A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 57 2012 Issue 2
DOI: 10.2478/v10172-012-0059-7

B. CHMIELA*, M. SOZAŃSKA*, J. CWAJNA*

IDENTIFICATION AND EVALUATION OF FRECKLES IN DIRECTIONALLY SOLIDIFIED CASTING MADE OF PWA 1426 NICKEL-BASED SUPERALLOY

IDENTYFIKACJA I OCENA WADY FREKLE W KIERUNKOWO KRYSTALIZOWANYM ODLEWIE Z NADSTOPU NIKLU PWA 1426

Manufacturing of modern aero engine turbine blades made of nickel-based superalloys is very complex and expensive. The thrust and performance of new engines must address constantly more demanding requirements. Therefore, turbine blades must be characterised by very good mechanical properties, which is possible only if the blades are free of casting defects. An important innovation has been the launching of directionally solidified (DS) and single crystal (SX) turbine blades. But, manufacturing procedures and the chemical composition of many superalloys promote the formation of casting defects that are characteristic only for directional solidification. One of these defects is freckles. Freckles are small equiaxed grains in the form of long chains parallel to the solidification direction and are located on the surface of the casting. Freckles decrease the mechanical properties of DS and SX blades; therefore, they should be always unambiguously identified to improve the manufacturing process. This work presents the possibilities of identifying and evaluating freckles in DS casting made of PWA 1426 superalloy by combining the scanning electron microscopy (SEM), electron probe microanalysis (EPMA) and electron backscatter diffraction (EBSD) techniques.

Keywords: freckles, EBSD, nickel-based superalloys, directional solidification

Wytwarzanie łopatek turbin nowoczesnych silników lotniczych jest bardzo złożone i kosztowne. Stale rosną też wymagania dotyczące ciągu i sprawności nowych silników. Dlatego łopatki turbin muszą charakteryzować się bardzo dobrymi właściwościami mechanicznymi. Jest to możliwe tylko wtedy, gdy łopatki nie wykazują wad odlewniczych. Ważną innowacją w wytwarzaniu łopatek było wprowadzenie łopatek kierunkowo krystalizowanych (DS) i monokrystalicznych (SX). Skład chemiczny wielu nadstopów i proces wytwarzania łopatek stanowią czasem czynniki sprzyjające powstawaniu wad odlewniczych, charakterystycznych tylko dla procesu kierunkowej krystalizacji. Przykładem takiej wady są frekle. Wada ta ma postać małych, równoosiowych ziaren tworzących łańcuchy, przeważnie równoległe do kierunku krystalizacji. Wada ta znacznie obniża właściwości mechaniczne łopatek DS i SX, dlatego powinna być zawsze jednoznacznie identyfikowana w celu usprawnienia procesu ich wytwarzania. W pracy przedstawiono możliwości identyfikacji i oceny wady frekle w kierunkowo krystalizowanym odlewie z nadstopu niklu PWA 1426 przy użyciu elektronowej mikroskopii skaningowej (SEM), mikroanalizy rentgenowskiej (EPMA) i dyfrakcji elektronów wstecznie rozproszonych (EBSD).

1. Introduction

Aero engines turbine blades are flight safety parts. They are manufactured by investment casting method using ceramic moulds [1]. The highest mechanical properties of turbine blades are obtained in case of directional solidification. Directionally solidified (DS) blades produced by Bridgman technique are prone to many casting defects and the common defects are freckles.

Freckles are chains of equiaxed grains with random orientations and enriched with elements that segregate to the interdendritic liquid (Al, Ti, Ta, Nb) during directional solidification [2÷9]. Freckled areas are characterised by higher contents of carbides, $(\gamma + \gamma')$ eutectics and porosity in comparison to other freckle-free parts of the casting. Freckles always appear in the interdendritic areas and are visible on the casting surface. Therefore identification of freckles is usually simple in case of large castings without any instruments, because they are clearly visible [2]. But in case of small castings, identification may be difficult and it is necessary to apply the chemical etching and light microscopy investigations [3]. The average size of these defects ranges from one to several millimeters in width, but the length is usually

* SILESIAN UNIVERSITY OF TECHNOLOGY, DEPARTMENT OF MATERIALS SCIENCE AND METALLURGY, 40-019 KATOWICE, POLAND

the same as the casting length. Many investigations revealed that the number of freckle chains increases with increasing casting size, and they are evenly distributed on the casting surface. Moreover, the number of freckle chains decreases with the distance from the chill plate as a result of the joining of neighbouring chains [2, 3].

Analysis of the reasons for freckle formation reveals that these defects result from the interaction among the following factors: strong segregation of some elements (W, Re) to the solid phase, which causes density inversion in the interdendritic areas; density inversion, which then creates buoyancy, and convection, which is due to the interdendritic liquid density difference caused by the temperature gradient [2÷9].

During directional solidification using the Bridgmann technique, tungsten and rhenium segregate to dendrite cores, but aluminium, titanium, tantalum and niobium segregate to the interdendritic liquid. Other elements such as chromium, cobalt or ruthenium do not display a strong tendency for segregation, and they are present in the dendrite cores and the interdendritic liquid [2÷10]. When the content of W and Re is high, during solidification, the liquid alloy is depleted of these elements in the interdendritic areas and is characterised by a density lower than that for the liquid alloy at the melting point. This phenomenon is called density inversion. A high concentration of tantalum decreases density inversion and simultaneously decreases the buoyancy working on for the liquid in the mushy zone. Similar effects cause an adequate reduction in the concentrations of W and Re. The result of density inversion is the development of buoyancy, causing the liquid alloy to move up, in the opposite direction of the thermal gradient. Due to the thermal gradient, the difference in temperature between the lower part of the casting in the mushy zone and the upper part in the liquid zone contributes to the development of convection. Mass transport occurs at a considerably lower degree than the diffusion of heat. In the mushy zone the liquid alloy (with a slightly changed composition) moves upward when the buoyancy is greater than the resistance to flow. Then, the liquid alloy flowing through the mushy zone is able to dissolute of the secondary dendrite arm tips, what causes the nucleation of new equiaxed grains–freckles. The resistance of the interdendritic liquid to flow increases with smaller primary dendrite arm spacing λ_1 (PDAS).

An adequate chemical composition minimizes the freckling tendency. Susceptibility to freckle formation is characterised by the freckling index F, which is connected to the chemical composition of the superalloys [10]:

$$F = \frac{C_{Ta} + 1.5C_{Hf} + 0.5C_{Mo} - 0.5C_{Ti}}{C_W + 1.2C_{Re}} \qquad (1)$$

where C_{Ta}, C_{Hf}, C_{Mo}, C_{Ti}, C_W and C_{Re} are the concentrations of the elements: tantalum, hafnium, molybdenum, titanium, tungsten and rhenium, respectively (in wt. %).

When the value of F is greater than 1, the probability of freckle formation is low.

The guidelines regarding modern superalloy chemical compositions that decrease the frequency of freckle formation is presented in U. S. patent No. 6096141 [11]. According to this patent, the concentration of the elements in nickel-based superalloys should be as shown in Table 1. Moreover, the total amount of Al and Ta should be greater than 12.45 wt. %.

TABLE 1

Concentration of elements in nickel-based superalloys decreasing the tendency of freckling [11]

Element	Concentration (wt. %)	Element	Concentration (wt. %)
B	0.003 – 0.01	Mo	0 – 2
C	0.1 – 0.15	Ru	0 – 6
Al	5 – 7	Hf	0.1 – 0.3
Cr	4 – 7.25	Ta	6 – 9.25
Co	7 – 15	W	4.75 – 6.5
Nb	0 – 1	Re	2.75 – 6.4

Two of the most important ways to either prevent freckle formation or considerably decrease the frequency of freckle appearance are adequately changing the solidification parameters (sufficient withdrawal rate and thermal gradient) and preventing freckle formation by slightly modifying the chemical composition [2÷9]. However, according to some authors, the most promising way is to increase the carbon content in the alloy [4]. Then, carbides with the script morphology fill the interdendritic areas, limiting the possibility of interdentritic liquid flow in the mushy zone. As a result, the probability of freckle formation is much lower.

2. Materials and experimental procedure

An experimental directionally solidified casting made of PWA 1426 nickel-based superalloy was used in this study. The casting was made in the Research and Development Laboratory for Aerospace Materials of Rzeszów University of Technology. The withdrawal rate was $5 \cdot 10^{-5}$ m·s^{-1}. The chemical composition of the alloy is shown in Table 2.

To perform investigations of the freckles, a part of the casting containing this defect was carefully cut out. Then the longitudinal section of this part near the casting surface was taken and it was cut in several specimens.

TABLE 2
Chemical composition of PWA 1426 superalloy (wt. %)

Ni	Cr	Co	Mo	W	Ta	Al	C	B	Zr	Hf	Re
bal.	6.5	12.5	1.7	6.4	4	6	0.1	0.015	0.03	1.5	3

The specimens were ground and polished according to a procedure that was slightly modified from the one described earlier [12] (diamond suspension: grit sizes of 9 μm, 3 μm, 1 μm and 0.25 μm; alumina suspension: 0.05 μm). The last stage of specimen preparation was vibratory polishing (alumina suspension: 0.05 μm grit size) for 5 hours to obtain as low surface roughness as possible. The microstructure, chemical composition and crystallographic orientation were characterised using a scanning electron microscope (SEM, Hitachi S-3400N) equipped with an energy dispersive spectrometer (EDS) Thermo NORAN (System Six) and electron backscatter diffraction (EBSD) detector INCA HKL Nordlys II (Channel 5). First, EBSD analysis was carried out. To locate the freckled area, the forescatter electron image (FSE) with orientation contrast was used. Then, an orientation map including the freckled area and neighbouring area of the casting was made. The next stage was EDS analysis to confirm the characteristic changes of the freckle chemical composition. To reveal the microstructure, the specimens were etched in a solution with the following composition (vol. %): 33% CH_3COOH, 33% HNO_3, 33% H_2O and 1% HF. The etched specimens were metallographically examined using an SEM.

3. Results and discussion

On the basis of the chemical composition of PWA 1426 superalloy, the freckling index was calculated. The value of F was 0.71, which was much lower than 1. This value indicates that the fraction of elements segregating to the liquid is too small to effectively counteract density inversion. Therefore, on the basis of the F value, the PWA 1426 superalloy will be prone to freckles.

The structure of the casting surface is shown in Fig. 1. In the freckle-free area properly formed primary and secondary dendrite arms were visible (Fig. 1a). But, in the freckled area dendrite arms were much shorter and displayed various orientations (Fig. 1b), which were connected to the various orientations of the freckle grains.

The primary dendrite arm spacing was about 500 μm. The freckled area was characterised by high porosity (Fig. 2a) and a high number of $(\gamma+\gamma')$ eutectics (Fig. 2b).

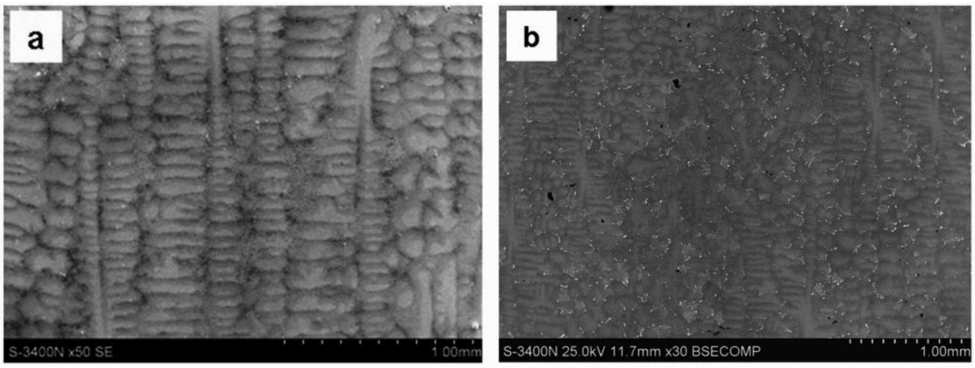


Fig. 1. Dendritic structure on the longitudinal section of the casting made of PWA1426 superalloy: (a) proper structure and (b) defective structure with freckles

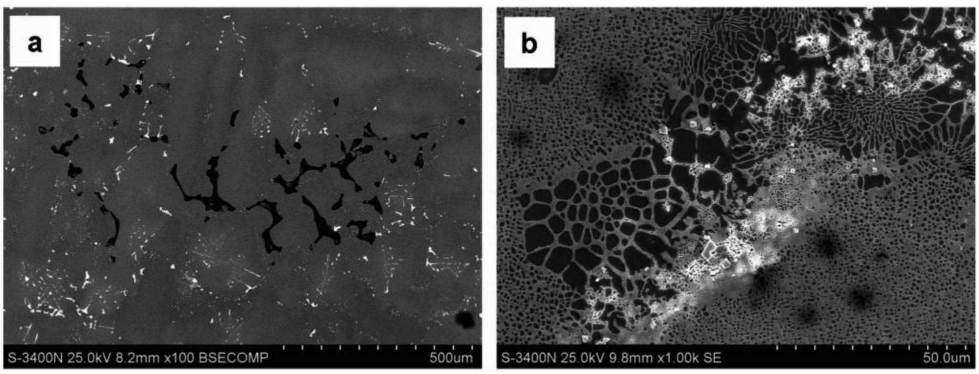
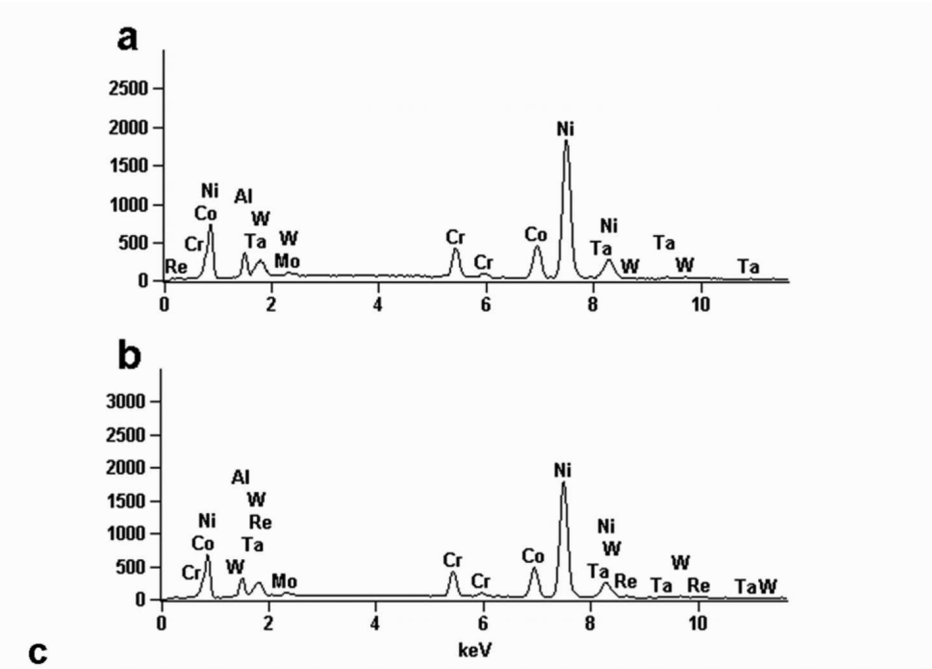


Fig. 2. Porosity (a) and eutectics (b) in freckled area



c

Element	Al	Cr	Co	Ni	Mo	Hf	Ta	W	Re
freckled area	6.2	6.7	10.8	59.8	1.8	1.9	5.2	5.2	2.4
freckles-free area	5.8	6.6	12.4	58.8	1.8	1.6	3.9	6.3	3

Fig. 3. EDS analysis of freckled (a) and freckle-free areas (b); chemical composition (wt. %) of investigated areas (c)

EDS analysis performed in areas visible in Fig. 1 confirmed changes in the chemical composition connected with the segregation of some elements to the liquid during solidification (Fig. 3). Freckles were enriched with aluminium and tantalum in comparison to other parts of the casting. Tantalum is an element with high density, but its concentration in the investigated alloy (4 wt. %) is insufficient to prevent density inversion. The density of the interdendritic liquid is thus too low, and the buoyancy forces the liquid to move up.

EBSD analysis provided a detailed description of the freckles. Fig. 4a presents a band contrast image with the grain boundaries revealed. Boundaries with a misorientation in the range of 1° ÷ 5° are highlighted with a thin line; boundaries with a misorientation in the range of 5° ÷ 10° are shown using a medium-thick line; and boundaries with a misorientation greater than 10° are shown using a thick line. The misorientation distribution of the grain boundaries reveals that a greater fraction displays boundaries with a misorientation in the range of 1° ÷ 5° and 30° ÷ 35° (Fig. 4b). The solid line in the histogram presents the distribution due to random orientation (MacKenzie plot).

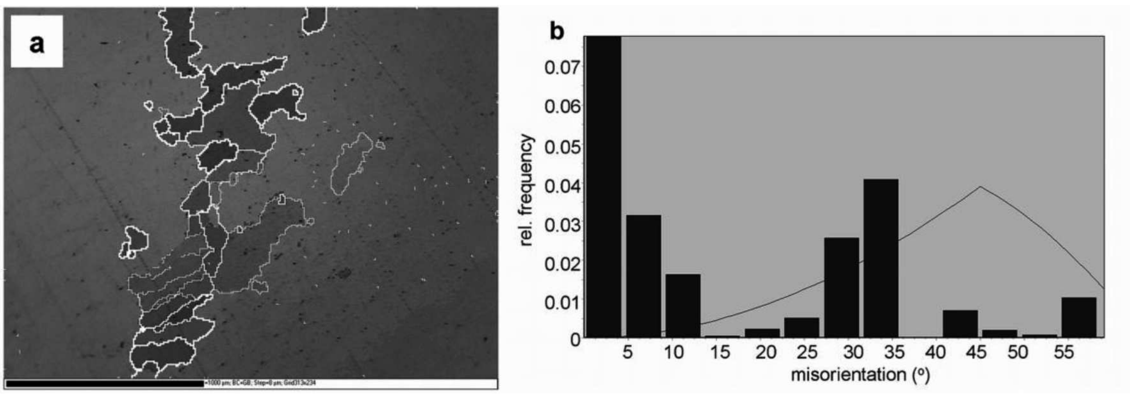


Fig. 4. Freckle grain boundaries (a) and misorientation distribution (b)

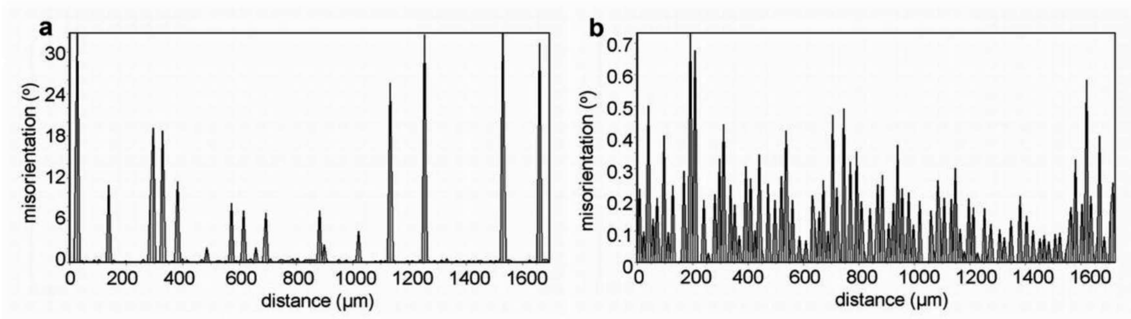


Fig. 5. Misorientation profiles in the freckled area: (a) freckles grains and (b) freckle-free area

Misorientation profiles in the freckled area confirmed the presence of high angle boundaries (Fig. 5a). But, in the freckle-free parts of the casting, the misorientation did not exceed 1° (Fig. 5b), which is typical for directionally solidified and single crystal castings made of superalloys.

Analysis of the freckle orientations revealed that some grains were characterised by an orientation close to that of the whole casting, but some grains displayed another orientation (Fig. 6). It is believed that grains with an orientation similar to the casting form as a result of nucleation in the area where resistance of the interdendritic liquid to flow is high. Therefore, these nuclei do not have an opportunity to freely rotate. These grains form low angle boundaries with the casting (misorientation of 1° ÷ 5°).

The remaining grains were characterised by random orientations (Fig. 6 and Fig. 7), which could indicate easy rotation for the nuclei during interdendritic liquid flow. These grains form high angle boundaries with the casting (misorientation of 30° ÷ 35°).

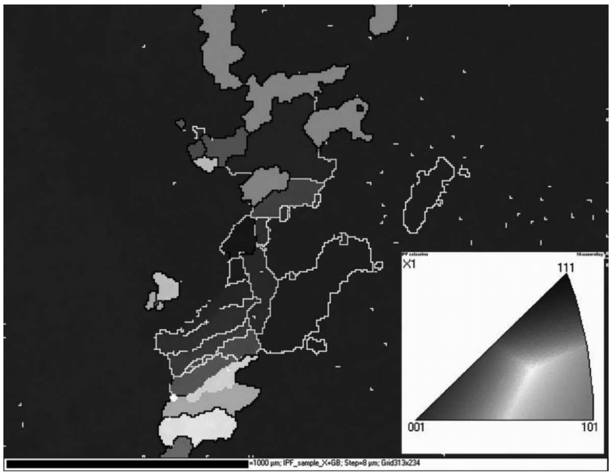


Fig. 6. Orientation map of the freckled area (IPF colouring)

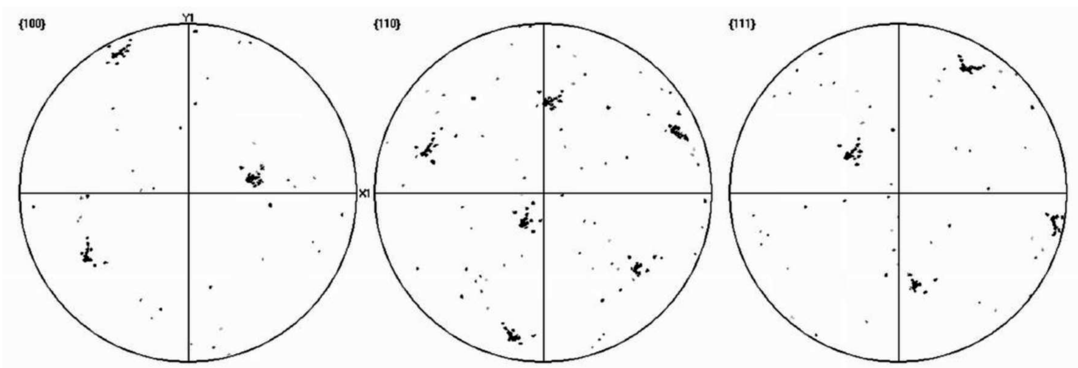


Fig. 7. Pole figures of freckled area

4. Conclusions

Freckles formed in the investigated casting result from the combination of many factors. An important factor is the chemical composition of the alloy–especially a too small concentration of tantalum in PWA 1426 superalloy. Although tantalum segregates to the interdendritic liquid during solidification, its concentration is insufficient to compensate for density inversion. Also important are incorrect values of the withdrawal rate and thermal gradient applied to the size of the casting and the type of alloy. The withdrawal rate used in this investigation was too small, which caused the primary dendrite arm spacing to be too large and thus facilitated the flow of the interdendritic liquid flow. To avoid the formation of freckles using PWA 1426 superalloy, it is necessary to apply a higher withdrawal rate and larger thermal gradient.

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", No. POIG.01.01.02-00-015/08-00 is gratefully acknowledged.

Received: 20 November 2011.

REFERENCES

[1] A. Onyszko, K. Kubiak, Archives of Metallurgy and Materials **54**, (2009).
[2] A.F. Giamei, B.H. Kear, Metallurgical Transactions **1**, 2185 (1970).
[3] E.M. Mueller, The characterization of freckle casting defects in directionally solidified nickel-base superalloy turbine blades, MSc Thesis, University of Florida, (2003).
[4] K.A. Al-Jarba, G.E. Fuchs, Materials Science and Engineering A **373**, 255 (2004).
[5] W. Yang, W. Chen, K. Chang, S. Mannan, J. deBarbadillo, Metallurgical and Materials Transactions A **32A**, 397 (2001).
[6] S. Tin, T.M. Pollock, Journal of Material Science **39**, 7199 (2003).
[7] C. Beckermann, J.P. Gu, W.J. Boettinger, Metallurgical and Materials Transactions A **31A**, 2545 (2000).
[8] P. Auburtin, T. Wang, S.L. Cockroft, A. Mitchell, Metallurgical and Materials Transactions A **31B**, 801 (2000).
[9] S. Tin, T.M. Pollock, W. Murphy, Metallurgical and Materials Transactions A **32A**, 1743 (2001).
[10] R.C. Reed, The Superalloys. Fundamentals and Applications, Cambridge University Press, New York (2006).
[11] W.T. King, T.M. Pollock, C.L. Zemsky, W.H. Murphy, U. S. Patent No. 6096141, (2000).
[12] A. Szczotok, B. Chmiela, M. Sozańska, Inżynieria Materiałowa **3 (175)**, 695 (2010).