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THE APPLICATION OF A FACTORIAL EXPERIMENT WITH REPEATED MEASUREMENTS IN THE INVESTIGATION OF PARAMETERS INFLUENCING THE PROPERTIES OF NIOBIUM MICROALLOYED COLD DRAWN WELDED STEEL TUBES

Summary

It has been observed that inhomogeneous deformations occur at the beginning of the plastic material flow during the cold drawing of welded tubes and after the proportionality limit R_p has been reached. Therefore, it is important to determine the most influential factor at the beginning of the plastic material flow. Tests were carried out on a welded joint and on the base material. The study was performed on two steels with different contents of the microalloying element niobium. Tubes were drawn with two different degrees of reduction. The factorial experiment consisting of three factors at two levels 2^3 with repeated measurements showed that the degree of reduction has the greatest influence on the proportionality limit.

Key words: *niobium microalloyed steel, cold drawn tubes, proportionality limit, factorial experiment*

1. Introduction

It is well known that a small addition of a microalloying element with a proper selection of hot rolling parameters results in a significant improvement in the mechanical and technological properties of a rolled product [1-3]. The optimal addition of the microalloying element niobium is about 0.05% [4]. During hot deformation, niobium in combination with carbon or nitrogen produces solid compounds of niobium nitrides, niobium carbides or niobium carbonitrides. The chemical composition and size of the precipitates are dependent on the deformation temperature and the niobium content in the steel. Precipitates are produced in the form of densely arranged carbonitrides and make a strong obstacle to the dislocation movement [2,3]. In a hot rolled strip, 10 nm precipitates are in interaction with dislocations. Therefore, recent research indicates that during the subsequent cold deformation, niobium microalloyed steels have different behaviour from the steel with the same or similar basic chemical composition but made without niobium.

Tests have shown that the subsequent cold deformation results in significant hardening [5]. Inhomogeneous deformations at the beginning of the plastic material flow were observed during the cold drawing of niobium microalloyed steel tubes [6,7]. These inhomogeneous deformations are associated with the appearance and propagation of Lüders bands. This phenomenon was observed in the 1950s [8, 9], but it could not be fully explained at the time. Today, this phenomenon is being intensively investigated [10-15] with the development of modern testing methods. Lüders bands appear after the proportionality limit has been reached, and therefore it is important to study which factors affect the proportionality limit. Cold drawn welded tubes are drawn from cold rolled longitudinally welded tubes. The material is heated to a temperature of about 1,400°C during a high frequency induction welding process. It is possible that small deformation induced precipitates of niobium, present in the niobium microalloyed steel, change their distribution and size at this temperature [16]. This can affect the properties of tubes on the welded joint and on the base material. It has been found that the coarsening of niobium precipitates occurs during strip heating. Heated steels contain precipitates of a size larger than 40 nm. In these steels, Lüders bands do not appear during the subsequent cold deformation [17].

Therefore, it is necessary to determine the most influential factor at the beginning of the plastic material flow, R_p. A factorial experiment consisting of three factors at two levels 2^3 with repeated measurements was used [18-20] to determine the most influential parameter. This methodology of planning and analysing the experiment is used to analyse the effects of certain parameters on the output variables of technological and production processes. The factorial experiment is used in order to reduce the number of experiments and to get as much new information on the amount and value as possible. There are one-factor and multi-factor experiments.

$$K^r \cdot n = N \tag{1}$$

where K is the number of levels to be applied in the study of each factor; r is the number of factors observed in the experiment; n is the number of experiments repeated under the same conditions; N is the total number of experiments [18].

The most common experiment used in practice is the factorial experiment consisting of three factors at two levels 2^3 with one repeated run.

2. Experimental part

The tests were carried out on two steel tubes: the first tube was made of steel containing an addition of 0.046% Nb and the second was made of steel containing an addition of 0.053% Nb. The chemical composition of the steels is shown in Table 1.

Table 1 Chemical composition of steel (wt. %)

Steel	C	Mn	Si	S	P	Nb	N
1	0.12	0.81	0.12	0.016	0.011	0.046	0.0080
2	0.14	0.82	0.13	0.015	0.011	0.053	0.0081

A 370x3 mm hot rolled strip was rolled out from both grades of steel. Precisely welded tubes with the dimensions of 57x3 mm were rolled out from strips containing 0.046% Nb and 0.053% Nb. The tubes were high frequency welded at a welding speed of 40 mm/min and current intensity of 13.5 A. From the 57x3 mm tube two dimensions of both steels were obtained during cold drawing (without mandrel): 43x3 mm and 50x3 mm. The achieved reduction degrees during cold drawing of tubes were calculated by the following equation and are shown in Table 2.

$$\varepsilon = \left[1 - \frac{s_1(D_1 - s_1)}{s_0(D_0 - s_0)}\right] \cdot 100 \quad (\%) \tag{2}$$

where D_0 is the initial tube diameter; D_1 is the final tube diameter; s_0 is the initial wall thickness of the tube; s_1 is the final wall thickness of the tube.

Standard metallographic preparation of the samples was performed. The samples were ground with abrasive papers of different gradations during constant water cooling. After grinding, polishing was carried out with the addition of alumina (Al_2O_3), and finally, etching was carried out with nital.

Table 2 Reduction degree of the tube cross section

Steel quality	Initial dimension of tube, mm	Final dimension of tube, mm	Reduction degree of tube cross section, %
0.046% Nb	57x3.00	43x3.0	25.93
		50x3.0	12.96
0.053% Nb	57x3.00	43x3.0	25.93
		50x3.0	12.96

3. Results and discussion

Mechanical tests were carried out on all cold drawn tubes on the welded joint and on the base material, Table 3.

Table 3 Mechanical properties of tubes

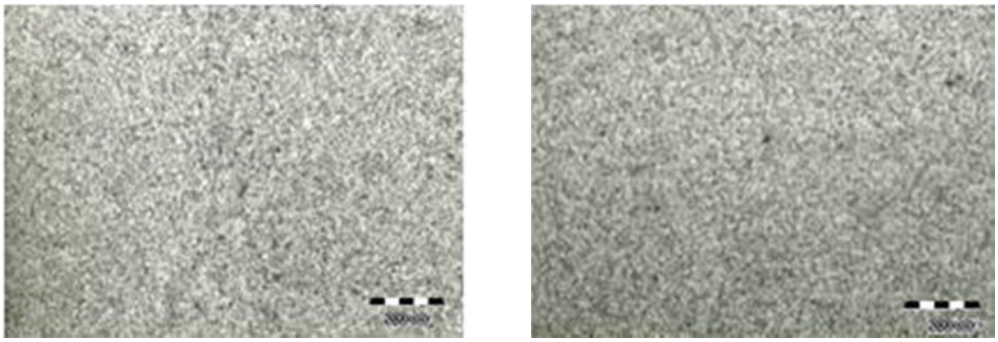
	0.046 % Nb		0.053 % Nb	
	43x3.0 mm	50x3.0 mm	43x3.0 mm	50x3.0 mm
Welded joint	R_p = 617 MPa	R_p = 586 MPa	R_p = 619 MPa	R_p = 560 MPa
Base material	R_p = 625 MPa	R_p = 550 MPa	R_p = 627 MPa	R_p = 562 MPa

All measurements were repeated and the obtained results are shown in Table 4.

Table 4 Mechanical properties of tubes, repeated experiment

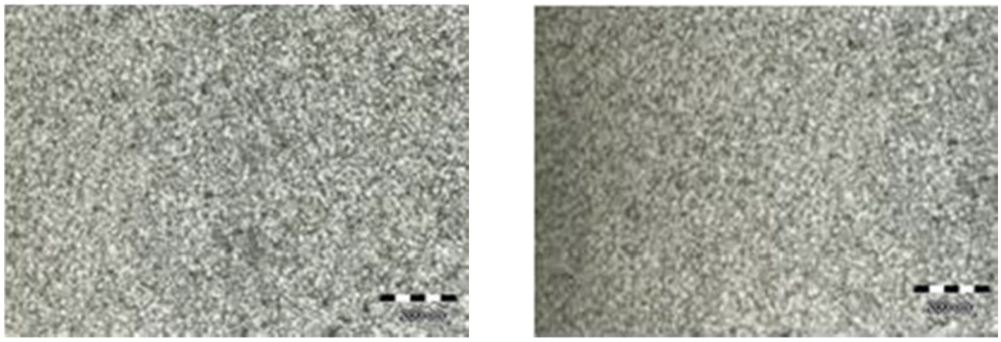
	0.046 % Nb		0.053 % Nb	
	43x3.0 mm	50x3.0 mm	43x3.0 mm	50x3.0 mm
Welded joint	R_p = 620 MPa	R_p = 592 MPa	R_p = 625 MPa	R_p = 561 MPa
Base material	R_p = 626 MPa	R_p = 556 MPa	R_p = 628 MPa	R_p = 565 MPa

The examination of the microstructure was carried out on the welded joint and on the base material of the tube. The obtained results are shown in Figs. 1 and 2.



100x a) 100x b)

Fig. 1 Tube microstructure on: a) the welded joint and b) the base material of 43x3.0 mm tube



100x a) 100x b)

Fig. 2 Tube microstructure on: a) the welded joint and b) the base material of 50x3.0 mm tube

Figs. 1 and 2 show that the tubes have a fine-grained homogeneous ferrite-pearlite microstructure. It has been observed that there is a small difference with a higher concentration of pearlite in the welded joint compared to the microstructure of the base material. However, there is no significant difference between the microstructure of the studied base material and that of the welded joint. The number of experiments, *N* is:

$$N = K^r \cdot \mathrm{n}$$

$$N = 2^3 \cdot 2$$

$$N = 16$$

where *K* is the number of levels; *r* is the number of factors; *n* is the number of experiment repeats.

The number of factors observed in the experiment (*r*) is as follows:

1. Steel containing:	0.046% Nb 0.053% Nb	A
2. Tube diameter:	ϴ 43x3 ϴ 50x3	B
3. Place of testing:	welded joint base material	C

The beginning of the plastic material flow, R_p is measured.

Table 5 Factorial experiment plan in general

	C_1 Welded joint		C_2 Base material	
	B1 ϴ 43x3	B2 ϴ 50x3	B1 ϴ 43x3	B2 ϴ 50x3
A1 0.046% Nb	(1)	(B)	(C)	(BC)
A2 0.053% Nb	(A)	(AB)	(AC)	(ABC)

Table 6 Factorial experiment plan in general, repeat

	C_1 Welded joint		C_2 Base material	
	B1 ϴ 43x3	B2 ϴ 50x3	B1 ϴ 43x3	B2 ϴ 50x3
A1 0.046% Nb	(1)	(B)	(C)	(BC)
A2 0.053% Nb	(A)	(AB)	(AC)	(ABC)

3.1 Preliminary experiments

Preliminary experiments were performed to obtain information on whether experiments could be performed and to determine the number of digits in the experiment (significant digits).

Table 7 Results before the experiments

y_{ijk}	C_1 Welded joint		C_2 Base material	
R_p, MPa 10^2	B1 ϴ 43x3	B2 ϴ 50x3	B1 ϴ 43x3	B2 ϴ 50x3
A1 0.046% Nb		5.86		5.5
		5.92		5.56
A2 0.053% Nb				

Table 8 Mean value calculation

	x	$x-\bar{x}$	$(x-\bar{x})^2$
	5.86	0.15	0.0225
	5.92	0.21	0.0441
	5.5	-0.21	0.0441
	5.56	-0.15	0.0225
$\sum$	12.84	0	0.1332

Mean value:

$$\bar{x} = \frac{\sum x_i}{N} = \frac{22.84}{4} = 5.71 \tag{3}$$

Standard deviation, σ:

$$\sigma = \sqrt{\frac{(x-\bar{x})^2}{N}} = \sqrt{\frac{0.1332}{4}} = 0.1816 = 18.16\% \tag{4}$$

For v = 18.16%, v = 4-40% it is necessary to measure R_p on three significant digits.

Table 9 Results of the experiments

y_{ijk} R_p, MPa	C_1 Welded joint		C_2 Base material	
	B1 Θ 43x3	B2 Θ 50x3	B1 Θ 43x3	B2 Θ 50x3
A1 0.046% Nb	617	586	625	550
	620	592	626	556
A2 0.053% Nb	619	560	627	562
	625	561	628	565

Table 10 Results of the experiments, repeat

y_{ijk} R_p, MPa	C_1 Welded joint		C_2 Base material	
	B1 Θ 43x3	B2 Θ 50x3	B1 Θ 43x3	B2 Θ 50x3
A1 0.046% Nb	617	586	625	550
	620	592	626	556
A2 0.053% Nb	619	560	627	562
	625	561	628	565

Table 11 Calculation of the influential factors

y_{ijk} R_p, MPa 10^2	C_1 Welded joint				C_2 Base material			
	B1 Θ 43x3		B2 Θ 50x3		B1 Θ 43x3		B2 Θ 50x3	
A1 0.046% Nb	6.17 6.20	12.37	5.86 5.92	11.78	6.25 6.26	12.51	5.50 5.56	11.06
A2 0.053% Nb	6.19 6.25	12.44	5.60 5.61	11.21	6.27 6.28	12.55	5.62 5.65	11.27

3.2 Effects

1. Total arithmetic mean:

$$\frac{\sum y_{ijk}}{N}=\frac{\begin{array}{c}6.17+6.20+6.19+6.25+5.86+5.92+5.60+5.61\\+6.25+6.26+6.27+6.28+5.50+5.56+5.62+5.6\end{array}}{16}=\frac{95.18}{16}=5.95 \tag{5}$$

2. Total squared deviation, narrow:

$$\begin{aligned}\text{Narrow} &= \left(\sum y_{ijk}\right)^2-\frac{\left(\sum y_{ijk}\right)^2}{N}=6.17^2+6.20^2+6.19^2+6.25^2\\ &+5.86^2+5.92^2+5.60^2+5.61^2+6.25^2+6.26^2+6.27^2\\ &+6.28^2+5.50^2+5.56^2+5.62^2+5.65^2-\frac{95.18^2}{16}=\\ &=567.18-566.2=1.58\end{aligned} \tag{6}$$

Table 12 Table for processing and analysing the results of the experiment

Source	$\sum y_{ijk}$	I.	II.	III.	Divisor	Effect ∑: Divisor	$\sigma_E{}^2$ $[\,]^2 : N$	Degrees of freedom, S_s	
1	12.37- ↓ ↑ +12.44	24.81- ↓ ↑ +22.99	47.80- ↓ ↑ +47.39	95.19	16	95.19:16=5.95	-	-	
A				0.89	8	0.89:8=0.11	0.89^2:16= 0.05	1	0.05:0.03 =1.67
B	11.78- ↓ ↑ +11.21	25.06- ↓ ↑ +22.33	0.64- ↓ ↑ +0.25	-4.35	8	-4.35:8=-0.54	$(-4.35)^2$:16= 1.18	1	1.18:0.03= 39.33
AB				0.67	8	0.67:8=0.08	$(0.67)^2$:16= 0.03	1	-
C	12.51- ↓ ↑ +12.55	0.07- ↓ ↑ +0.57	1.62- ↓ ↑ +2.73	-0.41	8	-0.41:8=-0.05	$(-0.41)^2$:16= 0.01	1	-
AC				-0.39	8	-0.39:8=-0.05	$(-0.39)^2$:16= 0.01	1	-
BC	11.06- ↓ ↑ +11.27	0.04- ↓ ↑ +0.21	0.50- ↓ ↑ +0.17	-1.11	8	-1.11:8=-0.14	$(-1.11)^2$:16= 0.08	1	0.08:0.03= 2.67
ABC				-0.33	8	-0.33:8=-0.04	$(-0.33)^2$:16= 0.01	1	-
$\sum = 95.18$							$\sum \sigma_E{}^2 = 1.37$		

Narrow: 1.58
-1.37

Residue: 0.21

$$\sigma_0{}^2 = \frac{Residue}{N_1} = \frac{0.21}{8} = 0.03$$

3. Residue:

$$\sigma_0 = Narrow - \sum \sigma_E{}^2 = 1.58 - 1.37 = 0.21 \tag{7}$$

4. $$\sigma_0{}^2 = \frac{Residue}{8} = \frac{0.21}{8} = 0.03 \tag{8}$$

Only values greater than $\sigma_0{}^2 = 0.03$ exert influence. For these values $F_{Calculated}$ is calculated.

5. Calculation $F_{Calculated}$:

$$F_{Calculated} = \frac{\sigma_E{}^2}{\sigma_0{}^2} \tag{9}$$

a) Influence of Nb (A):

$$F_{Calculated} = \frac{\sigma_E{}^2}{\sigma_0{}^2} = \frac{0.05}{0.03} = 1.67$$

b) Influence of the tube diameter (B):

$$F_{Calculated} = \frac{\sigma_E{}^2}{\sigma_0{}^2} = \frac{1.18}{0.03} = 39.33$$

c) Influence of the interaction (BC):

$$F_{Calculated} = \frac{\sigma_E{}^2}{\sigma_0{}^2} = \frac{0.08}{0.03} = 2.67$$

The analysis showed that the deformation degree has the greatest influence on the amount of the proportionality limit.

The niobium content has the lowest influence on the proportionality limit or the beginning of the plastic material flow. Previous research has shown that the niobium content has no influence on the appearance of Lüders bands [17].

4. Conclusion

The conducted investigation has clearly shown that the deformation degree has the greatest influence on the proportionality limit during the cold drawing of niobium microalloyed steel tubes. Therefore, the degree of deformation must be taken into account, especially in the first pass of the tube.

In addition to the degree of deformation, the welded joint mainly affects the proportionality limit. If prior to cold drawing the so-called "zero annealing of tube" is carried out, there is not much difference between the material behaviour on the welded joint and that on the base material.

This investigation has shown that the content of microalloying element niobium exerts the least influence. In further research, it is necessary to investigate in detail the effect of heating the tube prior to cold drawing and the effect of heating the tube between certain passes on the proportionality limit.

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