STUDY OF NICKEL-BASED SUPERALLOY CHEMICAL MICROHETEROGENEITY

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STUDIUM CHEMICKÉ MIKROHETEROGENITY NIKLOVÉ SUPERSLITINY

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Abstrakt

Slitiny na bázi niklu jsou během krystalizace, obdobně jako uhlíkové i slitinové oceli, charakterizovány intenzívním dendritickým odmíšením konstitutivních i doprovodných prvků a též příměsí. Pochod dendritického odměšování i jeho konečný stav, který určuje dendritickou heterogenitu uvažované niklové slitiny, lze přitom z fyzikálně-metalurgického hlediska popsat stejnými modely jako je tomu u ocelí. Avšak na rozdíl od ocelí, jejichž odměšování a výsledná dendritická heterogenita jsou již dlouhou dobu soustavně sledovány [1] a je pro jejich popis k dispozici poměrně rozsáhlý soubor konkrétních údajů (rozdělovacích koeficientů odměšujících prvků, indexů heterogenity i vztahů mezi heterogenitou a vlastnostmi příslušné slitiny), pro slitiny na bázi niklu je těchto dat neporovnatelně méně [2].

Cílem této práce bylo provést studium chemické mikroheterogenity niklové superslitiny IN 738 LC v litém stavu a po dlouhodobém žíhání při teplotě 850°C. K dosažení tohoto cíle byla aplikována původní metoda měření dendritické mikroheterogenity prvků na danou slitinu na bázi niklu a byly stanoveny základní statistické parametry heterogenity vybraných prvků slitiny a také byly stanoveny efektivní rozdělovací koeficienty jak pro vzorek v litém stavu, tak pro vzorky dlouhodobě žíhané.

Abstract

Nickel based alloys are characterised during crystallisation, similarly as carbon and alloyed steels, by an intensive dendritic segregation of constitutive elements and impurities, as well as admixtures. The process of dendritic segregation and its final state, which determines dendritic heterogeneity of the alloy under consideration, can be, nevertheless, described from the physical-metallurgical point of view with use of the same models that are used for steels. However, contrary to steels, segregation of which and final dendritic heterogeneity of which have been systematically investigated already for quite a long time [1] and we have thus at our disposal a comparatively large set of concrete data (partition coefficients of segregation elements, heterogeneity indexes and relationships between heterogeneity and properties of the relevant alloy), the number of such data for nickel based alloys is incomparably smaller [2].

This work was aimed at investigation of chemical micro-heterogeneity of the nickel super-alloy IN 738 LC as cast and after long-term annealing at the temperature of 850°C. In order to achieve this goal we have used the original method of measurement of dendritic micro-heterogeneity of elements for the given nickel based alloy, and we have determined basic statistic parameters of heterogeneity of selected elements of the alloy. We have also determined effective partition coefficients both for the sample as cast and for the samples after long-term annealing.

Key words: Ni-based superalloy, microsegregation, partition coefficients

Introduction

Modern metallic alloys generally have complex microstructures and may contain a large number of alloying elements. Even if the basic metallurgy is understood, the human brain is unable to explicitly visualise the role of the many interacting variables that contribute to the characteristics of the alloy. The important major components of commercial Ni-base superalloys are: Ni, Co, Cr, Mo, W, Al, Ti, Nb, Ta, Fe, Hf, Zr, Re, B and C. Of these 15 components, 10 components (Ni, Co, Cr, Mo, W, Al, Ti, Nb, Ta and Fe) are at least necessary for an adequate description of superalloys [3]. However, for a more complete treatment a 15 component data base is desirable. The number of constituent binary and ternary subsystems for such a 10 or 15 component data base is enormous and only a fraction of these subsystems have been in most cases realised for equilibrium conditions of solidification, that is why they can characterise non-equilibrium crystallisation of real alloys only very approximately.

Investigation of behaviour of these superalloys from the viewpoint of segregation of elements during solidification is very important as it has impact on final mechanical properties of metallic alloys. For instance, primary chemical segregation occurs during the solidification of alloys: dendrites grow with concentration gradients, and the final interdendritic medium composition is very different from nominal alloy composition. It has a low melting point, which can cause hot cracking during forging and/or welding [4]. Primary cast structure of alloys, determined particularly by chemical composition, has also influence on strength and creep properties, which are dependent on subsequent heat treatment of castings made of these alloys [2, 5, 6].

The basic value that describes an element segregation process during solidification of alloys is the distribution/partition coefficient. The partition coefficients play an important role in all microsegregation theories thanks to the fact that they define thermodynamic restrictions for element distribution.

For two phases, solid (S) and liquid (L), the equilibrium partition coefficient k_0 of the given element is defined as follows:

$$k_0 = C_s(j) / C_L(j),$$
 (1)

where $C_S(j)$ and $C_L(j)$ are element *j* concentrations, when both these phases are in equilibrium at the given temperature. The partition coefficient k_0 of the alloying element *j* describes the direction and extent of microsegregation during solidification. The closer the value k_0 approaches one, the smaller is element segregation.

Real metallic alloys, as it was already mentioned above, solidify under nonequilibrium conditions, that is why course of solidification of such an alloy can be described with use of equilibrium values only very approximately.

Within the frame of the extensive investigation of chemical and structural heterogeneity of nickel based superalloys, which was accomplished at the Faculty of Metallurgy and Materials Engineering of the VŠB-Technical University of Ostrava, we have among others calculated with use of the original mathematical models [7] the effective partition coefficients k_{ef} of analysed elements of selected nickel based superalloys. These coefficients integrate in them both the impact of segregation during alloy solidification, and also impact of homogenisation – during solidification, as well as during cooling down of the given alloy. Effective partition coefficients have been determined both for the as cast samples – where they express element distribution at alloy crystallisation, and also for the annealed samples – where they express redistribution of the given element between individual phases in structure of solidified annealed alloy.

This work was aimed at investigation of chemical and structural microheterogeneity of the nickel based superalloy IN 738 LC as cast and after long-term annealing at the temperature of 850°C. In order to achieve this goal we have used the original method of measurement of elements dendritic microheterogeneity for the given nickel based alloy, and we have determined basic statistic parameters of heterogeneity of selected elements. We have also determined effective partition coefficients both for the sample as cast and for the samples after long-term annealing.

Experimental material and used methods

We have investigated a set of seven samples of the nickel based superalloy IN 738 LC. One sample was as cast, while the remaining samples were annealed for a graduated time at the temperature of 850°C. Chemical composition of the alloy is given in the Table 1; parameters of annealing are given in the Table 2.

Element	С	Si	Mn	Р	S	*Ag _{max}	Al	*As _{max}	В	*Bi _{max}
Contents	0.10	0.03	0.01	0.002	0.002	5.0	3.35	15.0	0.008	0.5
Element	Со	Cr	Cu _{ma} x	Fe	*Ga _{max}	*Mg _{max}	Мо	*N	Nb	Ni
Contents	8.78	16.2 2	0.01	0.20	20.0	50.0	1.71	15	0.84	basis
Element	*0	*Pb _m	*Sb _m	*Se _{max}	*Sn _{max}	Та	Te _{max}	Ti	*Tl _{max}	W
Contents	16	5.0	2.0	1.0	20.0	1.77	0.5	3.37	0.2	2.63
Element	*Zn _m	Zr								
Contents	0.10	0.04								

Table 1 Chemical composition of the nickel alloy IN 738 LC in [mass %], or in ppm (elements marked with an asterisk *)

Evaluation of dendritic heterogeneity and determination of effective partition coefficients requires sufficiently large concentration sets of investigated elements that were

measured in an optimum way, and in this case in appropriated areas that were in all samples approximately the same.

Tuble 2 Marking of samples and datation of their announing at the temperature of 050 C										
Sample marking	20	185	285	385	485	585	685			
Sample marking	20	105	205	565	-0J	565	005			
Duration of annealing [h]	"as cast" state	100	500	1000	2000	5000	10000			
Duration of annearing [ii]	as cast state	100	500	1000	2000	5000	10000			

Table 2 Marking of samples and duration of their annealing at the temperature of 850°C

We have used an energy dispersion (ED) method of X-ray spectral microanalysis and microanalytical complex JEOL JXA-8600/KEVEX Delta V, Sesame for obtaining of these data. One concentration set of elements was measured for each sample along the line segment oriented in such a way that in each of the investigated samples it intersected approximately comparable number of structurally similar dendritic arms. Example of structures of measured samples is given in the Figures 1 and 2.



Fig.1 Structure of the sample 20 - as cast



Fig.2 Structure of the sample 685

On the basis of examination of microstructure of prepared samples, our own orientation microanalysis, and also on the basis of spectral resolution of the ED, we decided to measure concentration sets of aluminium, titanium, chromium, cobalt, nickel, niobium, molybdenum, tantalum and tungsten (i.e. altogether 9 elements) in each sample at 101 points lying on the line segment with the length $L = 500 \mu m$ (mutual distance of individual points $\Delta L = 5 \mu m$), as this length interval at the same time characterises structural resolution of the used analysis. Working conditions for analyses were identical for all the elements.

Results

We have determined from vast experimentally measured data sets the basic statistical parameters of normal (Gauss) statistical distribution of the measured elements concentrations (Al, Ti, Cr, Co, Ni, Nb, Mo, Ta and W) always for each sample of the alloy IN 738 LC, these results are given in the Table 3. These parameters comprise: c_m mean element concentration (arithmetical mean) in the selected section, σ_{n-1} standard deviation of the measured element concentration, and also c_{min} minimum element concentration and c_{max} maximum element concentration – which were always measured in the selected section of the sample.

We have calculated moreover from the data contained in this table the indexes of an element dendritic heterogeneity in measured segment of the individual samples, the heterogeneity index for the relevant element I_H being defined as a ratio of standard deviation σ_{n-1} and the concentration mean value c_m of the given element in the measured segment of the sample. Calculated indexes of heterogeneity of all analysed elements in the set of samples are arranged in the Table 4.

Afterwards we used the calculation process described in detail in the work [7] for calculation of the values of effective partition coefficients k_{ef} for each analysed element in the set from statistical data contained in the Table 3. These values are also arranged in the Table 4. Heterogeneity indexes I_H and effective distribution coefficients k_{ef} of analysed elements in the individual samples are in fact related to the same measured segment, and they thus represent for each of the analysed elements mutually related pairing values.

Sampla	Para-	Element [mass %]									
Sample	meter	Al	Ti	Cr	Со	Ni	Nb	Mo	Та	W	
	cm	3.108	2.719	16.092	10.375	62.235	0.313	2.079	0.706	2.372	
20	σ_{n-1}	0.703	0.865	3.083	1.597	3.109	0.207	0.305	0.545	0.489	
as cast	Cmax	4.76	4.99	21.96	13.41	69.35	0.75	2.81	1.70	3.29	
	Cmin	1.82	1.32	8.41	7.14	56.33	0.00	1.27	0.00	1.35	
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	cm	2.993	2.667	16.478	10.624	61.875	0.269	2.160	0.649	2.286	
	σ_{n-1}	0.813	0.922	3.426	1.665	3.262	0.245	0.339	0.604	0.489	
185	c _{max}	5.06	5.38	22.95	13.45	70.73	1.56	2.96	2.25	3.40	
	c _{min}	1.51	1.12	7.45	6.35	56.17	0.00	1.16	0.00	1.11	
	c _m	2.893	2.925	15.583	9.870	62.839	0.587	2.123	0.980	2.200	
	σ_{n-1}	0.811	2.891	3.586	1.869	6.735	2.245	0.667	2.352	0.805	
285	c _{max}	4.65	30.51	22.40	12.77	71.02	22.86	8.09	23.88	8.93	
	c_{min}	0.25	1.14	1.21	0.42	3.85	0.00	1.43	0.00	0.81	
	c_m	3.004	2.805	16.284	10.460	61.954	0.358	2.156	0.788	2.192	
	σ_{n-1}	0.914	0.933	3.845	1.870	3.547	0.243	0.360	0.746	0.464	
385	c _{max}	5.18	7.02	23.38	13.42	70.25	1.79	3.27	6.11	3.03	
	c _{min}	1.17	1.09	4.09	4.85	55.95	0.00	1.19	0.00	0.93	
	cm	3.047	2.799	16.710	11.135	61.088	0.235	2.137	0.612	2.237	
	σ_{n-1}	0.973	0.907	3.929	1.860	3.815	0.251	0.435	0.589	0.497	
485	c _{max}	5.62	5.57	23.68	14.25	71.65	0.90	3.08	2.38	3.54	
	c _{min}	1.27	1.26	5.93	6.13	54.74	0.00	0.98	0.00	0.87	
	cm	3.034	2.672	16.507	10.836	61.404	0.279	2.199	0.716	2.354	
	σ_{n-1}	1.105	1.089	4.657	2.260	4.466	0.237	0.475	0.644	0.329	
585	c _{max}	5.30	5.59	23.25	14.38	72.57	0.82	2.99	2.20	3.22	
	c_{min}	1.29	0.96	5.19	5.58	54.73	0.00	0.98	0.00	1.48	
		0.050	0.655	16.250	10.250	62.102	0.051	0.105	0.551	0.015	
	c_m	2.952	2.655	16.379	10.359	62.193	0.351	2.125	0.771	2.217	
60. 7	σ_{n-1}	0.999	1.047	4.379	2.132	4.258	0.190	0.368	0.566	0.431	
685	c _{max}	5.07	5.49	23.85	14.14	/3.00	0.79	2.76	2.11	3.21	
	c_{min}	1.21	0.94	6.26	5.51	54.79	0.00	1.23	0.00	1.02	

Table 3 Basic statistical parameters of the measured element concentrations in samples of the alloy IN 738 LC/850°C

Discussion of results

On the basis of comparison of mean values of concentrations of analysed elements determined from the set of concentration data measured on individual samples and presented in

the Table 3 with chemical composition of the alloy IN 738 LC in conformity with the test certificate (Table 1), it is possible to draw the following conclusions: contents of Al, Ti, Nb, Ta and W in all samples is below the values in the test certificate, contents of Cr is in majority of samples slightly above the limits of the test certificate. The sample "as cast" has also only the chromium mean values measured by us within the test certificate limits of chemical composition (test certificate gives 16.22), while the mean values of all other elements are either lower (Al, Ti, Nb, Ta and W), or higher (Co and Mo).

Table 4 Mean values of heterogeneity index $I_{\rm H}$ and effective distribution coefficient k_{ef} of elements in samples of the alloy IN 738 LC/850°C

Sample	Para-	Element								
	meter	Al	Ti	Cr	Со	Ni	Nb	Мо	Та	W
20 as cast	$\begin{matrix}I_{\rm H}\\k_{ef}\\\pm\sigma_{kef}\end{matrix}$	0.23 1.22 0.11	0.32 1.31 0.18	0.19 0.85 0.10	0.15 0.88 0.08	0.05 1.04 0.02	0.66 2.28 2.95	0.15 0.88 0.07	0.77 2.59 3.72	0.21 0.84 0.10
185	$\begin{array}{c} I_{\rm H} \\ k_{\rm ef} \\ \pm \sigma_{\rm kef} \end{array}$	0.27 1.27 0.14	0.35 1.33 0.21	0.21 0.85 0.12	0.16 0.88 0.09	0.05 1.04 0.03	0.91 2.65 4.22	0.16 0.87 0.08	0.93 2.88 4.70	0.21 0.84 0.11
285	$\begin{array}{c} I_{\rm H} \\ k_{\rm ef} \\ \pm \sigma_{\rm kef} \end{array}$	0.28 1.31 0.12	0.99 0.61 0.08	0.23 0.84 0.14	0.19 1.22 0.08	0.11 1.09 0.10	3.82 0.31 0.14	0.31 0.82 0.07	2.40 0.36 0.20	0.37 0.78 0.09
385	$I_{H} \\ k_{ef} \\ \pm \sigma_{kef}$	0.30 1.31 0.15	0.33 1.33 0.18	0.24 0.82 0.13	0.18 0.87 0.10	0.06 1.05 0.03	0.68 2.08 2.25	0.17 0.86 0.07	0.95 2.42 3.02	0.21 1.23 0.06
485	$I_{H} \\ k_{ef} \\ \pm \sigma_{kef}$	0.32 1.31 0.18	0.32 1.29 0.20	0.23 0.83 0.14	0.17 0.88 0.10	0.06 1.05 0.04	1.07 3.09 6.01	0.20 0.85 0.11	0.96 2.84 4.71	0.22 0.82 0.10
585	$I_{H} \\ k_{ef} \\ \pm \sigma_{kef}$	0.36 1.37 0.20	0.41 1.43 0.23	0.28 0.80 0.16	0.21 0.85 0.12	0.07 1.06 0.04	0.85 2.74 4.44	0.22 0.84 0.12	0.90 2.88 4.75	0.14 0.89 0.07
685	$I_{H} \\ k_{ef} \\ \pm \sigma_{kef}$	0.34 1.33 0.19	0.39 1.38 0.24	0.27 0.81 0.16	0.21 0.85 0.12	0.07 1.06 0.04	0.54 1.89 1.72	0.17 0.87 0.10	0.73 2.34 2.70	0.19 0.85 0.09

From the above-mentioned conclusion it therefore also follows, that samples cast from this alloy from one melt are characterised by comparatively high chemical macroheterogeneity. Extent of this macroheterogeneity exceeds in each case the length of the measured segment, which was 500 μ m.

It follows from the values of heterogeneity index of analysed elements arranged for individual samples in the Table 4, that heterogeneity index of majority of elements has with extended dwell duration at the same temperature (till duration of annealing of 5000 hours) an increasing tendency. The exceptions are Nb, Ta and W, for which it is possible to ascertain that their heterogeneity decreases with extended duration of annealing.

What concerns heterogenic distribution of elements in structure, it is possible to find between the analysed elements the following common characteristics of behaviour at dwell at the temperature of 850°C.

The lowest heterogeneity in structure of the alloy IN 738 LC as cast has been observed in elements Al, Ti, Cr, Co, Ni and Mo. The lowest heterogeneity in structure of the

alloy IN 738 LC after long-term annealing 10000 hours has been observed in elements Nb and Ta, or after annealing 5000 and 10000 hours has been observed in W. The highest heterogeneity in structure of the alloy IN 738 LC after long-term annealing 5000 hours has been observed in Al, Cr and Co.

The highest heterogeneity in structure of the alloy IN 738 LC after long-term annealing 500 hours has been observed in Ti, Ni, Nb, Mo, Ta and W. From the set of these six elements the heterogeneity of Ta, W and Nb will considerably decrease by further extension of dwell at the temperature. Duration of dwell of 500 hours at the temperature of 850 °C seems to indicate re-distribution processes accompanied by comparatively intensive creation of chemical components, or by creation of intermetallic phases.

What concerns the mean heterogeneity calculated from all the samples annealed at the temperature of 850°C and expressed by the parameter I_H , this value increases with the following order of elements: Ni, Co, Mo, W, Cr, Al, Ti, Ta, Nb from the value of 0.070 for nickel up to the value 1.312 for niobium [8].

Effective partition coefficients, numerical values of which are given in the Table 4, have been determined both for the as cast sample and for the annealed samples.

It is possible to conclude from the values of effective partition coefficients determined for the as cast state, that elements Al, Ti, Nb and Ta at crystallisation and subsequent cooling down of the nickel based superalloy IN 738 LC have a tendency to segregate into the matrix in direction to higher concentration of Ni, elements Cr, Co, Mo and W have a tendency to separate from nickel, i.e. they segregate into interdendritic melt.

We have found in the literature [2] for the alloy IN 738 LC the following values of equilibrium partition coefficients: $k_{0AI} = 1.2$; $k_{0Ti} = 0.6$; $k_{0Cr} = 1.05$; $k_{0Co} = 1.1$; $k_{0Nb} = 0.4$; $k_{0Mo} = 0.85$; $k_{0Ta} = 0.7$; $k_{0W} = 1.4$. It follows from the comparison of the values of effective partition coefficients calculated in this work (see Table 4) that real re-distribution of these elements at crystallisation of the nickel based alloy IN 738 LC and at its subsequent cooling down substantially differs from equilibrium crystallisation.

The fact, that behaviour of real nickel based superalloys at crystallisation differs from equilibrium conditions, is confirmed also by the works of Tancret et al. [6] and Ojo et al. [9]. The latter [9] examined the weld fusion zone microstructure of a commercial superalloy IN 738 for the reason that microstructural development in superalloy welds, which invariably controls properties and reliability of the weldments, can be significantly influenced due to microsegregation and non-equilibrium phase transformation that occur during solidification. The cooling rate within the fusion zone was estimated to be about 550 K/s and the initial partition coefficients at the start of solidification of the metallic alloying elements were calculated. The result indicated that W and Co with k > 1 segregate to the dendritic core while Mo, Ta, Ti, Nb and Al with k < 1 enrich the interdendritic regions. Ni and Cr having k values approximately equel to unity tend to exhibit uniform distribution between dendrite core and dendrite interstices.

Tancret et al. [6] investigated a nickel based superalloy for power plant application (with the following nominal composition [mass %]: 20Cr; 3.5W; 2.3Al; 2.1Ti; 5Fe; 0.4Si; 0.07C; 0.005B) from the viewpoint of segregation behaviour of selected elements at crystallisation of the given alloy. They had used the Scheil model for description of dendritic segregation of elements and they had discovered the following facts:

The concentrations of Cr and W in the liquid remain within +10% of the nominal composition until 87% of the material is solidified. Then Cr and W are removed from the liquid

to form $M_{23}C_6$, which will constitute a form of desired segregation, as this carbide is expected in the equilibrium phase diagram. $M_{23}C_6$ forms below 1309°C, instead of 1204°C at equilibrium. "M" in $M_{23}C_6$ is mainly Cr, with also several percent of W, Ni, and Fe, varying throughout solidification. Aluminium firstly segregates in the liquid, and then starts to be removed from the liquid around ~70% solid, as its solubility becomes higher in γ than in the liquid below ~1350°C.

At a higher solid fraction (0.99, ~1217°C), η precipitates, which corresponds to an increase of the Cr concentration in the liquid. It must be noted that this undesirable phase is expected to form when only 1% of the liquid is left. Finally, α precipitates at 1178°C (690°C at equilibrium) and, apparently, the "equilibrium" between this nearly pure α -Cr phase and the liquid stabilises the Cr concentration in the liquid until the end of solidification. Iron behaves differently, as it first slightly concentrates in the solid until solidification is 99.6% complete, and then strongly concentrates in the liquid to reach several times its nominal concentration. Titanium progressively concentrates in the liquid up to five times its initial concentration, as its solubility in both γ and M₂₃C₆ remains low. It finally forms γ' below 1176°C (1011°C at equilibrium). Carbon segregates to the liquid until M₂₃C₆ precipitates. Then, when α -Cr forms, Cr goes preferentially into α instead of M₂₃C₆, and C re-concentrates in the liquid [6].

The Scheil's model used in this work [6] assumes a perfectly homogeneous liquid, and that no diffusion occurs within the solid, which may not be true at high temperatures. Consequently, because back diffusion reduces microsegregation, the actual concentration profile is likely to be between those predicted assuming equilibrium and by the Scheil's method.

From the above mentioned follows that the partition coefficients are not constant during solidification process and they are strongly influenced by the cooling rate which controls the extent of solid-state diffusion during solidification and interdendritic arm spacing.

Complex re-distribution processes at annealing of the alloy IN 738 LC at the temperature of 850°C are evidenced also by the values of pairing correlation coefficients between individual analysed elements. The Table 5 gives an overview correlation matrix of elements with respect to nickel. For 101 measured points only such values of correlation coefficients are at the 95% level of significance, the absolute value of which is greater than 0.196. It follows from this table that correlation between nickel and all the analysed elements (with the exception of tungsten) is at the average very strong. Elements Al, Ti, Nb and Ta have a tendency to re-distribute themselves into the matrix in direction to the higher concentration of Ni, elements Cr, Co, Mo and W have a tendency to separate from nickel; this trend is valid both for process of crystallisation of the given alloy, and also for long-term annealing (the exceptions are the sample 285 and tungsten).

Some other remarkable facts follow from this table as well. It is possible to observe an anomalous behaviour of all the analysed elements in the sample 285 – duration of annealing 500 hours, some elements (Ti, Nb, Ta) get here re-distributed in an opposite manner than in all remaining samples, and coefficients of pairing correlation are moreover statistically highly significant. Another surprising fact is behaviour of tungsten at long-term annealing of the analysed nickel based super-alloy. In previously investigated nickel based alloys [10] the correlation between Ni and carbide-forming W was always very strong and mostly it increased with extended duration of annealing. For the alloy IN 738 LC annealed at the temperature of 850°C it is valid, that for longer durations of annealing the dependence between Ni and W is statistically insignificant – very low. Further work is being planned based on the results of the present work for the more precise description of microsegregation behaviour of nickel-based alloys.

Conclusion

The article presents selected results of investigation of chemical and structural microheterogeneity of high-alloyed nickel based alloy IN 738 LC as cast and after long-term annealing at the temperature of 850°C. In order to achieve this goal we have used the original method of measurement of dendritic micro-heterogeneity of elements for the given nickel based alloy, and we have also determined effective distribution coefficients of analysed elements (Al, Ti, Cr, Co, Ni, Nb, Mo, Ta and W) between liquid and solid phase of this alloy.

On the basis of experimental measurements and subsequent processing of concentration date we have ascertained the following basic facts:

- Dendritic heterogeneity of elements in the alloy IN 738 LC is at the average the lowest in "as cast", i.e. without any applied heat treatment. It can be thus concluded that original redistribution of elements in structure of the alloy IN 738 LC is determined by conditions of crystallisation of this alloy.
- It is possible to assess the tendency of elements to segregate dendritically during crystallisation from the heterogeneity index of the analysed elements; this value increases in the order of elements: Ni, Mo, Co, Cr, W, Al, Ti, Nb and Ta (see Table 4, sample 20). At the same time the elements Ni, Al, Ti, Nb and Ta have a tendency to segregate into the solid phase, while the remaining elements Mo, W, Cr and Co tend to segregate into the melt (see correlation matrices in the Table 5).
- It also follows from measurement of chemical heterogeneity of elements that processes, which characterise actual distribution of elements during crystallisation, differ significantly in qualitative and quantitative manner from processes determining equilibrium distribution of elements.

sample	Al	Ti	Cr	Со	Ni	Nb	Mo	Та	W
20	0.94	0.95	-0.99	-0.98	1	0.64	-0.80	0.80	-0.58
185	0.94	0.92	-0.98	-0.98	1	0.63	-0.88	0.83	-0.45
285	0.71	-0.72	-0.07	0.04	1	-0.86	-0.97	-0.78	-0.79
385	0.95	0.95	-0.99	-0.98	1	0.53	-0.88	0.66	0.08
485	0.92	0.93	-0.99	-0.98	1	0.75	-0.92	0.82	-0.28
585	0.97	0.97	-0.99	-0.99	1	0.82	-0.95	0.87	-0.09
685	0.96	0.97	-0.99	-0.99	1	0.64	-0.92	0.84	-0.07
average	0.91	0.71	-0.86	-0.84	1	0.45	-0.90	0.58	-0.31
deviation	+0.09	+0.63	+0.35	+0.39	0	+0.58	+0.06	+0.60	+0.31

Table 5 Overview correlation matrix of elements with respect to nickel for the alloy IN 783 LC

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Literature

- Chvorinov N.: Crystallization and non-homogeneity of steel (in Czech). NČSAV, Praha 1954
- [2] Sung P.K., Poirier D.R., Met. and Mat. Trans. A, Vol. 30A, No. 8, 1999, pp. 2173-2181

- [3] Kattner U.R., Boettingen W.J., Coriell, S.R. Z. Metallkd. Vol.87, No.7, 1996, pp.522-528
- [4] Tancret F., Bhadeshia, H.K.D.H., Mackay, D.J.C. Key Engineering Materials, Vols. 171-174, 2000, pp.529-536
- [5] Florian M.: Kovové Mater, Vol. 38, No. 5, 2000, pp.305-314
- [6] Tancret F., Bhadeshia, H.K.D.H. Mater. Sci. Technol., Vol. 19, March 2003, pp.291-295
- [7] Dobrovská V., Dobrovská J., Rek A., Stránský K.: Hutnické Listy LIV, 1999, No.7/8, pp.51-57
- [8] Rek A., Stránský K.: Research Report No. 611-16-01. VTÚO Brno, June 2003, Brno
- [9] Ojo O.A., Richards N.L., Chaturvedi M.C.: Scripta Mater, Vol. 51, 2004, pp.683-688
- [10] Dobrovská J.: Heterogeneity of Ni-based superalloys. Research Report MP 630014, VSB-TU Ostrava, November 2003