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The Relationship Between the Solidification Parameters and Chemical Composition of Nickel Superalloy IN-713C

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Abstract

The paper presents the results of studies on the development of correlation of solidification parameters and chemical composition of nickel superalloy IN-713C, which is used i.a. on aircraft engine turbine blades. Previous test results indicate significant differences in solidification parameters of the alloy, especially the temperatures T_{liq} and T_{sol} for each batch of ingots supplied by the manufacturer. Knowledge of such a relationship has important practical significance, because of the ability to asses and correct the temperatures of casting and heat treatment of casts on the basis of chemical composition. Using the statistical analysis it was found that the temperature of the solidification beginning T_{liq} is mostly influenced by the addition of carbon (similar to iron alloys). The additions of Al and Nb have smaller but still significant impact. Other alloying components do not have significant effect on T_{liq}. The temperature T_{eut} is mostly affected by Ni, Ti and Nb. The temperature T_{sol} is not in any direct correlation with the chemical composition, which is consistent with previous research. The temperature T_{sol} depends primarily on the presence of non-metallic inclusions present in feed materials and introduced during the melting and casting processes.

Keywords: Innovative casting materials and technologies, Nickel alloy IN-713C, ATD thermal analysis, Solidification parameters

1. Introduction

The temperature T_{liq}, at which the first solid state crystal are forming, and the temperature of the end of solidification T_{sol} are the most important solidification parameters for a given casting alloy. On the basis of T_{liq}, the optimal pouring temperature can be determined, bearing in mind the required fluidity of molten metal, lowest possible volumetric shrinkage and minimal gas and inclusions solubility. In turn, the temperature T_{sol} provides information about the operational capabilities of casts at elevated temperature and the selection of the heat treatment temperature. Various types of eutectic may form in the alloys solidification range. In case of IN-713C alloy we have the eutectic γ + carbides + intermetallic phases. The results of previously conducted studies indicate significant differences in the solidification parameters of IN-713C alloy, mainly T_{liq}, between different batches of feed ingots ("master heat") supplied by the manufacturer. What causes these differences? It seems appropriate, therefore, to establish an empirical relationship between the solidification parameters and the chemical composition of nickel alloys, based on the supplied certificate and additional analysis of the chemical composition of test casts. Solidification parameters can be easily determined by the ATD thermal analysis. The solidification process is associated with the emission of energy in exothermic effects. These effects can be easily identified by analysing the ATD chart (T=f(t) and dT/dt) as collapses, bends and temperature stops. The short data collection time is a particular advantage of ATD method.

2. Materials and methods of investigation

The tests were conducted using an IN-713C alloy. The samples of feed ingots from different batches were analysed using the ATD method.

Melting was carried out in the vacuum induction furnace Balzers VSG-02 using Al_2O_3 crucibles characterised by high stability of technological parameters which allow to obtain high purity materials. The mass of the charge was about 1,2 kg. During melting the vacuum of 10^{-3} was maintained. Before pouring the furnace chamber was filled with argon. The pouring was carried out in the argon atmosphere at a pressure of 900 hPa.

The test casts were designed as a cylinders with dimensions Ø30×120 mm with a 40×45×17 mm sprue. The temperature measurement point was placed at 1/3 height of the cast (from the bottom). The type S Pt-PtRh10 thermocouple was encased in quartz glass tube. Finished ceramic moulds, made using lost wax process in WSK Rzeszów, are shown on Fig. 1. Fig. 2 shows the mould inside the VIM furnace chamber.

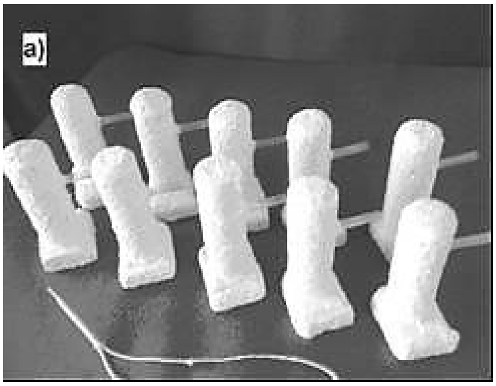
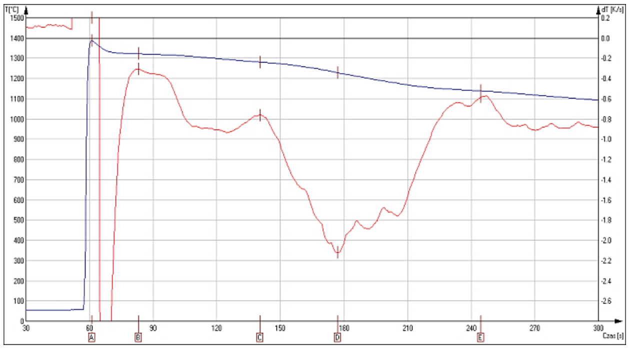


Fig. 1. Ceramic moulds for ATD analysis

Fig. 2. Balzers VSG-02 VIM furnace chamber

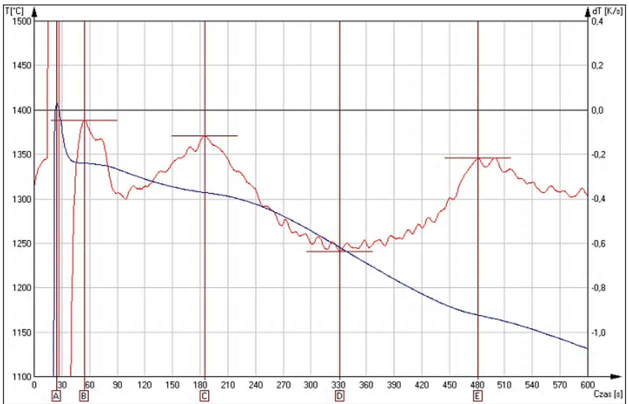
3. The results of investigations and discussion of results

Selected results of ATD analysis of IN-713C alloy samples made from master heat 3V5677/T5 and master heat 7V2124 are shown on Fig. 3 and Fig. 4.



T_{max}	A	61 s	1387°C
T_{liq}	B	83 s	1322°C
T_{Eut}	C	141 s	1281°C
T_{sol}	D	177 s	1228°C
T_{pst}	E	245 s	1144°C

Fig. 3. ATD graph for IN-713C (master heat 3V6577/T5)



T_{max}	A	25 s	1408°C
T_{liq}	B	55 s	1340°C
T_{Eut}	C	185 s	1307°C
T_{sol}	D	322 s	1245°C
T_{pst}	E	482 s	1169°C

Fig. 4. ATD graph for IN-713C (master heat 7V2124)

Nickel superalloys significantly differ in respect of main alloying components. In IN-713C, beside the Ni base, there are additions of C, Cr, Al., Mo and Ti. Therefore it can be assumed that the solidification process is largely dependent on these components. This applies mainly to the temperature of the beginning of the crystallisation T_{liq}.

The solidification parameters of all cast samples were determined using the ATD graphs. Each cast was also subjected to the chemical composition test using optical emission spectrometer Oxford Instruments FOUNDRY-MASTER. The results of ATD and OES tests are shown in Table 1.

Table 1.
Chemical composition and solidification parameters of IN-713C samples

No	Master Heat	Parameters, °C			Content, %							
		T_{liq}	T_{Eut}	T_{sol}	C	Cr	Co	Al	Ti	Nb	Mo	Ni
1	6V5580	1348	1312	1250	0.0439	12.23	0.0494	5.96	0.725	2.18	4.43	74.1
2	M3064 B	1340	1314	1237	0.0596	13.45	0.0673	6.03	0.962	2.48	4.63	71.8
3	M3064A	1333	1307	1237	0.0863	13.70	0.0655	6.03	0.947	2.47	4.59	71.8
4	7V2124	1340	1307	1245	0.0718	13.30	0.1860	6.11	0.954	2.23	4.18	72.8
5	3V4861 B	1343	1305	1262	0.0628	13.20	0.0487	5.98	0.930	2.23	4.40	72.9
6	3V4861A	1342	1305	1272	0.0711	13.20	0.0464	6.07	0.935	2.25	4.31	72.9
7	3V4552	1345	1306	1233	0.0612	12.90	0.1030	6.16	0.920	2.21	4.25	72.8
8	3V4553	1345	1307	1246	0.0584	13.10	0.0720	5.98	0.890	2.25	4.30	73.1
9	M3023	1334	1311	1233	0.0911	13.97	0.0500	5.84	0.942	2.23	4.32	72.8
10	3V5677/T3	1329	1286	1231	0.0895	13.65	0.0500	5.82	0.930	2.38	4.30	73.6
11	3V5677/T4	1328	1285	1204	0.0923	14.02	0.0500	5.84	0.920	2.33	4.31	73.7
12	3V5677/T5	1322	1281	1228	0.1210	14.35	0.0500	5.82	0.920	2.34	4.31	73.6
13	V1542	1324	1283	1239	0.1100	14.25	0.0200	5.70	0.990	2.14	4.12	73.7
14	4V4106	1340	1302	1242	0.0724	13.20	0.0883	6.13	0.915	2.25	4.32	72.7

3. The results of investigations and discussion of results

The evaluation of relationship between the liquidus temperature and selected solidification parameters was conducted by multiple regression statistical analysis.

Statistical significance of $p(\alpha) < 0,05$ was selected for the analysis. Calculated value of the probability p lower than 0.05 means that the given element have a significant influence on the considered characteristic.

The probability value p determines the intensity of influence for given element and the coefficient B sign (negative or positive) determines the direction of influence (reduction or increase). The calculations were performed using licensed Statistica 7.1 software package.

STEP 1		Dependent variable T_{liq}				
R= 0,9928 R^2= 0,9857						
F(8,5)=43,101 p<0,000		Standard error of estimation: 1,6033				
	BETA	St. error BETA	B	St. error. B	t(5)	level p
Variable			1675,984	316,468	5,2959	0,0032
Ni	-0,3194	0,2360	-3,902	2,884	-1,3532	0,2340
C	**-0,8208**	**0,2320**	**-318,658**	**90,081**	**-3,5375**	**0,0166**
Al	0,1846	0,1696	11,079	10,177	1,0885	0,3260
Cr	0,1063	0,3223	1,542	4,678	0,3297	0,7550
Co	-0,1086	0,1053	-22,812	22,124	-1,0311	0,3498
Ti	-0,2485	0,2107	-33,864	28,713	-1,1794	0,2913
Nb	-0,1504	0,1443	-12,198	11,701	-1,0425	0,3450
Mo	-0,2131	0,2590	-12,858	15,626	-0,8229	0,4480

Conclusion:
1. Carbon significantly reduces T_{liq}.
2. Influence of chrome was excluded!
3. The high value of the coefficient of determination (up to 97.57% of the results can be explained using the model)

STEP 2		Dependent variable T_{liq}				
R= 0,9927 R^2= 0,9853						
F(7,6)=57,834 p<0,000		Standard error of estimation: 1,4794				
	BETA	St. error BETA	B	St. error. B	t(6)	level p
Variable			1700,617	283,764	5,9931	0,0010
Ni	-0,3226	0,2176	-3,942	2,659	-1,4827	0,1887
C	**-0,7533**	**0,1006**	**-292,444**	**39,074**	**-7,4845**	**0,0003**
Al	0,1517	0,1265	9,104	7,593	1,1990	0,2757
Co	-0,0990	0,0934	-20,807	19,628	-1,0600	0,3299
Ti	-0,2137	0,1683	-29,128	22,941	-1,2697	0,2512
Nb	-0,1497	0,1331	-12,142	10,796	-1,1247	0,3037
Mo	-0,1973	0,2349	-11,902	14,168	-0,8400	0,4331

Conclusion:
1. Carbon significantly reduces T_{liq}.
2. Influence of molybdenum was excluded!
3. The high value of the coefficient of determination (up to 98,53% of the results can be explained using the model).

STEP 3		Dependent variable T_{liq}				
R= 0,9918 R^2= 0,9836						
F(6,7)=70,312 p<0,000		Standard error of estimation: 1,4480				
	BETA	St. error BETA	B	St. error. B	t(7)	level p
Variable			1496,068	142,601	10,4913	0,0000
Ni	-0,1680	0,1136	-2,053	1,388	-1,4792	0,1826
C	**-0,7233**	**0,0921**	**-280,779**	**35,747**	**-7,8547**	**0,0001**
Al	0,1970	0,1120	11,825	6,721	1,7593	0,1219
Co	-0,0489	0,0703	-10,271	14,777	-0,6950	0,5094
Ti	-0,0962	0,0915	-13,106	12,475	-1,0505	0,3284
Nb	**-0,2450**	**0,0682**	**-19,869**	**5,531**	**-3,5923**	**0,0088**

Conclusion:

1. Carbon and niobium have significant influence on the reduction of T_{liq} (carbon two times stronger).
2. Influence of cobalt was excluded!
3. The high value of the coefficient of determination (up to 98,36% of the results can be explained using the model).

STEP 4	Dependent variable T_{liq}					
R= 0,9912 R^2= 0,9825						
F(5,8)=90,100 p<0,000	Standard error of estimation: 1,4004					
	BETA	St. error BETA	B	St. error. B	t(8)	level p
Variable			1532,867	128,058	11,9701	0,0000
Ni	-0,1876	0,1064	-2,293	1,300	-1,7633	0,1159
C	-0,7290	0,0887	-283,010	34,433	-8,2192	0,0000
Al	0,1477	0,0838	8,866	5,031	1,7623	0,1160
Ti	-0,1120	0,0857	-15,270	11,684	-1,3070	0,2275
Nb	-0,2460	0,0659	-19,952	5,348	-3,7307	0,0058

Conclusion:

1. Carbon and niobium have significant influence on the reduction of T_{liq} (carbon two and a half times stronger).
2. Influence of titanium was excluded!
3. The high value of the coefficient of determination (up to 98,25% of the results can be explained using the model).

STEP 5	Dependent variable T_{liq}					
R= 0,9893 R^2= 0,9788						
F(4,9)=104,01 p<0,000	Standard error of estimation: 1,4545					
	BETA	St. error BETA	B	St. error. B	t(9)	level p
Variable			1422,175	99,758	14,2563	0,0000
Ni	-0,0898	0,0786	-1,097	0,960	-1,1433	0,2824
C	-0,8041	0,0702	-312,161	27,245	-11,4575	0,0000
Al	0,1630	0,0862	9,782	5,174	1,8904	0,0913
Nb	-0,2127	0,0632	-17,250	5,123	-3,3673	0,0083

Conclusion:

1. Carbon and niobium have significant influence on the reduction of T_{liq} (carbon tree times stronger).
2. Influence of nickel was excluded!
3. The high value of the coefficient of determination (up to 97,88% of the results can be explained using the model).

STEP 6	Dependent variable T_{liq}					
R= 0,9878 R^2= 0,9757						
F(3,10)=134,13 p<0,0	Standard error of estimation: 1,4766					
	BETA	St. error BETA	B	St. error. B	t(10)	level p
Variable			1311,979	26,110	50,2479	0,0000
C	-0,7893	0,0700	-306,426	27,187	-11,2709	0,0000
Al	0,2229	0,0695	13,379	4,171	3,2076	0,0094
Nb	-0,1685	0,0507	-13,668	4,115	-3,3217	0,0077

Conclusion:

1. Carbon and niobium have significant influence on the reduction of T_{liq} (carbon over three times stronger),
2. Aluminium increases T_{liq}.
3. The high value of the coefficient of determination (up to 97,88% of the results can be explained using the model).
4. High value of Fisher statistic indicates very good accuracy of the mathematical model.
5. A very small value of the estimation error, only 1,48

The final model is:

$$\mathbf{T_{liq} = 1311.97 - 306.43 * C(\%) - 13.67 * Nb(\%) + 13.36 * Al(\%)}$$

Identical analysis was performed to evaluate the connection between the chemical composition and eutectic temperature T_{Eut} and between the chemical composition and the solidus temperature T_{sol}. The results for the first, fourth and last step are presented below.

For the eutectic temperature T_{Eut}:

STEP 1	Dependent variable: T_{Eut}					
R= 0,9763 R^2= 0,9532						
F(8,5)=12,732 p<0,00618	Standard error of estimation: 4,0588					
	BETA	St. error BETA	B	St. error. B	t(5)	level p
Variable			3412,328	801,145	4,2593	0,0080
Ni	-1,3117	0,4270	-22,424	7,300	-3,0715	0,0277
C	-0,6083	0,4198	-330,401	228,042	-1,4489	0,2070
Al	-0,4468	0,3068	-37,520	25,764	-1,4563	0,2051
Cr	0,2103	0,5831	4,270	11,843	0,3606	0,7331
Co	0,1187	0,1905	34,888	56,008	0,6229	0,5607
Ti	-0,7265	0,3812	-138,537	72,687	-1,9059	0,1150
Nb	-0,3389	0,2610	-38,458	29,621	-1,2983	0,2508
Mo	-0,1891	0,4687	-15,958	39,558	-0,4034	0,7033

Conclusion:

1. Nickel reduces T_{Eut},
2. Influence of chromium was excluded!
3. The high value of the coefficient of determination (up to 95,32% of the results can be explained using the model).

STEP 4	Dependent variable: T_{Eut}					
R= 0,9674 R^2= 0,9360						
F(5,8)=23,408 p<0,00014	Standard error of estimation: 3,7521					
	BETA	St. error BETA	B	St. error. B	t(8)	level p
Variable			3064,665	343,102	8,9322	0,0000
Ni	-1,1233	0,2038	-19,202	3,484	-5,5122	0,0006
C	-0,4298	0,1698	-233,438	92,255	-2,5304	0,0352
Al	-0,2966	0,1605	-24,904	13,480	-1,8475	0,1019
Ti	-0,5060	0,1642	-96,501	31,304	-3,0827	0,0151
Nb	-0,4100	0,1263	-46,523	14,329	-3,2468	0,0118

Conclusion:

1. Nickel, carbon, titanium and niobium reduces T_{Eut} (nickel has the strongest influence).
2. Influence of aluminium was excluded!
3. The high value of the coefficient of determination (up to 93,60% of the results can be explained using the model).

STEP 6	Dependent variable: T_{Eut}					
R= 0,9372 R^2= 0,8784						
F(3,10)=24,096 p<0,00007	Standard error of estimation: 4,6252					
	BETA	level p	B	St. error. B	t(10)	level p
Variable			3008,386	209,026	14,3924	0,0000
Ni	-1,1733	0,1447	-20,058	2,473	-8,1095	0,0000
Ti	-0,7104	0,1254	-135,464	23,916	-5,6641	0,0002
Nb	-0,4566	0,1319	-51,807	14,964	-3,4621	0,0061

Conclusion:
1. Nickel, carbon, titanium and niobium reduces T_{Eut} (nickel has the strongest influence).
2. The high value of the coefficient of determination (up to 87,84% of the results can be explained using the model).
3. High value of Fisher statistic indicates very good accuracy of the mathematical model.
4. A very small value of the estimation error, only 4,63

The final model is:

T_{Eut} = 3008.38 – 20.06*Ni(%) – 135.46*Ti(%)– 51.81*Nb(%)

For the solidus temperature T_{sol}:

STEP 1		Dopondent variable: T_{sol}				
R= 0,7984 R^2= 0,6375						
F(8,5)=1,0993 p<0,48032		Standard error estimation: 15,492				
	BETA	St. error BETA	B	St. error. B	t(5)	level p
Variable			1612,462	3057,970	0,5273	0,6205
Ni	0,0885	1,1886	2,076	27,866	0,0745	0,9435
C	1,2988	1,1685	967,519	870,434	1,1115	0,3169
Al	-0,2398	0,8539	-27,617	98,343	-0,2808	0,7901
Cr	-2,3818	1,6230	-66,339	45,205	-1,4675	0,2022
Co	-0,0707	0,5302	-28,498	213,782	-0,1333	0,8992
Ti	1,1867	1,0609	310,345	277,446	1,1186	0,3142
Nb	-0,7171	0,7266	-111,597	113,064	-0,9870	0,3690
Mo	0,8551	1,3044	98,988	150,993	0,6556	0,5411

Conclusion:
1. None of the components has any significant impact on the temperature T_{sol},
2. Influence of nickel was excluded.
3. The low value of the coefficient of determination means that only 63,75% of the results can be explained using the model.

STEP 4		Dopondent variable: T_{sol}				
R= 0,7766 R^2= 0,6032						
F(5,8)=2,432 p<0,1266 E		Standard error of esimation: 12,816				
	BETA	St. error BETA	B	St. error. B	t(8)	level p
Variable			1516,577	240,343	6,3101	0,0002
C	1,1586	0,8730	863,096	650,304	1,3272	0,2211
Cr	-1,8278	0,9735	-50,910	27,116	-1,8775	0,0973
Ti	0,8789	0,3959	229,856	103,529	2,2202	0,0572
Nb	-0,8211	0,4510	-127,775	70,185	-1,8205	0,1062
Mo	0,8397	0,4845	97,202	56,080	1,7333	0,1213

Conclusion:
1. None of the components has any significant impact on the temperature T_{sol},
2. Influence of carbon was excluded.
3. The low value of the coefficient of determination means that only 60,32% of the results can be explained using the model.

STEP 8		Dopondent variable: T_{sol}				
R= 0,5314 R^2= 0,2853						
F(3,10)=4,7906 p<0,0491		Standard error of estimation: 14,042				
	BETA	St. error BETA	B	St. error. B	t(12)	level p
Variable			1440,268	91,60883	15,72193	0,000000
Cr	-0,5341	0,2440	-14,878	6,797	-2,1887	0,0491

Conclusion:
1. Only chromium reduces the solidus temperature T_{sol},
2. The very low value of the coefficient of determination means that only 28,53% of the results can be explained using the model.

The final model is:

$$T_{sol} = 1440.27 - 14.88*Cr(\%)$$

Very low values of the coefficient of determination and Fisher statistic indicate poor accuracy of the model. Because of this the model cannot be accepted as correct representation of reality. This results are confirmed by previous studies on the assessment of metallurgical quality of feed ingots [4-6]. Many impurities can be introduced to the alloy during melting. This impurities can originate from:

- contaminated feed materials,
- ceramic material of the crucible,
- contaminated furnace atmosphere (ex. with oxygen),
- products of reaction between the melt and the mould material, especially when pouring temperature is high.

Refining processes are impossible because of the vacuum requirement (closed furnace chamber) for melting the nickel superalloys. Thus any of the aforementioned factors can lead to contamination of the melt by intermetallics or gases. These phenomena can consequently lead to shrinkage porosity and non-metallic inclusions, in particular at the grain boundaries.

Most non-metallic impurities are characterized by low pour point, which causes their accumulation on the front of solidification as they crystallize last. Thus the temperature of the end of solidification is significantly reduced, in the case of contamination of the melt, irrespective of the influence of the main alloying elements.

4. Conclusions

Based on the research and the statistical evaluation of the obtained results it can be concluded that:

1. The temperature T_{liq} of the IN-713C alloy is influenced by the additions of carbon and niobium (reduction) and aluminium (increase). The final relationship is:

$$T_{liq} = 1311.97 - 306.43*C(\%) - 13.67*Nb(\%) + 13.36*Al(\%)$$

2. The eutectic temperature T_{Eut} of the IN-713C alloy is influenced by the additions of nickel, titanium and niobium, which reduce T_{Eut} (nickel the strongest). The final relationship is:

$$T_{Eut} = 3008.38 - 20.06*Ni(\%) - 135.46*Ti(\%) - 51.81*Nb(\%)$$

3. Only chromium has any impact on the temperature T_{sol} (reduction). Because of very low values of the coefficient of determination and Fisher statistic only 28,53% of results can be explained using the model:

$$T_{sol} = 1440.27 - 14.88*Cr(\%)$$

The model cannot be accepted as correct representation of reality. This results are confirmed by previous studies on the assessment of metallurgical quality of feed ingots.

4. It is possible to create the empirical relationship between the T_{liq}, T_{Eut} and the alloying elements of studied alloys.

5. The studied alloys are presently poured in temperature 1500 to 1520°C. On the basis of ATD analysis it can be concluded that this is too high. Pouring temperature should be between 1460 to 1480°C. However the liquid metal fluidity in case of thin walled castings of aircraft turbine blades should be taken into consideration.

6. In case of high content of impurities the temperature T_{sol} for IN-713C alloy was about 1235°C. This can lead to melting of the low melting point eutectic during heat treatment which can cause additional porosity.

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