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Original Research

„Above the Pack" Diffusion Aluminizing of Turbine Compressor Blades made of EI867 in the Aerospace Industry

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Abstract

In the publication, tests were conducted on compressor turbine working blades made of EI-867 material, in accordance with the TU 14-1-402-72 standard, which were subjected to the gas non-contact aluminizing process. Metallographic analyses of the produced aluminide layer were undertaken, and the phase components of the aluminide layer microstructure were identified. This identification was achieved by analyzing the chemical composition in micro-areas using the EDS attachment in a scanning electron microscope and using X-ray diffraction. Additionally, hardness and creep resistance tests of the blades, after undergoing solution and aging processes, were performed over different durations. The research was aimed at exploring the feasibility of the aluminizing process using the "above the pack" method on parts made of EI-867 material and understanding the process's impact on the creep resistance of the part. Experimental research have shown that aluminizing turbine blades with EI-867 using the "above the pack" method to obtain a layer thickness in the range of 0.03-0.06 mm is possible within 10 hours at 950ºC. Aluminizing with the analyzed method results in the formation of an aluminized layer with a three-phase structure that ensures the appropriate strength of the coating. However, it has been shown that the aluminizing process using the "above the pack" method with the applied time of 10 hours causes a significant reduction in the creep resistance of the material. Based on the obtained results, it was shown that the non-contact aluminizing method for turbine blades made of EI 867 material does not meet aviation requirements for safe operation due to a significant reduction in mechanical properties.

Keywords: diffusion aluminizing, compressor turbine blades, EI867 alloy, turbine engine, compressor turbine

1. Introduction

Various types of damage to turbine components may occur during operation. The most vulnerable element of the turbine is the blades. The technical condition of which significantly influences the durability and operational reliability of both the turbine and the entire engine (Albrecht, 2017). According to literature, the primary causes of blade damage are creep, overheating, and thermal fatigue of the material (Błachnio et al., 2014; Szczepankowski & Szymczak, 2016). This can be attributed to manufacturing defects, such as using insufficiently durable heat-resistant coatings or improperly applying them to the blade material (Błachnio et al., 2014). The selection of the protective coating and the material to manufacture the blade with the desired durability must consider its mechanical and thermal properties in the highest temperature zones (Błachnio, 2011).

Heat-resistant alloys, especially those used for turbine components in aerospace production, endure very high temperatures (around 1000ºC) (Golewski, 2015; Zasada et al., 2016). To augment their resistance to atmospheric and gas corrosion at these temperatures, thermo-chemical treatments, which involve diffusion saturation of the surface layer with aluminum, are used. Applying the aluminum coating enhances the parts' heat resistance, extending their service life (Godzimirski, 2011). In modern metallurgy, there are numerous aluminizing methods. It is crucial that the chosen method not only achieves the desired results after the aluminizing process but also maintains the mechanical properties of the base material. The EI 867 material is a heat-resistant nickel-based superalloy melted and casted in vacuum furnaces. Its chemical composition should adhere to standard TU 14-1-402-72 for EI 867 or TU 14-1-223-72 for EI 867 WD (Table 1). The EI-867 WD alloy falls within the group of nickel matrix superalloys that do not contain titanium. Notably, this alloy has a lower chromium content than other superalloys, rendering it more susceptible to corrosion. Consequently, components made from these alloys necessitate protective coatings, specifically aluminum layers.

Table 1. The chemical composition of the EI867 alloy (МЧМ, 1972).

Element	Min, wt.%	Max, wt.%
C	-	0.10
Si	-	0.60
Mn	-	0.30
Cr	8.5	10.5
Ni	remainder	
Al	4.2	4.9

In order to obtain the highest possible properties of the EI867 alloy, the alloy is subjected to heat treatment, i.e. precipitation hardening. This treatment consists in solution solution at 1220±10ºC for 4 to 6 hours and aging at 950±15ºC for 8 hours (МЧМ, 1972). Solution solution causes dissolution of some carbides and intermetallic phases in the matrix. On the other hand, in the aging process, dispersion precipitates of the above-described γ' coherent phase and carbides are formed, evenly distributed in the matrix. The annealing is carried out in an inert atmosphere or sometimes in a vacuum. The purpose of precipitation hardening is to obtain a structure in which the size, shape and arrangement of the phases strengthening the alloy will ensure optimal mechanical properties and maximum stability of the structure at the operating temperature. In order to obtain a heat-resistant coating on the EI867 alloy, a powder aluminization process is used. The parts are covered directly with a powder consisting of a mixture of ferroaluminum and ammonium chloride. The standard process is carried out at a temperature of 950ºC for 2 to 4 hours, depending on the requirements of the depth of the aluminized layer. Due to the guidelines of the TU 14-1-402-72 standard for EI 867, the technological process should be arranged in such a way that it does not last longer than 8 hours at 950ºC. The use of protective coatings provides more effective protection against heat loads and protection against the influence of the environment (Golewski, 2015). At the same time, it affects the possibility of increasing the operating temperature of the most thermally loaded engine components. These coatings should be characterized primarily by low thermal conductivity and high structural stability (Godzimirski, 2011). In order to reduce the brittleness of the aluminized layer caused by the formation of intermetallic phases and to increase the thickness of the layer, additional diffusion annealing is applied after aluminizing at 900÷1050°C for 3 to 5 hours.

Due to the continuous development of the aviation industry and the ever-higher guidelines for the acceptance of parts subject to thermal stress during operation, trials of diffusion aluminizing using the "above the pack" method of the EI 867 material were carried out. A semi-finished product (forgings) used for the production of working blades of the compressor turbine made of the EI 867 alloy was used for the tests.

2. Methodology and results

The initial phase of the research involved aluminizing samples, specifically blades made of the EI-867 (HN62MWKJu) alloy, using the non-contact gas process, also known as "above the pack." The objective was to achieve an aluminized layer thickness within the range of 0.03 to 0.06 mm. This thickness range would ensure a heat-resistant coating that is operationally safe. Three aluminizing experiments were conducted, with their parameters detailed in Table 2. For each experiment, two samples (blades) and 20 additional blades (serving as ballast) were used.

The conventional aluminizing process operates at 1000ºC. However, due to the restriction of not exceeding the 950ºC parameter (as mandated by the TU 14-1-402-72 standard aging temperature) for the EI867 material, the aluminization experiments were performed at 950ºC. Because of this temperature reduction, the process duration had to be extended to 10 hours to achieve the desired thickness for the aluminized layer. The microstructure of the obtained layer is shown in Figures 1 and 2.

Table 2. Parameters of the EI 867 aluminizing tests with the results of the main aluminized layer.

Test	Temperature, °C	Heating time, °C/min	Hold time, h	Layer depth, mm
1	950	2÷6	4	0.010÷0.048
2	950	2÷6	8.5	0.028÷0.040
3	950	2÷6	10	0.033÷0.044

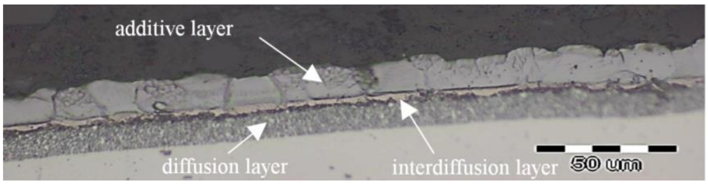


Fig. 1. Microstructure of the aluminized layer showing the in gray and orange; non-etched microsection of the blade's suction side.

Fig. 2. Microstructure of the aluminized layer; non-etched microsection treated with Kalling's reagent (blade's suction side).

Analyses of the aluminized layer were conducted using the OLYMPUS GX51 microscope, serial no. 8R14922, with a ×500 magnification. A sample for analysis was sourced from a cross-section of a blade. This metallographic specimen was etched using Kalling's reagent. The structure of the aluminum layer is influenced by various factors, including the aluminum's activity in the mixture, the substrate's chemical composition, and the aluminizing temperature and duration. Aluminide layers formed on a nickel based alloy’s substrate (rich in other alloying elements that enhance heat and high-temperature corrosion resistance) typically exhibit a triphasic structure. This is attributed to two opposing diffusion flows: a flow of aluminum atoms from the atmosphere and another of atoms, including nickel, moving from the substrate. The diffusion of nickel and certain alloying elements from the substrate towards the surface leads to their depletion, culminating in the creation of what is known as the interdiffusion layer (Fig. 3). Concurrently, nickel diffusion encourages the development of intermetallic phases and carbides in the intermediary layer, establishing a barrier against aluminum's diffusion into the substrate. The depth of the obtained layer is shown in Figure 4.

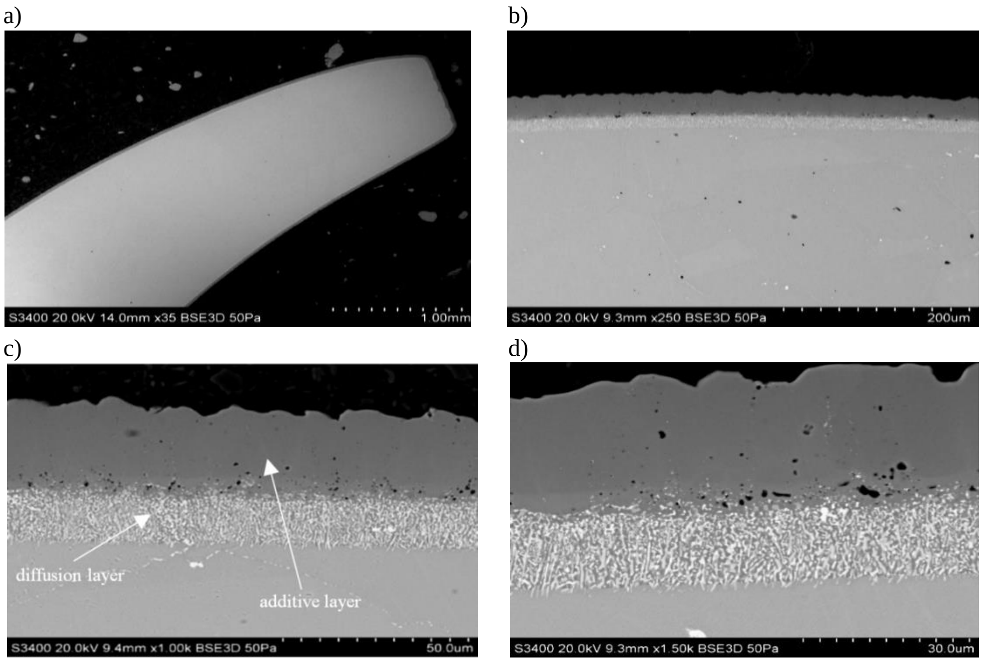


Fig. 3. a)-f) Microstructure of the aluminide layer formed in the thermochemical non-contact gas process (“above the pack”) on the surface of an EI-867 alloy blade.

e) f)

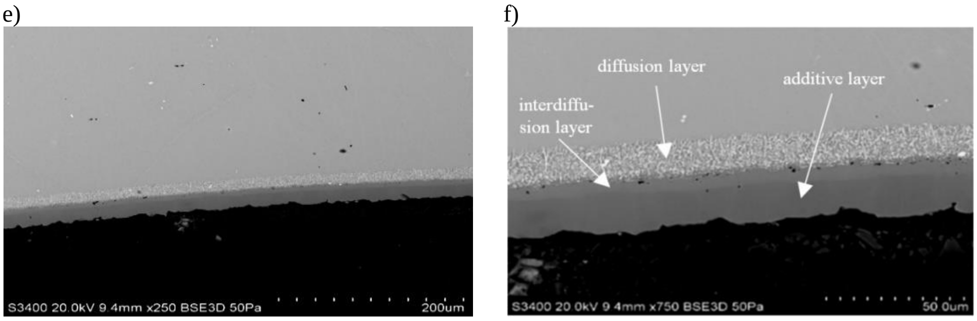


Fig. 3. *Cont.*

a) b)

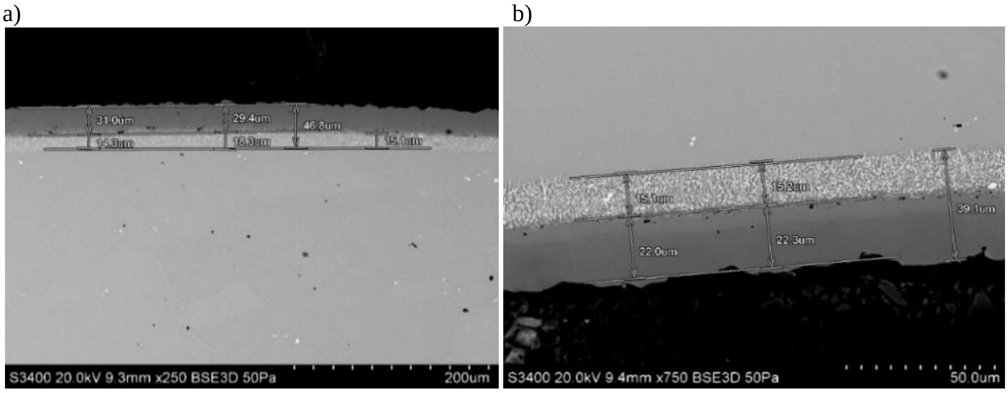
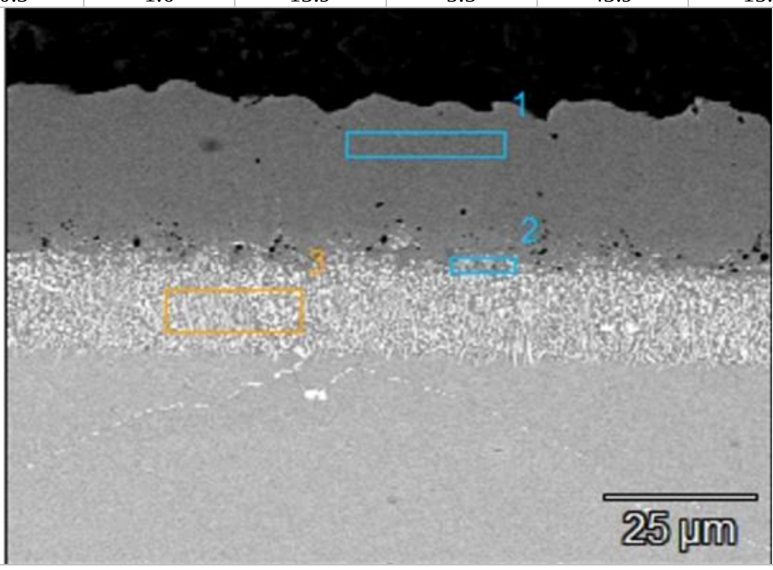


Fig. 4. Microstructure of the aluminide layer formed in the thermochemical process ("above the pack" method): a) suction side, b) pressure side – leading edge. The depth of the layer is indicated.

Table 3. Chemical composition of micro cross-sectional areas of the aluminide layer produced on the base of the blade in the thermochemical process using the "above the pack" method

Point	*Al-K*	*Si-K*	*Cr-K*	*Co-K*	*Ni-K*	*Mo-L*	*W-L*
1	32.3	2.6	1.8	2.5	60.8		
2	14.4	2.2	18.4	3.2	45.9	9.8	6.1
3	10.5	1.6	13.9	5.5	43.9	15.7	9.0



The phase components of the microstructure of the aluminide layer after the gas-free contact aluminizing process were identified by analyzing the chemical composition in micro-areas using the EDS attachment in a scanning electron microscope and by X-ray diffraction. The results of quantitative and qualitative (Fig. 5, Table 3) tests allow to conclude that, in accordance with the assumptions made, an aluminide layer consisting of the expected NiA phase was formed on the blade surface from the EI 867 superalloy substrate, ensuring its good heat resistance properties. XRD studies confirmed the presence of a stable β-NiAl phase in the aluminide layer (Fig. 6).

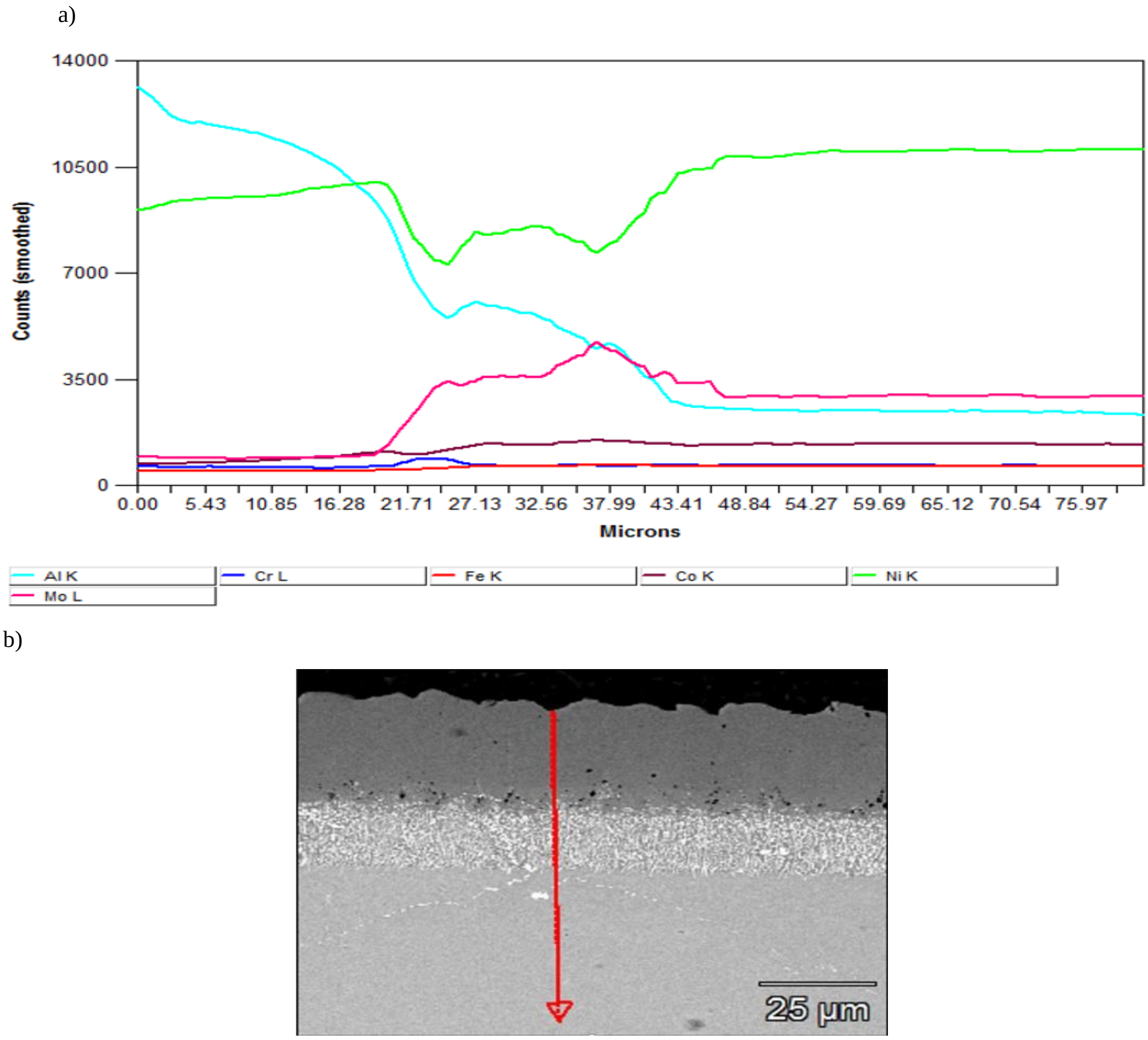


Fig. 5. a) Linear profile of changes in the content of elements along a given scanning line on b) the cross-section of a blade with an aluminide layer produced in the aluminizing process using the "above the pack" method.

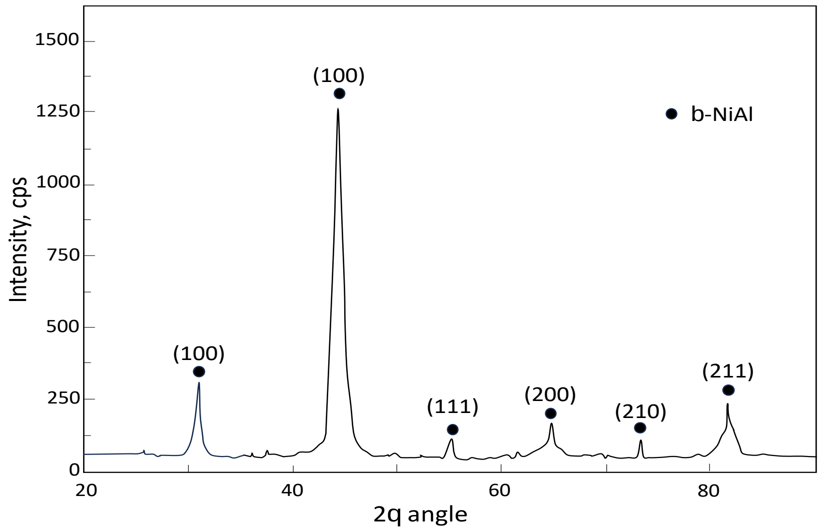


Fig. 6. XRD pattern of the aluminide layer deposited on the examined blades.

Extending the holding time at 950ºC affects the strength properties of the material working at elevated temperatures. Therefore, it was necessary to carry out control tests confirming the effect of extending the time on the creep resistance of the EI867 material. In the case of the turbine blades analyzed in the publication, apart from the need to have a heat-resistant layer, the parts must have a creep resistance of at least 50 hours due to aviation requirements for safe operation. 10 samples (semi-finished product in the form of forgings for turbine blades) were used for the tests. All samples underwent the solution treatment process for 4 h at 1220±10ºC. Then, the aging process was carried out within the endurance range of 9 to 18 hours (according to the TU 14-1-402-72 standard, the endurance time is 8 hours).

The assumptions of the creep resistance test assumed conducting the test until the sample broke. The requirement of the test was not to break the sample before the period of 50 hours. According to the assumptions of the Technical Conditions, this time was sufficient for the safe operation of the turbine blades. All samples (Fig. 7) were tested on the same creep test machine. The test parameters are presented in Table 4. Results of research are shown in Table 5 and Figure 8.

Fig. 7. Appearance of the sample used for creep resistance testing.

Table. 4. Creep resistance test parameters

Cross-section of the sample	circular
Nominal sample diameter, d0 nom,mm	4
Nominal test temperature, °C	900
Nominal test stress, MPa	216

Table. 5. The course of the aging test and the results of creep resistance and hardness tests.

Sample number	**Hold time, h**	**Creep resistance, h**	**Hardness, HBW**
P10	18	the sample failed	302
P9	17	56.2	302
P8	16	56.1	317
P7	15	42.1	302
P6	14	45.9	206
P5	13	52.6	302
P4	12	46.2	302
P3	11	45.4	302
P2	10	45	302
P1	9	43.9	302

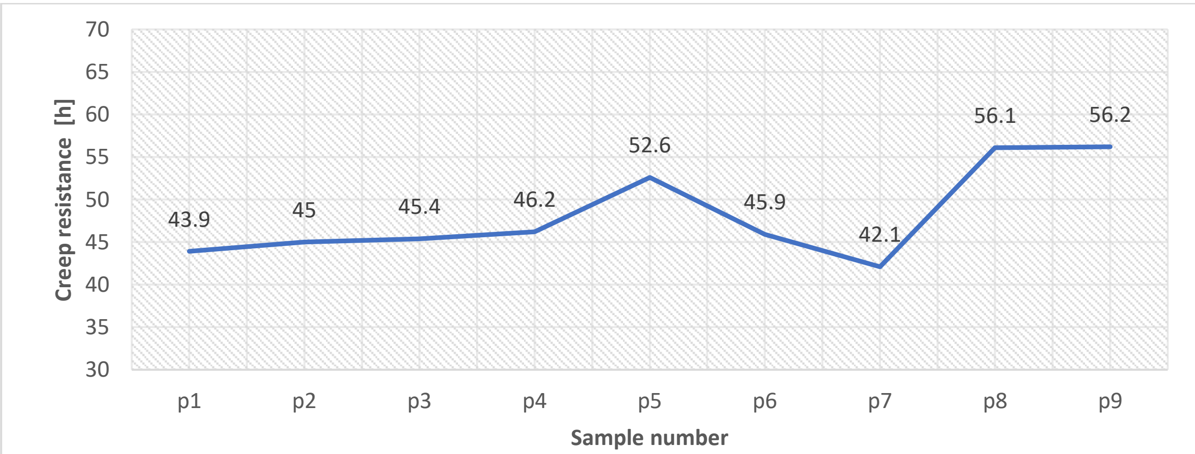


Fig. 8. Creep resistance results [h].

In addition, the hardness of the core of the samples was measured using the Manometer PBM No. 249 hardness tester according to PN-EN ISO 6506-1:2004-12 standard. The measurement was made on the HBW scale. Results are shown in figure 9.

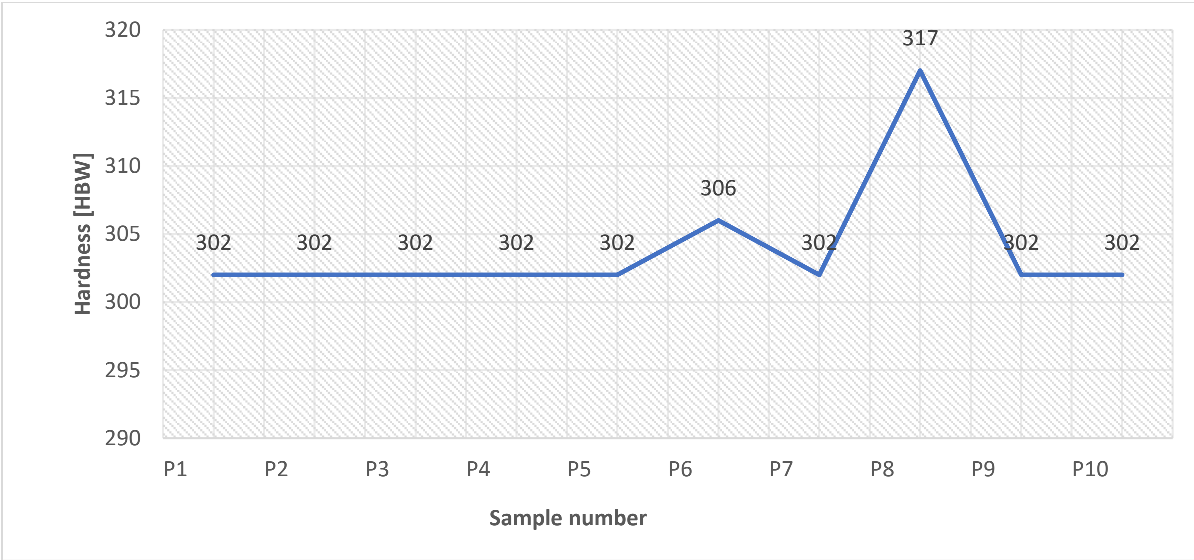


Fig. 9. Hardness results [HBW].

Additional tests on the samples subjected to the creep-strength test assessed the nature of the fracture and the place of initiation of the loss of cohesion by the material and the microstructure of the cross-section of the samples, and determining the grain size. Two samples broken during the creep resistance test were used for the tests (Figs. 10 and 11). The samples were labeled as P4 and P9. In both cases, the fracture has the characteristics of an intercrystalline fracture. No defects (inclusions, crimps, etc.) that could affect the initiation and development of cracks were observed on the samples. Grain distribution on the cross-section of both samples is shown in Fig. 12. In the case of sample P4, decohesion probably started along the grain boundaries from the left side, ending with an oblique fracture (tears are visible), while for sample P9, it can be seen that decohesion could have occurred evenly along the entire cross-section, ending with a fracture perpendicular to the axis of the sample.

The fracture surfaces of the broken samples were observed using SEM (Fig. 13). The SEM HITACHI SU3500 microscope equipped with the EDS/EDX micro-analyzer (Thermo Fisher model NS7; SN; 0914065) was used. Grain size checks were made by comparison using ASTM E112-13 Table 1 standards which cover grain sizes from 1 to 8.

Fig. 10. Sample cut from the P4 blade - the sample after the creep resistance test broke after 42.6 h.

Fig. 11. Sample cut from the P9 blade - the sample after the creep resistance test broke after 52.6 h.

a) b)

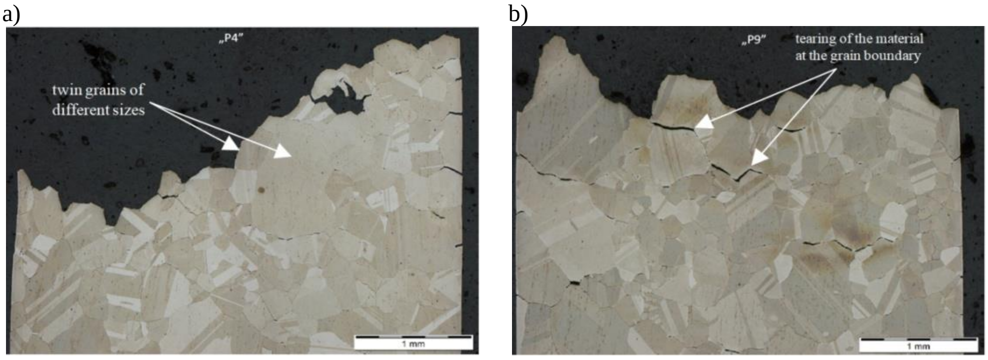


Fig. 12. Cross-sectional view of the zone adjacent to the fracture of the a) P4 sample b) P9 sample. In the structure, twin grains of different sizes and typical effects of the creep process (tearing of the material at the grain boundary) are visible. Etchant: Kalling's reagent.

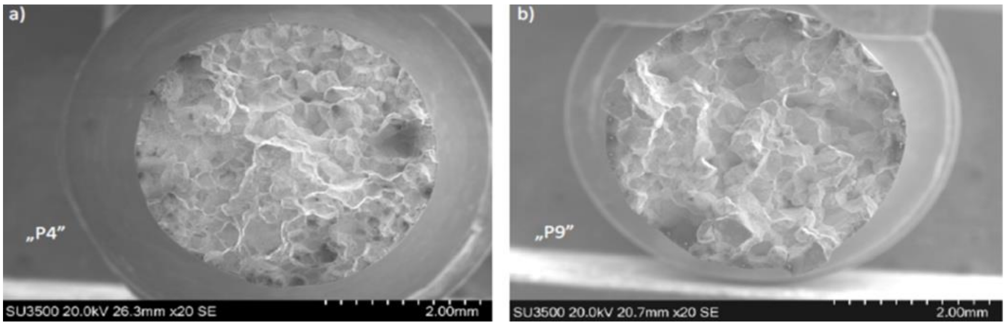


Fig. 13. SEM image (SE mode) of the fracture surface of two samples a) P4 blade, b) P9 blade.

3. Conclusions

Experimental research have shown that:

1) Aluminizing the Turbine Blades with the EI-867 material using the "above the pack" method to obtain a layer depth in the range of 0.03-0.06 mm is possible within 10 hours at 950ºC.
2) During the aluminizing of the EI-867 material, a layer with a three-phase structure is formed due to oppositely directed diffusion streams (from the surface and from the atmosphere). During the aluminizing of the EI-867 material, it has been observed that a layer with a three-phase structure forms. This is believed to be a result of oppositely directed diffusion streams, originating both from the surface of the material and from the surrounding atmosphere. This observation aligns with the results from multiple research teams, wherein similar material interactions exhibited the formation of multiphase structures under comparable conditions. The consistency in these findings across varied studies suggests that this behavior is not an anomaly, but rather a characteristic response of the EI-867 material to aluminizing processes.
3) Quantitative and qualitative analysis of the aluminized layer allowed to conclude that, in accordance with the adopted assumptions, an aluminide layer consisting of the expected NiA phase was formed on the blade surface from the EI 867 superalloy substrate, ensuring its good heat resistance properties. After a rigorous quantitative and qualitative analysis of the aluminized layer, we can deduce that the formation process aligns with our initial assumptions. An aluminide layer, predominantly consisting of the anticipated NiA phase, was identified on the blade surface derived from the EI 867 superalloy substrate. This specific phase composition is known in the research community to endow materials with enhanced heat resistance properties. Furthermore, comparable studies and examinations of similar superalloys have consistently shown the emergence of such layers when subjected to analogous aluminizing conditions, further strengthening our conclusion.
4) EI-867 forgings aged for 9, 10, 11, 12, 14, 15 h did not meet the limit value of creep resistance (assumed min. 50 h).
5) Forgings aged for 13, 16, 17 h met the requirement of the limit value of creep resistance (assumed min. 50 h).
6) The hardness of almost all forgings (except P8) remained at a constant level of 302-306 HBW.

7) During the metallographic tests, no defects of forging origin (inclusions, forging) were observed on the samples, which could affect the initiation and development of the crack.
8) The average grain size on the tested samples no. P4 and P9 was 1 on a scale from 1 to 8 according to ASTM E112-13 for nickel alloys. At the same time, in both tested blades, both much larger grains (several times) and clusters of smaller grains (grain no. 3 and a single smaller grain) are observed. The largest grain observed on blade P4 measured about 0.17 mm, while on blade P9 about 0.23 mm.
9) In both cases of the tested creep resistance samples, the fracture had the characteristics of intercrystalline fracture after rupture.

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Aluminiowanie dyfuzyjne metodą „above the pack” łopatek roboczych turbiny sprężarki z materiału EI867 stosowanych w lotnictwie

Streszczenie

W pracy przeprowadzono badania łopatek roboczych turbiny sprężarki wytwarzanych z materiału EI-867 wg normy TU 14-1-402-72 poddawanych procesowi gazowego bezkontaktowego procesu aluminiowania. Wykonano badania metalograficzne wytworzonej warstwy aluminiowanej oraz poddano identyfikacji składniki fazowe mikrostruktury warstwy aluminidkowej za pomocą analizy składu chemicznego w mikroobszarach z użyciem przystawki EDS w skaningowym mikroskopie elektronowym oraz metodą dyfrakcji rentgenowskiej. Ponadto wykonano badania twardości oraz żarowytrzymałości łopatek poddawanych procesowi przesycania i starzenia w różnych czasach wytrzymania. Praca miała na celu określenie możliwości prowadzenia procesu aluminiowania metodą „above the pack” części z materiału EI-867 przy jednoczesnym określeniu wpływu procesu na zmianę żarowytrzymałości części. Na podstawie badań eksperymentalnych wykazano, że aluminiowanie łopatek turbin materiałem EI-867 metodą „above the pack” do uzyskania grubości warstwy w zakresie 0.03-0.06 mm możliwe jest w ciągu 10 godzin w temperaturze 950˚C. Aluminiowanie analizowaną metodą powoduje powstanie warstwy aluminiowanej o strukturze trójfazowej zapewniającej odpowiednią wytrzymałość powłoki. Wykazano jednak, że proces aluminiowania metodą "above the pack" z zastosowanym czasem 10 godzin powoduje znaczne obniżenie żarowytrzymałości materiału. Na podstawie uzyskanych wyników wykazano iż metoda bezkontaktowego aluminiowania w przypadku łopatek turbiny z materiału EI 867 nie spełnia wymogów lotniczych dla bezpiecznej eksploatacji ze względu na znaczne obniżenie własności mechanicznych.

Słowa kluczowe: aluminiowanie dyfuzyjne, łopatki turbiny sprężarki, stop EI867, silnik turbinowy, turbina sprężarki