Quantitative Microstructural Analysis of a Ni-based Superalloy After Different Heat Treatments

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The efficiency of time-temperature treatment (T-TT) on metal melts can be microstructurally analysed through their degree of purity in non-metallic inclusions. In the case of the Ni-based super alloy under discussion (MSRR 7045) the heat treatment was the undercooling consequences both on the durability of the casting environment (ingots-refractories) and on the internal structure of the metal (porosity, microstructural isotropy).

Keywords: time-temperature treatment, undercooled melt, non-metallic inclusions, purity, microstructural isotropy

One of the modern ways to correct the structure of metal melts in order to obtain qualitative improved solid products is the approach of heredity or metallurgic genetics [3].

The dedicated literature of the past decades shows the existence of two ways to improve the quality of a solid metal products depending on the genetic cycle of interaction of the two condensed states of aggregation when processing a metallic material:

- the genetic cycle of interaction of solid-liquid-solid, i.e. the impact of the metallic materials of the charge upon the metal melt and therefore upon the properties of the newly obtained solid alloy [2,4].
- Thermo-Time Treatment (T-TT):
- The parameters of the heat treatments from our work consists of the following:

1. overheating the melted metallic alloy by about 120-250°C above the melting point (T1) up to the critical temperature (Tcr);
2. isothermal maintaining the temperature of the melted metallic alloy for 10-30 minutes;
3. fast cooling of the melted metallic alloy for 10-15 minutes up to the casting temperature (Tt) or up to an undercooling temperature (Ts) which is 20-80°C below Tt;
4. isothermal maintaining this level of temperature of the melted metallic alloy for 8-15 minutes [1].

The two ways of improvement do not exclude one another; on the contrary, they can be simultaneously applied during the production of the metallic material.

The metallic material used in the experimental section is a Ni-based superalloy – MSRR 7045 (*M*aterials *S*uper Alloys *R*olls-*R*oyce *7045*), which is also used in aeronautics for the hot and highly corrosive areas of aircraft engines.

Table 1
CHEMICAL COMPOSITION OF THE MSRR 7045 SUPERALLOY

Samples composition, %											
C	Si	Mn	*Cr*	Mo	*W*	*Al*	Co	*Ti*	Fe	Other elements	*Ni*
0.09	0.56	0.57	22.3	10.6	0.48	0.85	0.98	2.56	0.45	Bi 0.0008	rest
Standard composition											
≤0.1	≤0.6	≤0.6	20-23	9-10.5	≤0.6	0.7-0.9	≤1	2.4-2.8	≤0.5	Ag + Bi + Pb = 0.0016	base

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Experimental part

When we analyse the production technology of the Ni superalloy we think that primary production could be the optimal phase to apply a thermal treatment on the liquid metal product in order to improve its heredity from at least 3 points of view:

- -the bars that are cast during the primary production become raw material for the secondary melt which gives the metallic material its final quality;
- -the possible impurifications during primary production, with different causes: the refractary lining of the cupel, the quality of the raw material in the charge;
- -the final destination of the resulted product after the secondary production and the huge demands in thermo-corrosive environment.

Finding the actual work temperature

-The critical temperature Tcr was found by analysing the anomaly of the polithermal of the surface tension, at cooling process starting from the highest thermal threshold (1675°C): 1630°C [1]

-The liquidus temperature, $T_L = 1360^oC$

-The overheating indicated in the specialized literature, between: [T_L+120°C, T_L+250°C]

-The optimal working temperature of the production aggregate CIV Leybold-Heraues IS-1 (Vacuum Induction Melting Furnace): 1600°C-1750°C

Therefore, we chose the initial temperature T-TT- 1640°C as effective work temperature

Sampling the approval test, p_M

-Primary development of MSRR 7045 according to Leybold-Heraues IS-1 10 kg CIV oven technology

-Sampling of the approval test

Time-temperature treatment including undercooling of the MSRR 7045 superalloy

There were performed three sets of experiments where the primarily developed test alloy was treated in its liquid stage according to the liquid treatment pattern involving undercooling.

With each sub-version, we modified only the isothermic preservation time at the treatment temperature of 1640°C;

We chose the undercooling temperature to be 50°C below the casting temperature, Tsr = 1420°C

We subsequently sampled p_1,p_2, p_3 test sets to test the alloy in its solid stage

Hereinbelow, the treatment patterns:

Test 1, p_1:

-overheating the melted metal alloy at the pre-set temperature: 1640°C;

-isothermic preservation for 10 minutes;

-IS-1 oven disconnected, fast cooling for 6 minutes up to the undercooling temperature of 1420°C;

-Isothermic preservation for 10 minutes at undercooling temperature

Test 2, p_2:

-overheating the melted metal alloy at the pre-set temperature: 1640°C;

-isothermic preservation for 20 minutes;

-IS-1 oven disconnected, fast cooling for 6 minutes up to the undercooling temperature of 1420°C;

-Isothermic preservation for 10 minutes at undercooling temperature

Test 3, p_3:

-overheating the melted metal alloy at the pre-set temperature: 1640°C;

-isothermic preservation for 30 minutes;

-IS-1 oven disconnected, fast cooling for 6 minutes up to the undercooling temperature of 1420°C;

-Isothermic preservation for 10 minutes at undercooling temperature

Results and discussions

The present article shows the quantitative micro-structural analysis by computer-aided optical microscopy , made on an Reichert Univar microscope.

This procedure analyzes the purity of non-metallic inclusions following the above-mentioned thermo-temporal treatment.

The proportion and dimension class distribution test of non-metallic inclusions was automatically performed by the computer. The option to present the results consisted of the number of objects bar chart depending on the measurements performed, i.e. the dimension class percentage of inclusions depending on the class (figures 1-4)

In order to be automatically analyzed, p_M, p_1, p_2 și p_3 samples were taken on the two normal directions: transversal and longitudinal

By this bi-directional analysis we meant to point out the following:

-the purity of non-metallic inclusions following T-TT

-the possible microstructural isotropy

Quantitative analysis on the sampling direction

Following the unitary analysis of the 8 histograms we found that the sampling direction influenced the absolute values of the inclusions quantity, as follows:

-for all 3 samples, the inclusions quantity was smaller in the transversal sampling direction

-in both sampling stages, for all samples, the largest inclusions quantity, an average of more than half, consisted in small particles (20µm), as follows:

47-64% Small particles (20µm);

25 –35% Medium particles (40 - 80 µm);

< 10% Large particles (> 100 µm)

Table 2 synthesizes the histograms data concerning nonmetallic inclusions purity.

Table 2

THE NON-METALLIC INCLUSIONS CONTENT IN THE ANALYZED SAMPLES

Sample	Direction of Sampling			
	Transversal		Longitudinal	
	MMB*,%	Inclusions, %	MMB, %	Inclusions, %
p_M	97.54	2.46	97.06	2.94
p_1	97.95	1. 94	97.94	2.06
p_2	98.38	1.62	98.04	1.96
p_3	98.58	1.42	98.42	1.58

(*MMB = metal mass base/groundmass)

The table analysis results in the following hypotheses:

-The inclusions rate decreases regularly in the metallic groundmass (MMB, table 2) in favor of long T-TT

By comparison with the approval test, we get the decrease rates of the number of impurities in sample 3:

transversally: : 43%;

longitudinally: 47%

Although the decrease rate is higher in the longitudinal direction, there is, on the whole, a reduction of anisotropy in the sampling directions, as previously shown.

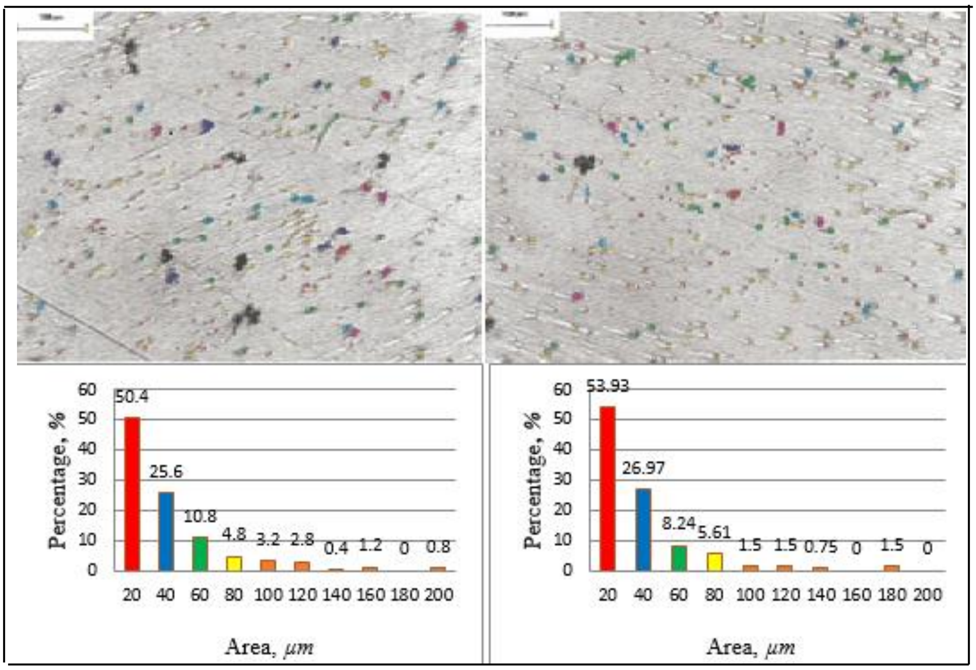

Fig. 1. Non-metallic inclusions distribution in p_M in the longitudinal (left side) and transversal (right side) directions

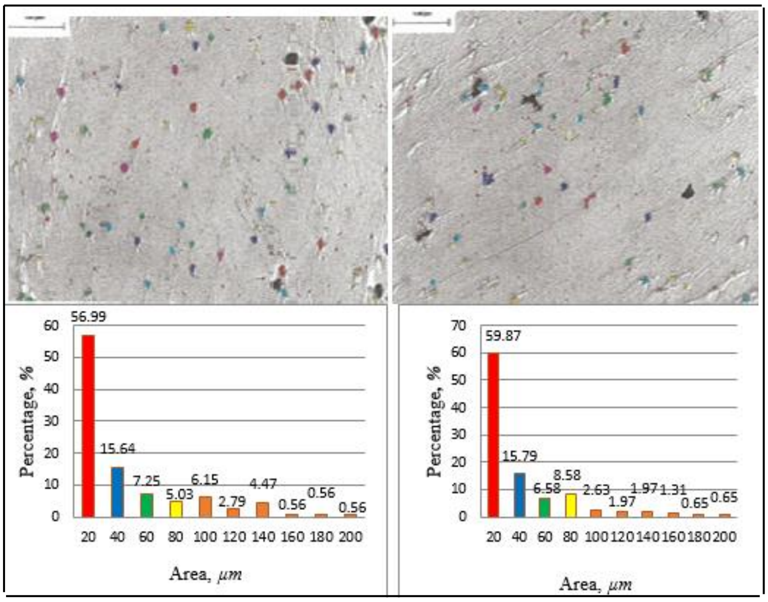

Fig. 2. Non-metallic inclusions distribution in p_1 in the longitudinal (left side) and transversal (right side) directions

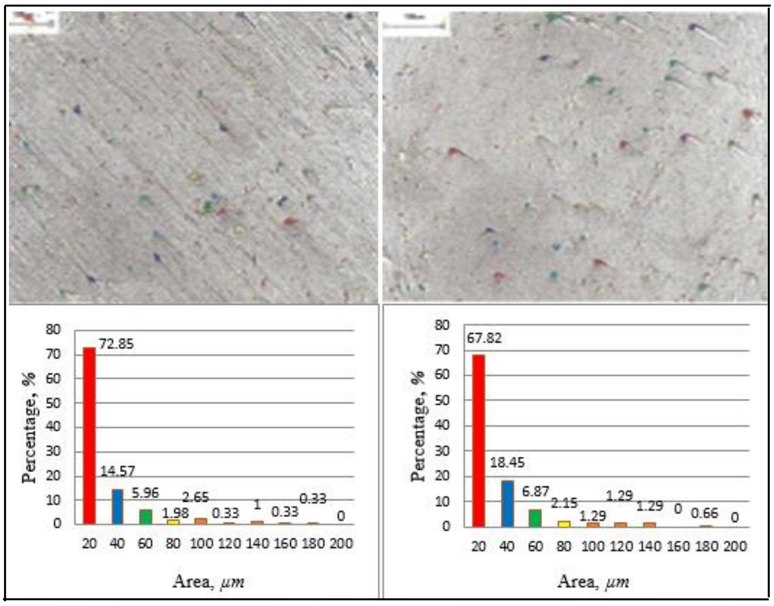

Fig. 3. Non-metallic inclusions distribution in p_2 in the longitudinal (left side) and transversal (right side) directions

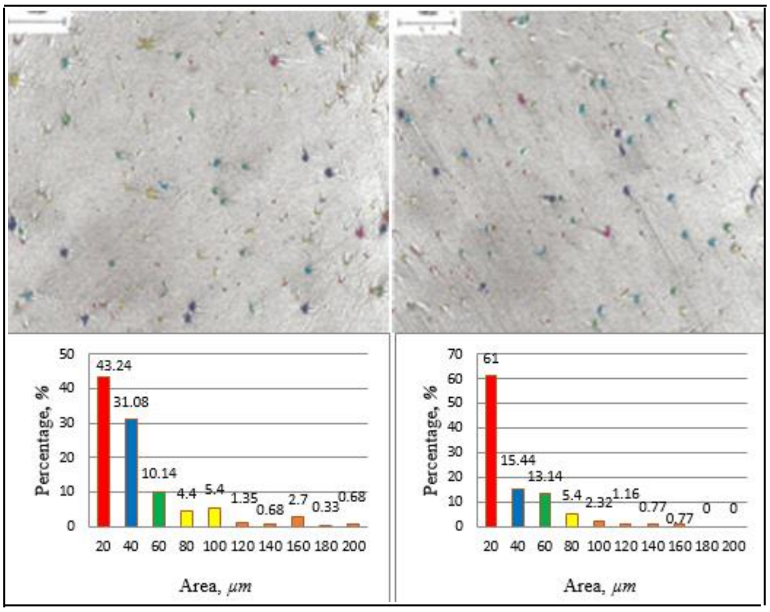

Fig. 4. Non-metallic inclusions distribution in p_3 in the longitudinal (left side) and transversal (right side) directions

Quantitative analysis on the sampling direction

The data required concerning quantitative analysis on the direction of the sample are included in Table 3

Table 3
THE NON-METALLIC INCLUSIONS CONTENT DEPENDING ON THE DIMENSION CLASSES AND THE SAMPLING DIRECTION

Sample	Sampling Direction									
	Transversal, µm					Longitudinal, µm				
	20	40	60	80	>100	20	40	60	80	>100
p_M	53.93	26.97	8.24	5.61	5.24	50.4	25.6	10.8	4.8	8.4
p_1	59.87	15.79	6.58	8.58	9.18	56.99	15.64	7.25	5.03	15.09
p_2	67.82	18.45	6.87	2.15	4.53	72.85	14.57	5.96	1.98	4.64
p_3	61	15,44	13.14	5.40	5.02	43.24	31.08	10.14	4.40	11.14

The analysis of the data above results in the following conclusions:

The small vs large size particles rate (>80µm) mainly increases transversally. From this point of view, there is a certain balancing of the metallic material microstructure because the small size metallic inclusions could lead to smaller ”distorsions” within the melt clusters.

The increase is more obvious at medium T-TT of 20 minutes.

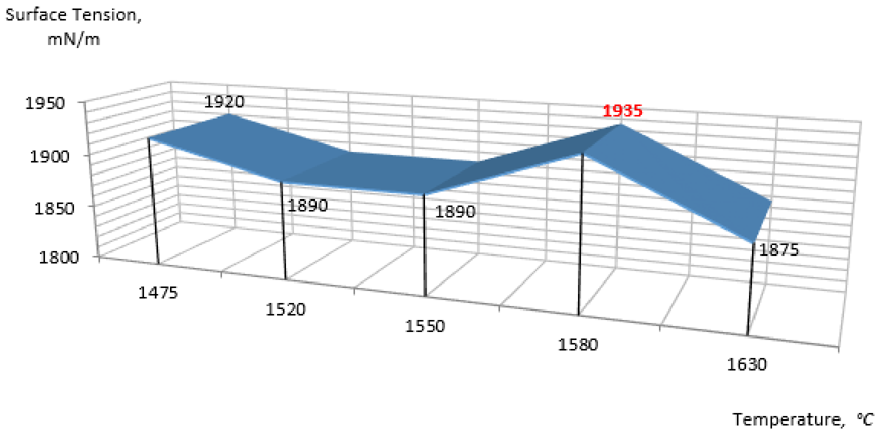

Fig. 5. Surface Tension on Temperature for Mutual Process

Conclusions

The critical temperature of the alloy which has not been thermically treated in its liquid stage is 1630°C.

The temperature in liquid state was obtained from the analysis of the anomaly of the superficial tension polithermal.

The verification by “inverse process”, i.e. the reversal of the actual liquid-solid-T-TT cycle with a solid-liquid cycle by applying T-TT to a sample of the previously treated liquid material (the test bars waste of the T-TT under-cooled for 30 minutes) led to a new critical temperature value of 1580°C.

On the whole, there is a tendency of increase of the surface tension with the temperature as well as the decrease of the maximum point by approximately 50°C which shows a possible micro-homogenization of the superalloy structure.

The heat treatment applied to the liquid MSRR 7045 - T-TT superalloy led to positive results in approaching metallurgic genetics.

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