

INFLUENCE OF CHEMICAL COMPOSITION ON CREEP STRENGTH PARAMETERS OF THE ALLOY IN 713 LC

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VLIV CHEMICKÉHO SLOŽENÍ NA PARAMETRY PEVNOSTI PŘI TEČENÍ SLITINY IN 713 LC

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Abstrakt

V této práci jsou uvedeny výsledky matematicko-statistické analýzy souboru hodnot zkoušek meze pevnosti při tečení vysokolegované niklové slitiny IN 713 LC.

Této analýze byly podrobeny soubory dat zahrnující parametry zkoušek meze pevnosti při tečení spolu s korespondujícími údaji o chemickém složení. Při zkouškách meze pevnosti při tečení byla za přesně definovaných podmínek stanovována doba do lomu t [h], tažnost A_5 [%] a kontrakce Z [%]. K dispozici byly tři soubory taveb o rozdílném chemickém složení. K získání informací o vlastnostech souborů jednotlivých taveb, které lze chápat jako podsoubory, i o vlastnostech společného souboru všech tří taveb o 101 členu, byla využita základní statistická analýza, korelační analýza a vícenásobná regresní analýza.

Bylo zjištěno, že rozptyl chemického složení jednotlivých analyzovaných prvků projevující se v celkovém souboru tří taveb slitiny má statisticky významný vliv na parametry zkoušek meze pevnosti při tečení zkoumané slitiny IN 713 LC.

Abstract

The paper describes results of mathematical-statistical analysis of the set of values of creep strength tests of the high-alloyed nickel alloy IN 713 LC. The analysed set of data comprised parameters of creep strength together with the corresponding data about chemical composition. The following characteristics were determined during creep strength tests under strictly defined conditions: time to rupture t [h], ductility A_5 [%] and contraction Z [%]. Three sets of heats with different chemical composition were available. Basic statistical analysis, correlation analysis and multiple regression analysis were used for obtaining information about properties of data sets from individual heats, which can be interpreted as sub-sets, and about properties of the aggregate set of the whole three heats containing 101 items.

It was ascertained that dispersion of chemical composition of individual analysed elements manifested in the aggregate set if three heats of the alloy has statistically significant influence on parameters of creep strength of the investigated alloy IN 713 LC.

Key words: Ni-based superalloy, creep strength, mathematical-statistical analysis

Introduction

The resulting micro-structure and mechanical properties of nickel based super-alloys are function of their chemical composition and heat treatment, as it is demonstrated e.g. by recent research [1, 2].

The alloy INCONEL 713 LC has been used in the long run for the components of internal-combustion turbines that are subjected to temperature, thermal and stress exposition and dynamic loads [3, 4]. This paper deals with the nickel alloy melted in vacuum, which is used for manufacturing of cast wheels of internal-combustion turbines. According to [3] the guiding chemical composition of the alloy IN 713 LC is the following in [mass %]: 0.05 C, 12.0 Cr, 4.5 Mo, 0.6 Ti, 5.9 Al, 2.0 Nb, 0.1 B, 0.1 Zr and the rest is Ni.

The set of data comprising parameters of creep strength together with the corresponding data about chemical composition was subjected to mathematical-statistical analysis. Three sets of heats were available. They contained the above mentioned data consisting of 23 (heat M87), 19 (heat M50) and 59 (heat Y22) items. Basic statistical analysis, correlation analysis and multiple regression analysis were used for obtaining information about properties of data sets from individual heats, which can be interpreted as sub-sets, and about properties of the aggregate set of the whole three heats containing 101 items.

Research methodology

The set of 101 test bars with nominal diameter of 4.0 mm and nominal measured length of 20.0 mm was subjected to evaluation. The test specimens were cast from three heats with different chemical composition, each specimen was subjected to chemical analysis. These chemical analyses were made in laboratory of the company PRVNÍ BRNĚNSKÁ STROJÍRNA Velká Bíteš, a. s. Chemical composition of statistically analysed nickel alloy in the set of 101 analysed specimens varied from minimum to maximum contents in the following ranges in [mass %]: 0.03 – 0.07 C, 11.4 – 13.0 Cr, 4.3 – 5.2 Mo, 0.69 – 0.85 Ti, 5.6 – 6.5 Al, 2.0 – 2.4 Nb, 0.011 – 0.015 B, 0.08 – 0.15 Zr and the rest was Ni, including other admixtures.

All the creep strength tests were made at the temperature of 870°C and stress of 353 MPa in the laboratory of properties of high-temperature materials of the research institute SVÚM, a. s. Areál VÚ Běchovice, in compliance with the conditions of ZP01-23. The following characteristics were determined during creep strength tests under these conditions: time to rupture t [h], ductility A_5 [%] and contraction Z [%].

There were therefore available always the pairs values of mechanical properties and chemical composition for each test bar for mathematical-statistical analysis. Completed analyses had character of certification tests. The certification creep strength tests and the corresponding chemical analyses served for verification of properties of internal-combustion turbine wheel. This type of test requires a minimum time to rupture of 30 hours at the temperature of 870°C and stress of 353 MPa.

Obtained results and their discussion

Influence of chemical composition within the frame of individual heats

Basic statistical evaluation of individual heats is given in the table 1. This table gives only mean values of the observed parameters, i.e. arithmetic mean \bar{x} , standard deviation s_x , and for each of investigated heats also heat designation and number of creep strength tests n . It is obvious from mutual comparison of data contained in the table 1, that the highest values of mean

times to rupture, ductility and contraction are characteristic for the heat Y 22. Chemical composition of this heat is at the same time characterised by higher contents of carbon, silicon, chromium, molybdenum and lower contents of aluminium, iron, boron, zirconium, phosphorus, sulphur and tantalum, in comparison with two preceding heats (i.e. M87 and M50).

Table 1 Values of parameters of tests and chemical analyses of each of three basic heats

Parameter	Heat M87, n = 23		Heat M50, n = 19		Heat Y22, n = 59	
	x	s _x	x	s _x	x	s _x
time to rupture <i>t</i> [h]	48.007	6.909	47.763	7.527	57.851	7.462
ductility <i>A₅</i> [%]	6.013	1.179	5.322	0.792	6.128	1.091
contraction <i>Z</i> [%]	7.427	2.524	3.941	0.743	8.412	1.860
C [wt%]	0.035	0.005	0.064	0.005	0.068	0.004
Mn [wt%]	<0.05	-	<0.05	-	<0.05	-
Si [wt%]	<0.05	-	<0.05	-	0.054	0.005
Cr [wt%]	12.583	0.215	11.767	0.228	12.609	0.279
Ti [wt%]	0.810	0.023	0.706	0.020	0.754	0.012
Al [wt%]	6.224	0.200	6.351	0.174	5.952	0.164
Fe [wt%]	0.125	0.010	0.213	0.018	0.066	0.017
B [wt%]	0.0148	0.0004	0.0130	0.0001	0.0124	0.0007
Zr [wt%]	0.120	0.009	0.120	0.005	0.081	0.005
P [wt%]	0.0120	0.0017	0.0131	0.0018	0.0100	0.0033
S [wt%]	0.0074	0.0012	0.0106	0.0015	0.0047	0.0094
Nb [wt%]	2.301	0.069	2.147	0.041	2.182	0.065
Ta [wt%]	0.107	0.040	0.284	0.045	0.098	0.052
Mo [wt%]	4.855	0.223	4.628	0.095	4.871	0.216
Co [wt%]	0.153	0.010	0.055	0.021	0.052	0.012
Cu [wt%]	<0.05	-	<0.05	-	<0.05	-
Ni rest [wt%]	72.504	0.529	73.505	0.286	73.086	0.416

In order to verify how important are the differences of concentration of the analysed elements on parameters of creep strength, there were in the first instance determined the correlations coefficients between the test parameters and chemical composition for each of these heats. Determined values of the correlations coefficients were afterwards tested by usual manner due to critical values of correlation coefficients on the reliability level of $\alpha = 0.05$. Tests of statistical significance have ascertained for each of the heats M87, M50 and Y22, that none of correlations coefficients determined for the elements in accordance with the table 1 is statistically significant on the chosen reliability level for time to rupture, ductility and contraction. It is therefore possible to state that dispersion of chemical composition of each of analysed elements, shown in each of three analysed heats, has no statistically significant influence on analysed parameters of the creep strength tests.

Influence of chemical composition within the frame of the aggregate set of three heats

In the next stage we assessed influence of differences of concentrations of analysed elements on parameters of creep strength within the frame of the aggregate set of all three heats. Results of basic statistical computing of data contained in the aggregate set of all three heats M87, M50 and Y22 are given in the table 2. Apart from arithmetic mean x and standard deviation s_x the table 2 comprises moreover the following quantities: variation coefficient in %, minimum and maximum value of investigated parameter, median and eighty percent quantile Q80.

Table 2 Values of parameters of tests and chemical analyses of all three heats

Parameter	Aritmetic mean \bar{x}	Standard deviation s_x	Variation coeff. [%]	Minimum	Maximum	Median	Quantile Q80
time to rupture t [h]	53.71	8.793	16.73	31.00	75.75	53.50	60.00
ductility A_5 [%]	5.950	1.097	18.44	3.81	9.00	5.80	6.73
contraction Z [%]	7.347	2.532	34.46	2.48	15.36	7.38	8.86
C [wt%]	0.060	0.014	23.8	0.03	0.07	0.07	0.07
Mn [wt%]	<0.05	-	-	-	-	-	-
Si [wt%]	0.052	0.004	7.95	0.05	0.06	0.05	0.06
Cr [wt%]	12.44	0.424	3.41	11.36	12.99	12.55	12.83
Ti [wt%]	0.757	0.038	5.01	0.69	0.85	0.75	0.78
Al [wt%]	6.092	0.247	4.05	5.58	6.49	6.06	6.36
Fe [wt%]	0.107	0.060	55.55	0.04	0.22	0.08	0.14
B [wt%]	0.013	0.0011	8.62	0.011	0.015	0.013	0.014
Zr [wt%]	0.097	0.020	20.68	0.08	0.15	0.08	0.12
P [wt%]	0.011	0.0030	27.48	0.004	0.015	0.012	0.013
S [wt%]	0.006	0.0024	39.27	0.004	0.013	0.005	0.008
Nb [wt%]	2.203	0.084	3.83	2.05	2.40	2.18	2.27
Ta [wt%]	0.136	0.087	64.04	0.05	0.31	0.10	0.23
Mo [wt%]	4.812	0.226	4.70	4.25	5.20	4.82	5.07
Co [wt%]	0.073	0.044	59.30	0.05	0.17	0.05	0.15
Cu [wt%]	<0.05	-	-	-	-	-	-
Ni rest [wt%]	73.04	0.536	0.73	71.664	74.304	73.1	73.47

It follows from data contained in the table 2 that variation coefficient if investigated parameters, which is proportion of standard deviation of the relevant parameter divided by its arithmetic mean and multiplied by one hundred, descends in the order of quantities in the following manner: concentration of tantalum, cobalt, iron, sulphur, contraction Z , concentration of phosphorus, zirconium, carbon, ductility A , time to rupture t , concentration of boron, silicon, titanium, molybdenum, aluminium, niobium, chromium and residual nickel in the range of ultimate values from 64.04 % to 0.73 %.

The difference between arithmetic mean and median expresses in the first approximation in what extent the statistical distribution of the relevant quantity differs from normal (Gauss) distribution. The smaller the difference, the closer resemblance of the established statistical distribution to a normal distribution. It follows from comparison of values of arithmetic mean and median of the analysed parameters, given in the table 2, that it is possible to expect higher deviations from normal distribution only in case of iron, zirconium, sulphur, tantalum and cobalt. What concerns the remaining parameters it is possible to expect a normal (Gauss) distribution of values within the frame of the aggregate set of all three heats.

Correlation analysis describing relations between chemical composition and parameters of creep strength gave remarkable results. Coefficients of pairs correlations of elements forming the alloy IN 713 LC determined in relation to the time to rupture t , ductility A and contraction Z are given in the table 3. Positive value of the correlation coefficient indicates, that the given element increases the test parameter, and negative value decreases the test parameter. For $n = 101$ items of the analysed set the number of degrees of freedom is $\nu = 99$, and on the reliability level $\alpha = 0.05$ the critical value of the correlation coefficient is $r_{krit} = 0.19$; on the reliability level $\alpha = 0.01$ the critical value of the correlation coefficient is $r_{krit} = 0.25$ [5]. In the cases when ascertained value of correlation coefficient r of the element in the table 3 is

higher than critical value, it is possible to consider influence of the given element on the test parameter on the chosen level α as statistically significant. For example, if the critical value of the correlation coefficient is $r \geq r_{krit} = 0.25$ for the reliability level $\alpha = 0.01$, it means that we commit a fault of only 1%, if we presume that the given element has either positive or negative statistically significant influence on parameter of creep strength. Statistically significant coefficients of pairs correlations on the reliability level that are equal to or better than 0.01 are printed in bold font in the table 3.

Table 3 Correlation coefficients r of elements of the alloy IN 713 LC in respect to parameters of ultimate strength test at creep: t , A_5 and Z

T	C	Cr	Ti	Al	Fe	B	Zr
1.00	0.45	0.27	-0.04	-0.46	-0.47	-0.38	-0.55
A_5	C	Cr	Ti	Al	Fe	B	Zr
1.00	0.04	0.19	0.20	-0.25	-0.24	-0.06	-0.15
Z	C	Cr	Ti	Al	Fe	B	Zr
1.00	0.06	0.44	0.35	-0.46	-0.62	-0.11	-0.49
t	P	S	Nb	Ta	Mo	Co	
1.00	-0.23	-0.42	-0.12	-0.25	0.16	-0.36	
A_5	P	S	Nb	Ta	Mo	Co	
1.00	-0.14	-0.17	0.20	-0.19	0.19	0.01	
Z	P	S	Nb	Ta	Mo	Co	
1.00	-0.32	-0.56	0.11	-0.57	0.33	0.01	

On the basis of values given in the table 3 it is now possible to make the following conclusions for the set of heats of the alloy IN 713 LC on the reliability level $\alpha = 0.01$:

Time to rupture t is statistically significantly increased by elements C and Cr. The same parameter is at the same time statistically significantly decreased by elements Al, Fe, B, Zr, S, Co and also Ta. Influence of remaining elements, i.e. Ti, P, Nb and Mo, cannot be considered as statistically significant. In case of elements Ti, P and Nb we, however, observe negative influence on time to rupture, in case of Mo this influence is positive.

Ductility A_5 is not increased by any of the elements contained in the table 3 in a statistically significant manner, but in case of elements C, Cr, Ti, Nb and Mo we observe a positive influence on ductility. Ductility is decreased in a statistically significant manner only by Al. Nevertheless, we observe a negative tendency of influence of elements on ductility in case of elements Fe, B, Zr, P, S and Ta. Cobalt - Co has practically no influence at all on ductility.

Contraction Z is increased in a statistically significant manner by elements Cr, Ti and Mo. The same parameter is significantly decreased by elements Al, Fe, Zr, P, S and Ta. Influence of remaining elements cannot be considered as statistically significant. The elements C and Nb have, however, a tendency to increase contraction, the element B has negative influence on this parameter. Cobalt - Co has no influence at all.

It follows from the information given above that carbon, chromium and molybdenum have a positive influence on all parameters of creep strength, while aluminium, iron, boron, zirconium, phosphorus, sulphur and tantalum have negative influence on all parameters of this test.

Mutual correspondence between elements of the alloy in the set of three heats

In order to obtain information about manner of mutual influencing of constitutive elements, impurities and admixture elements we have determined a matrix of correlations coefficients, which is given in the table 4. The set contains $n = 101$ items, to which corresponds

the number of degrees of freedom $\nu = 99$. In order to assess statistical significance of correlation coefficients we have chosen the reliability level $\alpha = 0.05$. Correlation coefficients, which are higher than the critical value ($r_{krit} = 0.19$), are in the table printed in bold font.

Table 4 Matrix of correlation coefficients between elements of the alloy IN 713 LC in the set of three heats

Element	C	Si	Cr	Ti	Al	Fe	B
C	1.00	0.25	-0.04	-0.65	-0.39	-0.27	-0.85
Si		1.00	0.31	0.10	-0.29	-0.30	-0.05
Cr			1.00	0.61	-0.40	-0.72	0.04
Ti				1.00	-0.13	-0.38	0.63
Al					1.00	0.72	0.33
Fe						1.00	0.32
B							1.00
Element	Zr	P	S	Nb	Ta	Mo	Co
C	-0.69	-0.15	-0.30	-0.60	0.11	-0.09	-0.94
Si	-0.35	0.23	-0.25	-0.06	-0.01	-0.11	-0.22
Cr	-0.48	-0.19	-0.58	0.43	-0.66	0.30	0.18
Ti	0.11	0.04	-0.22	0.69	-0.48	0.30	0.77
Al	0.67	0.20	0.63	0.04	0.41	-0.03	0.33
Fe	0.82	0.46	0.88	-0.10	0.80	-0.34	0.16
B	0.67	0.45	0.42	0.57	0.07	0.00	0.88
Zr	1.00	0.44	0.79	0.28	0.55	-0.21	0.61
P		1.00	0.53	0.14	0.57	-0.22	0.18
S			1.00	-0.01	0.79	-0.31	0.24
Nb				1.00	-0.27	0.01	0.63
Ta					1.00	-0.48	-0.18
Mo						1.00	0.12
Co							1.00

It follows from the table 4 that there exists a number of mutual relations, which are statistically significant, between the elements of the alloy IN 713 LC. The most distinct relations are observed in case of sulphur, which has, with the exception of niobium, statistically strongly significant relations to all remaining elements in the alloy. In respect to aluminium, iron, boron, zirconium, phosphorus and tantalum and cobalt these relations are positive, while in respect to carbon, silicon, chromium and titanium these relations are negative. It means that bodies and their zones with higher contents of Al, Fe, B, Zr, P and Ta will be enriched by sulphur, while sulphur will be depleted from bodies and their structural zones with higher concentrations of C, Si, Cr and Ti. It is possible to conclude an analogical consideration for phosphorus and other impurities and admixture elements, such as e.g. boron, iron, zirconium, tantalum and cobalt.

It is also worthwhile to assess relation of elements of the alloy IN 713 LC in respect to nickel, which forms a basis of the assessed alloy, in spite of the fact that its contents was not separately analysed. Its approximate contents in the alloy can be, however, determined as remaining part to 100%, which is the content of Ni rest given in the tables 1 and 2. The values of correlation coefficients in respect to nickel contents determined in this manner are given in the table 5. The pairs correlation coefficients, which are statistically significant on the reliability level $\alpha = 0.05$ are printed in bold font in the table 5.

If we consider the fact that crystallisation of test specimens occurs in such a manner that constitutive and tramp elements have in the alloy IN 713 LC a tendency to liquate into molten mass, it is then natural that correlation coefficients of chromium, titanium, aluminium, niobium, molybdenum and also cobalt have negative values of correlation coefficients, because manufactured and subsequently analysed test specimens will be influenced by this chemical

heterogeneity. Positive and at the same time statistically strongly significant correlation coefficients of carbon and tantalum in respect to nickel cannot, however, be unambiguously well explained in the given case.

Table 5 Values of correlation coefficients of elements in respect to nickel in the set of three heats

Ni 1.00	C 0.47	Si -0.03	Cr -0.65	Ti -0.66	Al -0.32	Fe 0.14	B -0.45
Ni 1.00	Zr -0.15	P -0.05	S 0.05	Nb -0.56	Ta 0.34	Mo -0.55	Co -0.57

Relations between parameters of creep strength and chemical composition

For determination of multiple linear regression between parameters of creep strength and contents of elements in the alloy IN 713 LC we used an absolute value of correlation coefficient as criterion for selection of elements. In order to limit the number of elements in regression relations and to increase reliability of resulting reactions we have chosen for selection of elements the reliability level better than in previous cases, namely $\alpha = 0.001$. To this reliability level corresponds the critical value of correlation coefficient $r_{krit} = 0.32$.

According to the table 3 the following elements have for the time to rupture t an absolute value of correlation coefficient that satisfies the inequality $r \geq 0.32$: C, Al, Fe, B, Zr, S and Co. The time to rupture t was expressed subsequently with use of method of least squares by a linear regression function

$$t[\text{h}] = 82.8 + 317C - 6.54Al - 3.68Fe - 119B - 126Zr + 66.2S + 75.2Co, \quad (1)$$

in which concentration of elements is given in mass percents. Total correlation coefficient has the value $R = 0.59$ and it is strongly statistically significant for the number of degrees of freedom $\nu = n - 8 = 101 - 8 = 93$.

It follows moreover from the table 3 that correlation coefficient of none of elements given in this table corresponds to the reliability level $\alpha = 0.001$ for ductility A_5 . Regression function for ductility A_5 was therefore not determined due to its low reliability. Correlation coefficients of the following elements fulfil the previous condition for contraction Z: Cr, Ti, Al, Fe, Zr, S, Ta and Mo. Contraction Z was afterwards expressed with use of method of least squares by a linear regression function

$$Z[\%] = 11.6 - 1.16Cr + 26.0Ti - 1.48Al + 1.07Fe - 54Zr + 6.14S - 5.57Ta + 1.11Mo, \quad (2)$$

in which concentration of elements is given in mass percents. Total correlation coefficient has the value $R = 0.67$ and it is also strongly statistically significant for the number of degrees of freedom $\nu = n - 9 = 101 - 9 = 92$.

Conclusion

The following main results follow from mathematical-statistical analysis of sub-sets of data from three basic vacuum heats of the alloy IN 713 LC:

1) Parameters of creep strength (870°C, 353 MPa), i.e. time to rupture, ductility and contraction of individual heats are different, which corresponds with different mean chemical composition of these heats.

2) The heat Y 22 is characterised by the longest time to rupture and the highest ductility and contraction. Chemical composition of this heat is also characterised by higher contents of carbon, silicon, chromium, molybdenum, and lower contents of aluminium, iron, boron, zirconium, phosphorus, sulphur and tantalum, in comparison with the both preceding heats (i.e. heats M87 and M50).

3) Dispersion of chemical composition of each of the analysed element, shown within the frame of each of investigated heats, does not have statistically significant influence on analysed parameters of creep strength.

The following main results follow from mathematical-statistical analysis of the aggregate set of data from all three heats of the alloy IN 713 LC:

4) Dispersion of chemical composition of individual analysed elements shown in the aggregate set of three heats of the alloy has statistically significant influence on parameters of creep strength, i.e. on total time to rupture, ductility and contraction.

5) Dispersion of chemical composition of elements at the same time influences in a statistically more significant manner rather contraction and time to rupture than ductility. At evaluation of influences of elements through coefficients of pairs correlation the correlation coefficients of chromium, titanium, aluminium, iron, zirconium, sulphur, tantalum and molybdenum, expressing influence of these elements on contraction, were statistically significant on the reliability level better than $\alpha = 0.001$. Correlation coefficients of carbon, aluminium, iron, boron, zirconium, sulphur and cobalt, expressing influence of these elements on time to rupture, were on the same level of statistical significance. On the other hand correlation coefficients of elements expressing their influence on ductility were statistically significant on the reliability level worse by more than one order ($\alpha = 0.01$).

6) It follows from the facts given above that time to rupture and contraction reacts more sensitively to differences of chemical composition of the alloy IN 713 LC than ductility does. Multiple regression relations (1) and (2) were determined for certain compensation of this influence. They make it possible to modify chemical composition of the alloy in case those parameters of creep strength – particularly time to rupture – do not meet the requirements of certification tests.

7) It is possible to apply the preceding results and conclusions ensuing from them only subject to condition that chemical composition of elements will be within their experimentally verified interval given in the table 2.

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