www.czasopisma.pan.pl PAN www.journals.pan.pl

ARCHIVES
of
FOUNDRY ENGINEERING

DOI: 10.2478/v10266-012-0099-5

ISSN (2299-2944)
Volume 12
Issue 4/2012

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

5 – 10

Evaluation of Metallurgical Quality of Master Heat IN-713C Nickel Alloy Ingots

F. Binczyk[a]*, J. Cwajna[b], S. Roskosz[b], P. Gradoń[a]
[a] Chair of Materials Technology
[b] Chair of Materials Science
Silesian University of Technology, Krasińskiego Str. 8, 40-019 Katowice, Poland
* Corresponding author. E-mail address: franciszek.binczyk@polsl.pl

Received 18.06.2012; accepted in revised form 03.09.2012

Abstract

The paper presents the results of evaluation of the metallurgical quality of master heat ingots and of the identification of non-metallic inclusions (oxides of Al., Zr, Hf, Cr, etc.), which have been found in the shrinkage cavities formed in these ingots. The inclusions penetrate into the liquid alloy, and on pouring of mould are transferred to the casting, especially when the filtering system is not sufficiently effective. The specific nature of the melting process of nickel and cobalt alloys, carried out in vacuum induction furnaces, excludes the possibility of alloy refining and slag removal from the melt surface. Therefore, to improve the quality of castings (parts of aircraft engines), it is so important to evaluate the quality of ingots before charging them into the crucible of an induction furnace. It has been proved that one of the methods for rapid quality evaluation is an ATD analysis of the sample solidification process, where samples are taken from different areas of the master heat ingot. The evaluation is based on a set of parameters plotted on the graph of the dT/dt derivative curve during the last stage of the solidification process in a range from T_{Eut} to T_{sol}.

Keywords: ATD thermal analysis, Nickel superalloys, Master heat ingot, Solidification, Non-metallic inclusions

1. Introduction

Creep-resistant nickel alloys are the primary material used for cast components of aircraft engines, both static and rotary steering parts, operating at elevated temperatures under the action of mass forces [1, 2]. The requirements imposed on these castings include, among others, high fatigue resistance, creep resistance at high temperature and corrosion resistance in a medium containing products of fuel combustion. The performance properties of these elements depend on the macro-and microstructure formed during solidification and on the presence of non-metallic inclusions, usually precipitated in the last stage of solidification [3]. These processes are related with the liberation of higher or lower amounts of energy (exothermic processes) and therefore can be analysed by an ATD thermal analysis [4-7]. Especially valuable for an analysis of this type is the information provided by the shape of the dT/dt derivative curve. Even small amounts of heat that usually leave the shape of the T = f (t) curve unchanged, do change its derivative resulting in visible collapses, inflections and temperature arrests. Due to the complex chemical composition of nickel alloys, besides the base eutectic, shortly before the end of the solidification process, the precipitation of various other eutectics, usually characterised by a low melting point, may occur. The composition of these eutectics often includes the impurities present in the shrinkage cavities and microporosities formed in the primary ingots (so called "master heat" ingots). These eutectics reduce the high temperature performance characteristics of castings. Furthermore, during the high temperature heat treatment (e.g. applied to cast blades), as a result of melting and re-dissolving of the components of these eutectics in the alloy matrix, a considerable increase of the shrinkage microporosity may occur. One way to improve the structure and properties of nickel alloy castings is by surface modification [8, 9] and bulk modification [10-15]. In this scope, numerous tests were performed under the key project.

A particular advantage of the ATD method is the short time in which data are obtained to evaluate the tested material and the quality of the examined master heat ingots [16, 17].

2. Research problem

For the majority of nickel alloys, four main stages can be distinguished on the ATD curves. They are shown in Figure 1 on the example of a cylinder (φ40x100mm) cast from the IN-713C alloy.

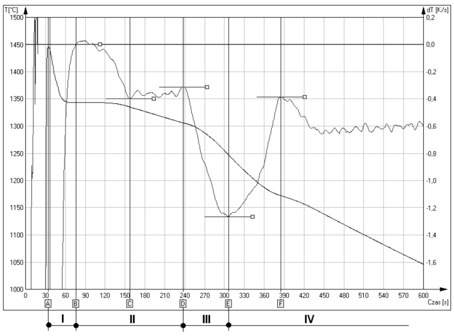


Fig. 1. Main stages in the solidification and cooling of casting made from the IN-713C alloy

In stage I, from T_{max} to T_{lik} (points A to B), the ATD graphs can provide information about the process of the formation of the nuclei of crystallisation (the effect of modification, primary carbides).

In stage II, from T_{lik} to T_{Eut} (points B to D), the ATD graphs are the basis for quantitative evaluation of the metal matrix composition (γ phases, eutectics composed of carbides + intermetallic phases).

In stage III, from T_{Eut} to T_{sol} (points D and E), the ATD graphs can serve as a point of reference for the evaluation of alloy "purity". The presence of gas and impurities in the melt usually leads to the formation of low-melting point eutectics, the occurrence of which considerably prolongs the duration of the final stage of solidification and reduces the value of the dT / dt derivative (the rate of temperature decrease). The slope of the tangent to the derivative curve also changes, depending on the degree of alloy contamination. When the dT / dt curve slope angle assumes low values, it means that the alloy is heavily contamined with low-melting point non-metallic inclusions. On the contrary, high value of the slope angle (over 70°) suggests high-purity of the examined alloy.

In stage IV, from T_{sol} to T_{ot} (from point E), the ATD graphs allow judging about the phase transformations that take place already in the solid state.

The first rough examination of the solidification curve of nickel alloys has already indicated that the ATD analysis is a good tool in evaluating the metallurgical quality of these alloys [9]. The source of contaminations in castings can be:

- melting and casting conditions (time of melting, the temperature of overheating, pouring temperature),
- solidification conditions (mould temperature, thermophysical properties of moulding material),
- side effects of surface and bulk modification,
- the quality of master heat ingots (the presence of oxides and other inclusions).

The mere technology of melting nickel alloys (closed melting chamber) makes the process of refining, deslagging, etc. impossible. Therefore, any of the above mentioned factors may eventually lead to a contamination of nickel alloys with non-metallic inclusions or gas.

Non-metallic inclusions are usually present in the composition of low-melting point eutectics, which are the last ones to solidify on grain boundaries, changing the "image" of an ATD curve in stage III of the solidification process. The analysis of parameters typical of this stage, such as the temperature T_{sol}, the value of the dT / dt derivative, the slope of the derivative curve, the duration of stage III of the process, and other factors disturbing the normal run of the curve, allows us to assess the alloy quality prior to its final use in the manufacture of castings.

An analysis of the dT/dt derivative curve, showing the rate of temperature drop during this period, helps in qualitative evaluation of the consequences of the alloy being contaminated with non-metallic inclusions and gas, as schematically shown in Fig. 2.

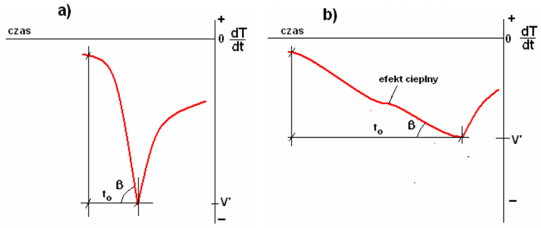


Fig. 2. The shape of dT/dt derivative curve in the last stage of solidification: a) high quality alloy, b) low quality alloy

3. Materials and methods of investigation

Studies of metallurgical quality and of the presence of non-metallic inclusions done by the method of ATD thermal analysis were conducted on master heat ingots and ready castings made from the IN-713C nickel alloy with an average content of 0.03% Co, 13.26% Cr, 5.85% Al, 4.10 % Mo, 0.85% Ti, 2.27% (Nb + Ta).

Examples of axial sections of "master heat" ingots are shown in Figure 3.

Fig. 3. Axial section of the IN-713C nickel alloy master heat ingot

Samples taken from these sections weighing about 1.2 kg were remelted in a Balzers VSG-02 induction furnace with an Al_2O_3 crucible in which, owing to the stability of technological parameters, high purity materials were produced. Melting was conducted in a vacuum of about 10^{-3}. Before pouring of moulds, the working space of the furnace was flushed with argon. Pouring was carried out in an argon atmosphere at a pressure of about 900hPa.

The shape of the test casting was a cylinder of φ30xx120 mm dimensions with a 40x45x17mm riser. The area of temperature measurement was designed at 1/3 of the casting height (counting from the bottom). The Pt-PtRh10 thermocouple wires 0.5 mm thick were enclosed in a quartz tube. Ceramic moulds adapted for tests were made by the investment process in WSK Rzeszów. A view of mould in the chamber of an induction furnace is shown in Figure 4.

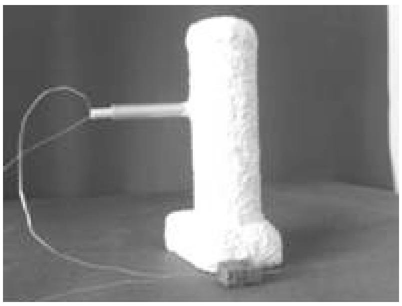


Fig. 4. Ceramic mould ready for ATD analysis

4. The results of investigations and discussion of results

Non-metallic inclusions in shrinkage cavities

Figure 5 is a picture of shrinkage cavities located in the axis of master heat ingots.

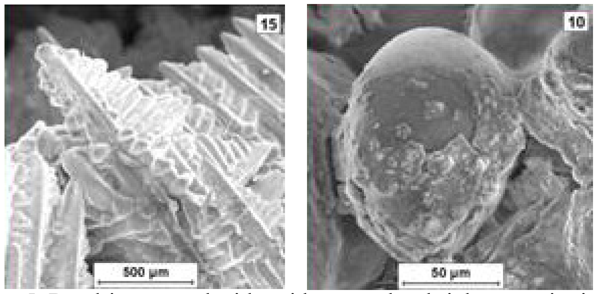


Fig. 5. Dendrites coated with oxides near the shrinkage cavity in a master heat ingot made from the IN-713C alloy

Observations of microstructure and chemical composition microanalysis were performed on a Hitachi S-3400N scanning electron microscope coupled with Thermo Noran EDS X-ray microanalysis system equipped with a System Six software. The results of these investigations for the four randomly selected areas of the shrinkage cavities are shown in Figures 6 to 9.

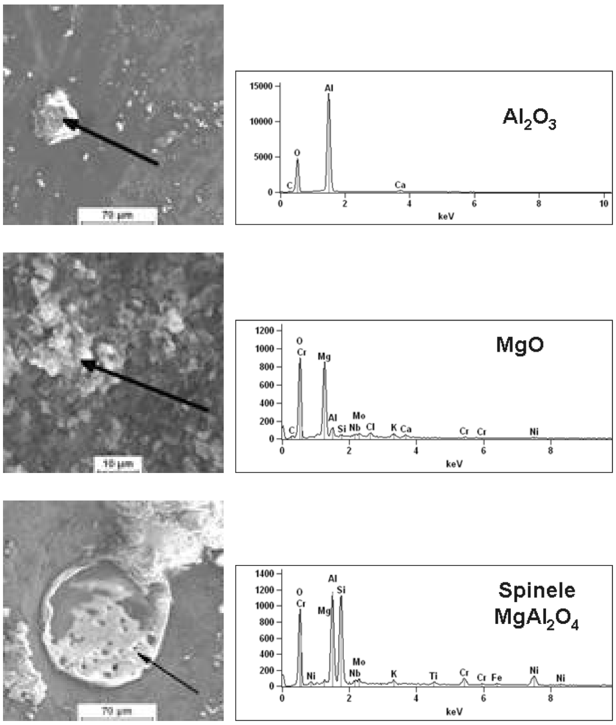


Fig. 6. Non-metallic inclusions on the surface of dendrites in shrinkage cavities

ATD thermal analysis

The ATD thermal analysis was carried out on fragments of a master heat ingot without the defects and with large shrinkage cavities, as shown in Figure 7.

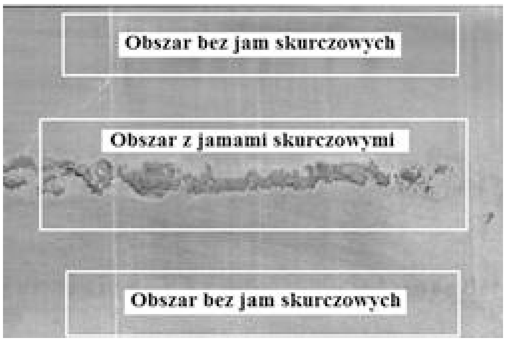
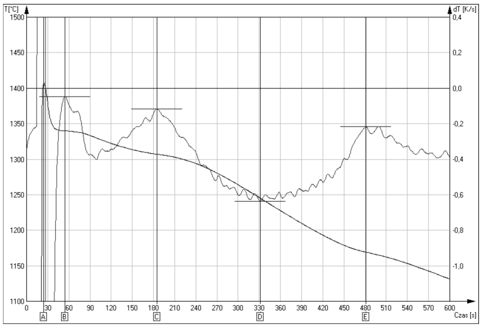


Fig. 7. Areas in master heat ingots where samples were taken for the ATD thermal analysis

The results of the ATD thermal analysis are shown in Figures 8 and 9.

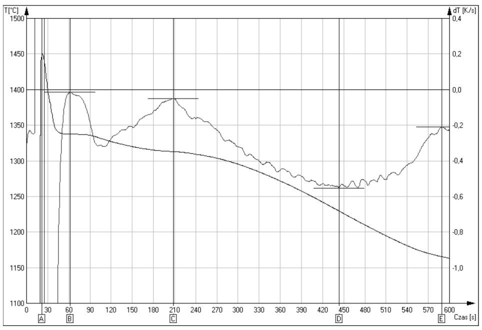


T_{max}	[A]	25 [s]	1408 [°C]
T_{lik}	[B]	55 [s]	1340 [°C]
T_E	[C]	185 [s]	1307 [°C]
T_{sol}	[D]	332 [s]	1245 [°C]
T_{ps}	[E]	482 [s]	1169 [°C]

$T_E - T_{sol} = 62^oC$

$t_D - t_C = 147s$

Fig. 8. ATD analysis curve plotted for the IN-713 C alloy (ingot without shrinkage cavities)



T_{max}	[A]	22,0 [s]	1450,0 [°C]
T_{lik}	[B]	62,0 [s]	1336,0 [°C]
T_E	[C]	209,0 [s]	1313,0 [°C]
T_{sol}	[D]	444,0 [s]	1230,0 [°C]
T_{ps}	[E]	590,0 [s]	1165,0 [°C]

$T_E - T_{sol} = 83^oC$

$t_D - t_C = 235s$

Fig. 9. ATD analysis curve plotted for the IN-713 C alloy (ingot with shrinkage cavities)

During melting, impurities of various types can penetrate to the liquid alloy, their source being usually:

- contaminated charge materials,
- ceramic material of the crucible,
- atmosphere in the furnace chamber (e.g. argon contaminated with oxygen),
- products of reaction taking place between the moulding material and liquid alloy, especially when moulds are poured at high temperature.

The technology of melting nickel alloys in closed melting chamber makes the process of refining, deslagging, etc. impossible. Therefore, any of the above mentioned factors may eventually lead to a contamination of nickel alloys with non-metallic inclusions or gas. Consequently, all these phenomena can contribute to the formation of shrinkage microporosities and non-metallic inclusions, especially on grain boundaries.

Most of the non-metallic inclusions are characterised by low solidification point, which makes them gather ahead of the solidification front and solidify as the last ones in stage III of the solidification process (Fig. 1).

An analysis of the dT/dt derivative curve showing the rate of temperature drop during this period helps in qualitative evaluation of the consequences of the alloy being contaminated with non-metallic inclusions and gas.

The presence of gas and impurities in the melt usually leads to the formation of low-melting point eutectics, which significantly prolong the final stage of solidification and reduce the value of the dT/dt derivative curve (the rate of temperature drop). The slope of the tangent to the derivative curve also changes, depending on the degree of contamination. When the slope angle has a low value, it means a high degree of the alloy contamination with low-melting point non-metallic inclusions.

When the slope angle has a high value (over 70°), it suggests a sufficient purity of the examined alloy.

The results of ATD analysis carried out on the examined alloys in stage III of the solidification process have been confirmed by observations of the surface of test ingots shown in Figure 10.

a) b)

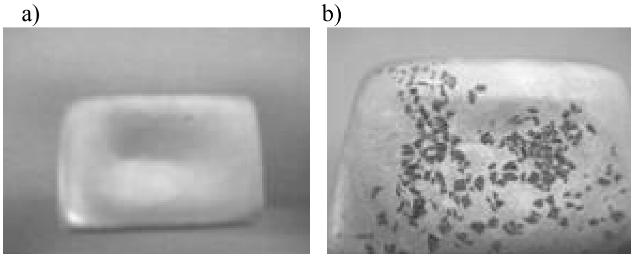


Fig. 10. Top surface of ingot cast in the ATD sampler: a) specimen taken in the area without shrinkage cavities, b) specimen taken in the area with shrinkage cavities

Microanalysis of precipitates on the surface of ATD cast ingots

Specimens taken from the surface of ATD cast ingots were subjected to an X-ray microanalysis and the results are shown in Figure 11.

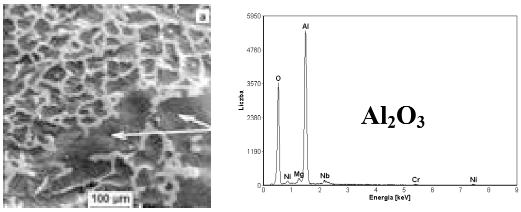


Fig. 11. X-ray microanalysis of dark spots on the surface of ingot cast in the ATD sampler

The dark, partially cracked film is probably Al_2O_3 with a small amount of magnesium.

The presence of non-metallic inclusions was also detected on the surface of a shrinkage cavity formed in ingot cast in the ATD sampler. The results of these studies are shown in Figures 12 and 13.

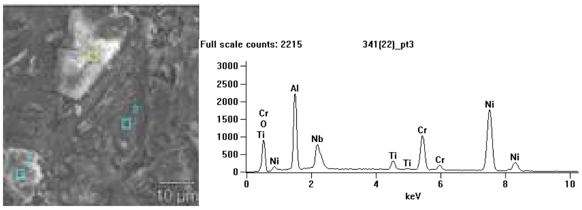


Fig. 12. Non-metallic inclusions of Al_2O_3 on the surface of a shrinkage cavity in ingot cast in the ATD sampler

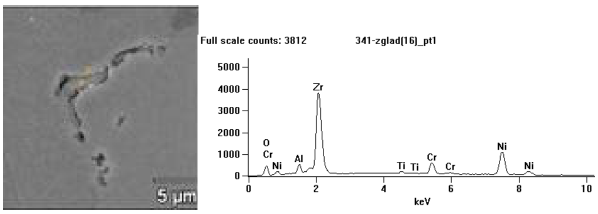


Fig. 13. Non-metallic inclusions of $Zr0_2$ on the surface of a shrinkage cavity in ingot cast in the ATD sampler

Analysis of the cast blades quality

Tests carried out on 238 blades cast from the IN 713C nickel superalloy showed that, compared to the blades cast from master heat ingots with shrinkage cavities, the blades cast from master heat ingots without the shrinkage cavities were characterised by a similar number of cracks, distinctly less numerous non-metallic and slag inclusions, lower lever of rejects, and the yield higher by 17%.

Sample analysis was conducted on the blade shown in Figure 14.

Fig. 14. General view of a fragment of the cast blade with marked places where the defects have occurred

The results of the X-ray microanalysis of precipitates present in the cast blade metal matrix are shown in Fig. 15.

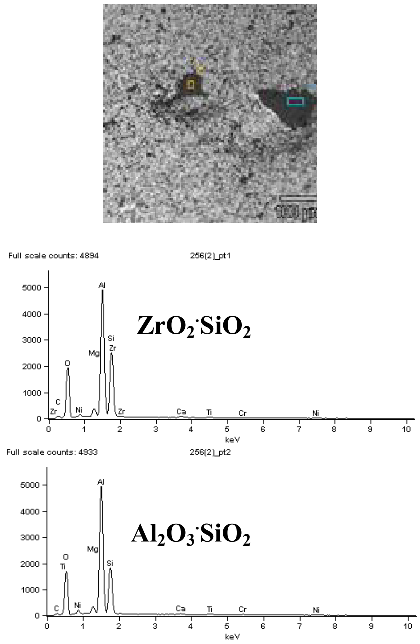


Fig. 15. The results of an identification of non-metallic inclusions present in cast blades

The results of the analysis carried out in another area of the cast blade are shown in Figure 16.

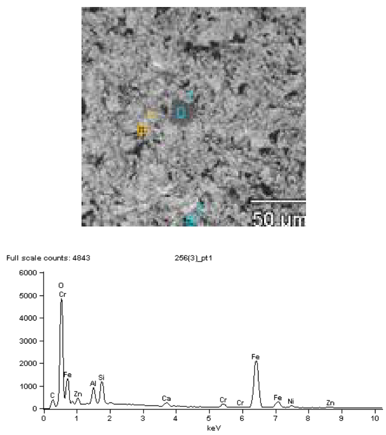


Fig. 16. Complex non-metallic inclusions containing Cr, Fe, Si, Al and Zn

As it follows from the conducted studies, the impurities present in master heat ingots during the initial period of melting are also present in the cast parts of aircraft engines (cast blades). Therefore, the ATD thermal analysis enables excluding from use the defective, i.e. contaminated, master heat ingots at an early stage of the technological process.

Proper interpretation of the ATD curves requires repeatable conditions maintained in a mould-molten alloy system, in particular:

- ceramic sampler standardised in terms of materials and dimensions,
- equal weight of the tested nickel alloy samples (~ 600g),
- similar pouring temperatures (from 1460 to 1480^{o}C),
- constant mould temperature ("cold" mould),
- temperature measured only with selected Pt-PtRh10 thermocouples.

Based on the obtained results, the shape and dimensions of a test ingot were established and adapted to the conditions necessary for evaluation of the solidification parameters and metallurgical quality of "master heat" ingots. The possibility of further adaptation of a Balzers induction furnace to the test conditions was also considered. The design of a test ingot is shown in Figure 17.

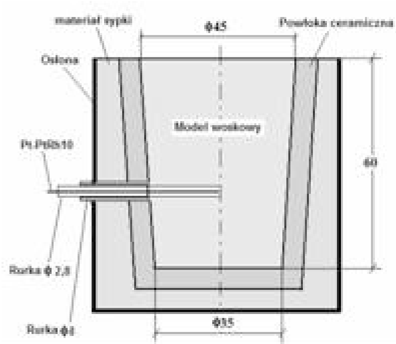


Fig. 17 Test ingot design adopted for the ATD thermal analysis under the conditions of melting in a Balzers vacuum furnace

Acknowledgements

Financial support of Structural Funds in the Operational Programme - Innovative Economy (IE OP) financed from the European Regional Development Fund - Project "Modern material technologies in aerospace industry",
Nr POIG.01.01.02-00-015/08-00 is gratefully acknowledged.

References

[1] Zupanic, F., Boncina, T., Krizman, A. & Tichelaar, F.D. (2001). Structure of continuously cast Ni-based superalloy Inconel 713C. *Journal of Alloys and Compounds.* 329 (1-2), 290-297.

[2] Tabuchi, M., Kubo, K., Yagi, K., Yokobori A.T. Jr, & Fuji, A. (1999). Results of a Japanese round robin on creep crack growth evaluation methods for Ni-base superalloys. *Engineering Fracture Mechanics.* 62 (1), 47-60.

[3] Chmiela B., Sozańska M., & Rodak K. (2012). Phase identification in nickel-based superalloys using EBSD/SEM and electron diffraction in STEM. *Solid State Phenomena.* 186, 58-61.

[4] Jura S., Sakwa J., & Borek K. (1980). Zastosowanie analizy termicznej i różniczkowej dla określenia parametrów składu chemicznego. Krzepnięcie Metali i Stopów. 2.

[5] Binczyk F., & Krzemień E. (1980). Zastosowanie różniczkowych krzywych krzepnięcia w badaniach krystalizacji żeliwa. In Mat. Konferencyjne Krzepnięcie Metali i Stopów: PAN, Mechanika 23, Gliwice.

[6] Jura S. (1989). Krzywa kalorymetryczna w analizie termicznej i derywacyjnej procesu krystalizacji metali i stopów. *Krzepnięcie Metali i Stopów.* 14.

[7] Pietrowski S. & Władysiak R. (1996). Kontrola metodą ATD siluminów tłokowych. *Krzepnięcie Metali i Stopów.* 28.

[8] Zielińska M., Sieniawski J. & Poreba M. (2007). Microstructure and mechanical properties of high temperature creep resisting superalloy Rene 77 modified $CoAl_2O_4$. *Archives of Materials Science and Engineering.* 28 (10), 629-632.

[9] Zielińska M., Sieniawski J. & Wierzbińska M. (2008). Effect of modification on microstructure and mechanical properties of cobalt casting superalloy. *Archives of Metallurgy and Materials.* 53 (3), 887-893.

[10] Binczyk, F. & Śleziona J. (2010). Effect of modification on the mechanical properties of IN-713C alloy. *Archives of Foundry Engineering.* 10 (1), 195-198.

[11] Binczyk F. & Śleziona J. (2010). Mechanical properties and creep resistance behaviour of IN-713C alloy castings. *Archives of Foundry Engineering.* 10 (4), 9-13.

[12] Binczyk F., Śleziona J. & Gradoń P. (2011). Modification of the macrostructure of nickel superalloys with cobalt nanoparticles. *Composites.* 49-55.

[13] Binczyk, F., Śleziona, J. & Gradoń P. (2011). Ceramic filters for bulk inoculation of nickel alloy castings. *Archives of Foundry Engineering.* 11 (Special Issue 3), 29-33.

[14] Binczyk, F., Śleziona, J., Szymszal, J. & Gradoń P. (2011). Effect of technological parameters on structure of castings made from IN-713C nickel alloy. *Archives of Foundry Engineering.* 11 (4), 9-13.

[15] Binczyk, F. Śleziona, J. & Mikuszewski T. (2010). Effect of repeated remelting on the chemical composition and structure of nickel alloys. *Archives of Foundry Engineering.* 10 (Special Issue 1), 189-194.

[16] Binczyk F., Śleziona J., Cwajna J. & Roskosz S. (2008). ATD and DSC analysis of nickel super alloys. *Archives of Foundry Engineering.* 8 (3), 5-9.

[17] Binczyk, F., & Śleziona J. (2010). The ATD thermal analysis of selected nickel superalloys. *Archives of Foundry Engineering.* 10 (2), 13-19.