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HETEROGENEITY OF INCONEL 713 LC AND INCONEL 738 LC

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ABSTRACT
The contribution deals with the chemical and structural heterogeneity of a creep resistant Ni-based alloy Inconel 713 LC and Inconel 738 LC. These materials belong to the cast poly-crystalline nickel super-alloys which are used for the production of circulating wheels of small combustion turbines for supplementary energetic units in aviation industry. In operational conditions of combustion turbines as part of turbo engines, blades of the circulating wheels are stressed in time, temperature and stress variable cycles during the loading procedure. The blades are exposed to a series of degradation factors such as high-temperature corrosion, fatigue processes and creep during the operation. Measurement of the chemical microheterogeneity of elements was realized in as-cast state before and after exploitation. As microanalytical complex was applied JEOL JXA 8600/KEVEX Delta V Sesame and EDS. In the analysis of chemical microheterogeneity, the concentrations of Al, Ti, Cr, Ni, Zr, Nb, Mo were measured in 101 points of the structure. Then the indices of dentritic microheterogeneity I_H were estimated. The redistribution of elements is discussed.
Keywords: cast nickel alloy, chemical heterogeneity, mathematical-statistical analysis

1. INTRODUCTION

Some materials, especially steels and nickel-based superalloys, solidify at the dendrits formation. Impurity segregation causes decrease of materials melting temperature. It sets up a negative thermal gradient. A tendency to balance the thermal gradient leads to the dendritic structure creation. Several elements segregate into dendritic zone and the rest to interdendritic zone. It causes a concentration heterogeneity. It also forms disequilibrium phases, enclosers or eutectic phases with low melting temperature. Interatomic bonds are weakened and it deteriorates mechanical and creep properties of mentioned alloys [1].

This paper aims to evaluate the dendritic heterogeneity and to measure the concentration sets of selected elements as found in the samples of INCONEL 713LC and 738LC. For details on the measurement methodology see literature [1-3]. To assess the dendritic heterogeneity and to determine the effective distribution coefficients of the elements, it was necessary to measure, in a defined manner, large concentration sets of the considered elements, in this case those encountered in the structurally suitable areas of the samples featuring the greatest possible measure of likeness [1-3, 6].

Data in following contribution were originally measured for another purpose for Inconel 713 LC and for Inconel 738 LC. Measurement methodology and expositions have a comparable features. And thus is possible trade off the mathematical-statistical results.

2. EXPERIMENTAL MATERIAL AND METHODS

This contribution deals with two most practically used nickel-based superalloys Inconel 713LC and 738LC (LC = low carbon variant), see the chemical composition in Table 1. The experimental material Inconel 713LC (without Co) in „Master quality“ (50% returns, 50% new charge) was cast to accurate wax pattern, after that was worked to creep test sample (Fig.1). The samples were exposed by creep tests, conditions you can see in Table 2. Measurements were situated to samples from shank of the test sample (temperature-tension exposition) and head (temperature exposition) of creep test sample. Inconel 738LC (with Co) also in „Master quality“ was heat treated (solution annealing at 1120 °C for 2 hours and precipitate annealing at 850 °C for 24 hours). Samples were vacuum cast into cylinder shape (diameter 23 mm, height 9 mm). The samples were temperature exposed by annealing at 850 °C for 100 – 10 000 hours, see Table 2.

Table 1. Alloy compositions at wt. %

	INCONEL 713 LC		INCONEL 738 LC	
	Average	**Range**	**Average**	**Range**
C	0,05	0,03 – 0,07	0,1	0,09 – 0,13
Mn	< 0,05	< 0,25	< 0,05	< 0,20
Si	< 0,05	< 0,5	< 0,05	< 0,30
Cr	12,08	11,0 – 13,0	16,22	15,7 – 16,3
Ti	0,75	0,40 – 1,00	3,37	3,20 – 3,70
Al	5,91	5,50 – 6,50	3,35	3,20 – 3,70
Fe	< 0,10	< 0,5	0,2	< 0,35
B	0,010	< 0,015	0,008	< 0,009
Zr	0,10	0,05 – 0,15	0,04	0,03 – 0,08
Nb	2,02	1,50 – 2,50	0,84	0,60 – 1,10
Ta	< 0,05	-----	1,77	1,50 – 2,00
Mo	4,58	3,80 – 5,20	1,71	1,50 – 2,00
W	-----	-----	2,63	2,40 – 2,80
Co	< 0,05	< 1,0	8,78	8,00 – 9,00
Cu	< 0,05	< 0,5	< 0,05	< 0,10
P	0,004	< 0,015	< 0,004	< 0,010
S	< 0,004	< 0,015	0,002	< 0,015
Ni	Base	Base	Base	Base

Table 2. Survey of used exposition

INCONEL 713 LC		INCONEL 738 LC	
Creep test		**Annealing**	
[ºC]	**[hours]**	**[ºC]**	**[hours]**
As received		As received	
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Head of test sample		850	100
850	1220	850	500
870	39	850	1000
Shank of test sample		850	2000
850	1220	850	5000
870	39	850	10000

Figure 1. Creep test sample, ø 5 mm, length 25 mm

Using a *Protopress* machine the samples were pressure embedded into *Mounting Resins* a conductive thermoplastic mounting material manufactured by the STRUERS Company. When prepared, the samples were etched with an etching agent containing 35 ml of HCl, 10 drops of H_2O_2, 65 ml of ethyl alcohol and 1 g of $CuCl_2$ for IN713LC and Marble for IN738LC. Then an EPITYP optical microscope was utilized to select areas suitable to be analyzed, the areas thus selected were machine encircled and each area so encircled was marked (using a HANEMANN microhardness gage under a NEOPHOT 2 optical microscope) with a row of micro-sized dots; the dots constituted a vector line from which the relevant concentration sets were to be taken for each sample used to assess the dendritic segregation of the selected elements. After, the samples were treated to the ED microanalysis on the JEOL JXA-8600/KEVEX Delta V (15kV beam voltage, 50sec time take-off).

To properly assess the level of dendritic heterogeneity and to determine the effective distribution coefficients, sufficiently large concentration sets of the monitored elements had to be available. In the case of Inconel 713 LC were observed concentration of Al, Ti, Cr, Ni, Zr, Nb, Mo and elements such as Al, Ti, Cr, Co, Ni, Nb Mo, Ta, W were gauged for Inconel 738 LC. In this case we have decided to subject each sample to the ED microanalysis so as to obtain one concentration set of 101 dots placed alongside a vector line; the line was to be oriented so that it intersected, in each analyzed sample, a roughly comparable number of structurally similar dendritic cells and their boundaries. To keep the number of dendritic axes intersected by the measured line roughly the same, we have chosen different lengths of the section to be measured [4 - 5]. For all samples the length was L = 500 μm (the spacing of the adjacent dots was ΔL = 5 μm). The dendritic structure was evaluated based on half distance between dendritic axes. The Table 3 states the dendritic structure parameters:

Table3. Number of interdendritic spaces intersected

INCONEL 713LC	n	L/2n	L [μm]
As received	8	31,25	500
Head 870ºC - 39hrs	9	27,78	500
Head 850ºC – 1220hrs	8	31,25	500
Shank 870ºC - 39hrs	6	41,67	500
Shank 850ºC–1220hrs	9	27,78	500

INCONEL 738LC	n	L/2n	L [μm]
As received	8	31,25	500
All annealing 850ºC	8	31,25	500

Note*) L/2n is half of distance measured between the dendritic branches, n is number of intersected ribs, L means measured section.

3. DISCUSSION OF THE ANALYSIS

The Table 4 lists the average concentrations of the measured elements XS; the standard deviation SX; the index of heterogeneity of the measured element content I_{Het} = SX/XS and the index of segregation I_s= Max/XS (Max means maximum content); efficective distribution coefficient $K_{ef} \approx I_s^{-1}$ and the chemical composition of the samples being measured (Quoted):

Table 4. Selected measurement & analysis results (wt.%)

Sample		Al	Ti	Cr	Ni	Co	Nb	Mo	Ta	W
INCONEL 713 LC As received	XS (wt.%)	**5,49**	**0,58**	**11,65**	**76,38**	-	**1,64**	**4,24**	-	-
	SX (wt.%)	0,69	0,53	1,90	4,17	-	3,17	0,91	-	-
	Quoted (wt.%)	**5,91**	**0,75**	**12,08**	**base**	-	**2,02**	**4,58**	-	-
	I_{Het}=SX / XS (-)	0,13	0,92	0,16	0,06	-	1,66	0,22	-	-
	Is = Max / XS (-)	1,27	9,72	1,43	1,04	-	20,10	2,85	-	-
	$K_{ef} \approx I_s^{-1}$	0,79	0,10	0,70	0,96	-	0,05	0,35	-	-
INCONEL 738 LC As received	XS (wt.%)	**3,11**	**2,72**	**16,09**	**62,24**	**10,38**	**0,31**	**2,08**	**0,71**	**2,37**
	SX (wt.%)	0,70	0,87	3,08	3,11	1,60	0,21	0,31	0,55	0,49
	Quoted (wt.%)	**3,35**	**3,37**	**16,22**	**base**	**8,78**	**0,84**	**1,77**	**1,71**	**2,63**
	I_{Het}=SX / XS (-)	0,23	0,32	0,19	0,05	0,15	0,66	0,15	0,77	0,21
	Is = Max / XS (-)	1,53	1,84	1,37	1,11	1,29	2,40	1,35	2,41	1,39
	$K_{ef} \approx I_s^{-1}$	0,65	0,55	0,73	0,90	0,77	0,42	0,74	0,42	0,72

Note*)The higher is I_{Het} (Is), the more element segregates at the solidification. The more vary K_{ef} from 1, the more element segregates. In case K_{ef} <1 the element segregates into the rest of melt (Interdendritic Zone), K_{ef} >1 means the element segregates into dendrite.

The measured and calculated concentration sets of the analyzed elements are quoted at Fig. 3 and structures of the same sample, see Fig. 2. Segregation running of given element versus solid phase rate we can demonstrate by means of distribution curve of dendritic segregation, see Fig.4 for Inconel 738LC as received and Fig.5 for Inconel 713LC after creep test (Shank 870ºC 39 hrs), [3]. Measured concentration sampling is sequenced in upward order (element segregates to interdendritic zone - IZ) or in downward order (into dendrite - DZ). Solid phase rate means ratio i-th measurement versus total number of a measurements.

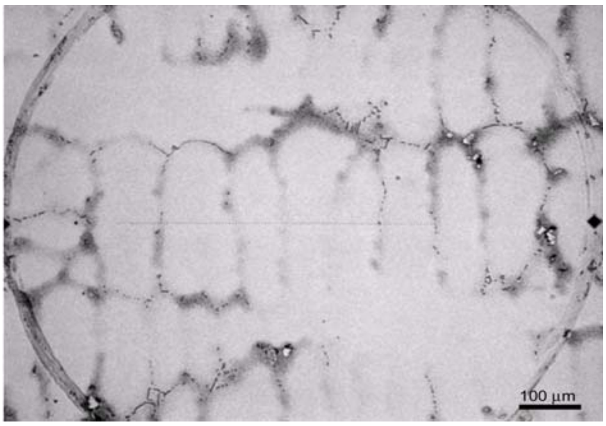

Figure 2. Structure of Sample (Shank 870ºC–39hrs) INCONEL 713LC

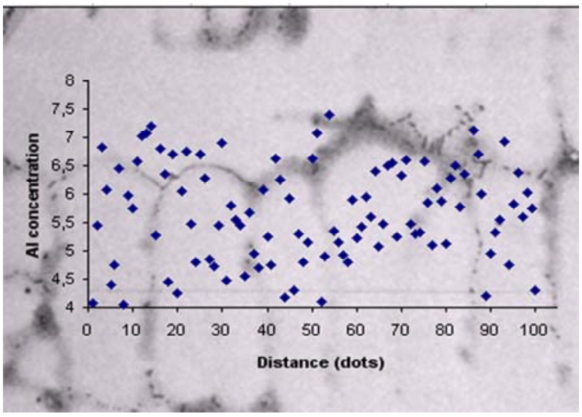

Figure 3. Al concentration versus the distance over a section 500 μm (101 dots)

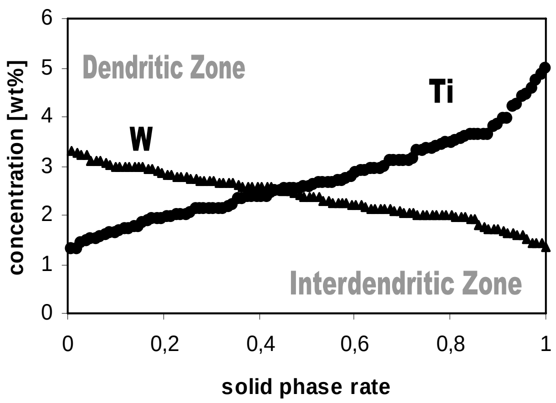

Figure 4. Distribution curve of dendritic segregation; INCONEL 738LC

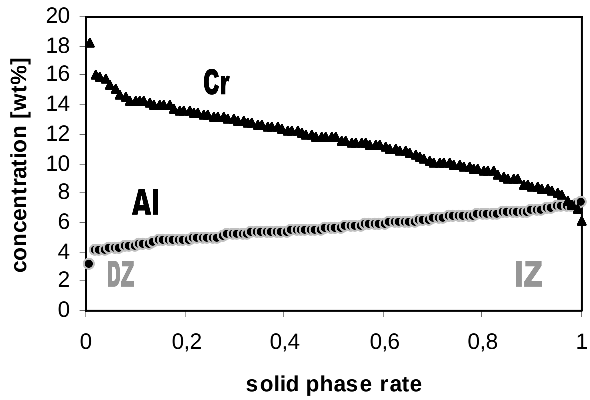

Figure 5. Distribution curve of dendritic segregation;INCONEL 713LC

4. CONCLUSION

Under the measurements of chemical heterogeneity of both superalloys were found following facts:

- Dendritic heterogeneity of elements of Inconel 738 LC sample as received i.e. cast state is approximately the lowest. Annealing at temperature 850^{o}C increases a chemical heterogeneity of presented elements as Al, Ti, Cr, Co, Ni and Mo. On the other hand; chemical heterogeneity of Nb, Ta and W goes down into minimum with the longest period of annealing i.e. 5000 and 10000 hours. Heterogeneity of constitutive elements i.e. Al, Ti, Cr, Co, Ni a Mo escalates per their redistribution in structure and it creates a chemical coumpounds or intermediary phases.
- Dendritic segregation of elements such as Nb, Ti, Cr, Al, Ni of Inconel 713 LC turns up with time and temperature of creep test. It doesn´t obtain for Mo because its index of segregation is decreasing. An index of segregation growth tendency is higher in the shank compared to the head of creep sample. Measurement results adverts to two processes – precipitation and homogenization; that are in progress of creep load case. The first one increases a system heterogeneity (Al, Ti, Ni, Nb – γ′ hardening). Intensity of precipitation also rises with a tension in the shank of creep test sample. The second one the heterogeneity again decreases.
- A dendritic segregation tendency of elements we can qualify according to reciprocal value of segregation index ($k_{ef} \approx I_S^{-1}$). The first approximation means a local effective distribution coefficient of appropriate element. It was found; an intensity element segregation in Inconel 738 LC grow up according to order: Nb, Ta, Mo, Ti, Al, W, Cr, Mo, Co and Ni. Elements such as Ni, Al, Ti, Nb and Ta have a tendency segregate into solid phase and Mo, W, Cr or Co into interdendritic zone. An order of segregation tendency in Inconel 713 LC is following: Zr, Nb, ti, Cr, Al, Mo and Ni.
- The analyses results demonstrate that the mean distances of the dendritic branches found in all samples of INCONEL 738 LC are approximately same. It come also up to second alloy.

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