN  $\alpha$  FROM THAT AT 20-100 °C TO 20-700 °C (A) AND FROM 20-100 °C TO 20-900 °C (B) OF INVESTIGATED STEELS

Steel No.	Temperature range, °C										A x 10 <sup>-6</sup>	B x 10 <sup>-6</sup>
	20-100	20-200	20-300	20-400	20-500	20-600	20-700	20-800	20-900			
Standard	15.50	---	17.50	---	18.20	---	18.60	---	---	3.1	--	
1	16.70	17.98	18.4	18.50	18.82	19.2	19.35	19.41	19.47	2.65	2.77	
2	11.94	11.98	11.99	12.23	12.63	12.70	12.74	13.17	13.50	0.8	1.56	
3	16.88	16.95	17.69	18.30	18.66	18.89	18.91	19.18	19.39	2.03	2.51	
4	11.25	11.67	12.14	12.63	12.91	12.93	12.79	13.07	13.41	1.54	2.16	
5	17.10	17.61	18.12	18.35	18.69	18.90	19.10	19.32	19.52	2	2.42	
6	16.88	16.95	17.69	18.04	18.45	18.54	18.02	17.89	17.90	1.14	1.02	
7	17.50	17.22	17.50	18.11	18.54	18.62	18.82	19.10	19.31	1.32	1.81	
8	17.56	17.81	18.24	18.71	19.19	19.34	19.43	19.50	19.34	1.87	1.78	

#### IV. CONCLUSIONS

- The solubility of nitrogen in steels depends mainly on type and amount of alloying elements in steels, nitrogen pressure of melting & casting processes and cast temperature.
- In the range of chemical composition of investigated steels and casting at 1600 °C, the nitrogen content can be predicted using the equation:  $[N\%] = 0.0078 + 0.0406 * X$ , where X is function in chemical composition and nitrogen pressure.
- $X = [(200 * [Mn\%] + 110 * [Mo\%] + 480 * [Cr\%] + 15 * [W\%]) / (1300 * [C\%] + 490 * [Si\%] + 100 * [Ni\%] + 5 * [Cr\%]^2)] * (P_{N_2})^{1/2}$
- It is very difficult to derive a general equation to predict of nitrogen content in steels covering different chemical compositions.
- Both partial and total replacement of nickel using 0.33-0.50% nitrogen, is effective to produce a modified fully austenitic stainless steels X45CrNiW 18-9.

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