



The 20<sup>th</sup> International Conference: Machine Modeling and Simulations, MMS 2015

## Degradation of aluminide coatings deposited on nickel superalloys

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### Abstract

Cyclic oxidation and hot corrosion behaviours of aluminide and Si modified aluminide diffusion coatings deposited on two superalloys are compared in the paper. Samples based on cast nickel superalloys with and without protective coatings were studied under cyclic oxidation and hot corrosion conditions. The main goal of the paper was to compare the cyclic oxidation and hot corrosion resistance of protective aluminide coatings deposited on two types of nickel superalloys, Inconel 713LC and MAR-M247. All samples were exposed to cyclic oxidation and hot corrosion environment at Silesian University of Technology in Katowice, Poland. Hitachi SEM with EDS microanalysis was used for analysis of surfaces and cross-sections of the samples. Experimental cyclic oxidation test was carried out in inductive furnace at 1100 °C and samples were exposed to 24 cycles. Heating time took 23 h. After each oxidation cycle, the samples were taken out of furnace and cooled in the air. Mass changes of the samples were written down during each second cycle to observe the progress of cyclic oxidation. The resulting graph of the sample masses revealed that resistance to cyclic oxidation of IN713 LC samples without protective coating exposed to 24 cycles was very good and without any noticeable damage. The oxidation of the superalloy surface of MAR-M247 without the protective coating was very fast. Hot corrosion is the accelerated oxidation of a material at elevated temperature induced by a thin film of fused salt deposit. Superalloys samples were coated by help of the method out-of-pack. Corrosive environment was created using tablets of Na<sub>2</sub>SO<sub>4</sub> at temperature 920 °C. Results of tested samples revealed that the samples with Si modified aluminide coating had the highest life-time.

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Peer-review under responsibility of the organizing committee of MMS 2015

*Keywords:* hot corrosion; cyclic oxidation; superalloys; diffusion coatings

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## 1. Introduction

The nickel-based superalloys are mainly used in aircraft industry as turbine components which are exposed to oxidation environments during service. Aluminide coatings in aircraft industry are mainly designed to protect nickel-based superalloys, which operate in difficult environments, such as high temperatures, hot corrosion or cyclic oxidation. Protective coatings were developed to serve as physical barriers between aggressive environment and the substrate. The most widely used coatings are those based on the intermetallic compound of NiAl which is formed by the diffusion interaction of Al, Cr, Si or their combination with surfaces of Ni-superalloys [1, 2]. Aluminum obviously has the greatest effect in relation to oxidation resistance which generally increases with Al concentration. Cr additions to  $\beta$ -NiAl compounds and coatings have been investigated to improve their resistance to hot corrosion. At high operation temperatures, the aluminide elements create thin and adhesive alumina scale of its oxides to provide oxidation resistance. Up to now, however, no coating that would fully survive the aggressive turbine environment has been found [3]. The most serious degradation modes are as follows [4]:

- high-temperature oxidation,
- hot corrosion,
- damage by thermal and thermomechanical fatigue,
- mechanical damage by erosion,
- creep degradation during overheating.

Inter-diffusion of elements at the interface with the substrate resulting in a creation of undesirable phases is sometimes also mentioned as an independent degradation mode.

Hot corrosion is the accelerated oxidation of a material at elevated temperature induced by a thin film of fused salt deposit. Fused  $\text{Na}_2\text{SO}_4$  is an ionic conductor, so that the corrosion mechanism is electrochemical in nature [5]. Hot corrosion attack is minimized by the selection of an alloy or coating which is able to form a protective oxide which is resistant to the presence of the salt film [6]. There are generally two stages of hot corrosion. The first stage is known as the initiation stage and involves the breakdown of the protective oxide scale. The second stage is called the propagation stage, in which the salts have access to the unprotected metal and corrosion continues unabated at exceedingly high rates [2]. Hot corrosion is usually subdivided into two types: Type I (HTHC) and Type II (LTHC). Various parameters may affect the development of these two forms, including alloy composition and thermo-mechanical condition, contaminant composition and flux rate, temperature and temperature cycles, gas composition and velocity and erosion processes [7, 8]. A protective scale can be maintained when the aluminium, chromium or silicon do not fall below their critical levels, e.g., 4–5 of weight % for Al. A rapid spallation of TGO can be also caused by internal oxidation, i.e., the diffusion of oxygen into the coating. Disruption and spallation of the protective scale are exacerbated by a presence of impurities (mainly sulphur) on the metal/TGO interface. A general indicator of the protectiveness of the oxide scale is given by the Pilling-Bedworth Ratio (PBR) which is defined by the volume ratio oxide formed/metal consumed. The scale becomes fully protective when  $\text{PBR} \sim 1$ .  $\text{PBR} \ll 1$  means that the oxide is porous and consequently, loses any protective properties.  $\text{PBR} > 1$  is also connected with the fact that the oxide scale is highly compressed thus resulting in buckling and spallation [2].

## 2. Philosophy of experiments

In the experimental work, cyclic oxidation and hot corrosion resistance of nickel superalloys with and without coatings were studied. Two types of Ni-superalloys, such as Inconel 713LC and MAR M-247 (Table 1) were used for investigation. These superalloys were deposited by aluminide coating and Si modified aluminide coating using CVD out-of-pack process.

Table 1. Chemical composition of tested superalloy in wt. %.

	Cr	Al	Mo	W	Nb	Ti	Ta	Zr	Hf	C	B	Co	Ni
IN 713 LC	11.85	5.8	4.54		2.27	0.72		0.11		0.04	0.015		bal.
MAR M-247	8.4	5.5	0.7	10.0		1.0	3.0	0.05	1.5	0.15	0.015	10.0	bal.

Experimental samples casted from IN 713LC and MAR-M247 for cyclic oxidation test had cylindrical shape with dimensions of  $14 \times 5$  mm. Their microstructure consisted of the  $\gamma$  matrix strengthened by  $\gamma'$  phase with the shape of cuboidal particles of  $\text{Ni}_3(\text{Al,Ti})$  as coherent precipitates and complex carbides [4]. Three types of samples for two kinds of superalloys were used for hot corrosion and cyclic oxidation tests:

- samples of IN 713 LC and MAR-M247 without coating,
- samples of IN 713 LC and MAR-M247 with aluminide coating and,
- samples of IN 713 LC and MAR-M247 with Si modified aluminide coating.

Aluminide coatings were applied by the “out-of-pack” method and Si modified aluminide coatings were made by the method of “pack-cementation”.

### 2.1. Philosophy of cyclic oxidation test

Disc samples in ceramic bowl were placed into induction furnace with the temperature  $1100^\circ\text{C}$ . After 23 h, the tested samples were taken from furnace and they were immediately exposed to the cooling process in the air at room temperature for 1 h and then the mass changes were measured. This description represented one oxidation cycle. Mass changes of samples were written down after the each second cycle and the photos were done.

### 2.2. Philosophy of hot corrosion test

Three types of samples with tablets of  $\text{Na}_2\text{SO}_4$  were placed into the furnace and heated to  $900^\circ\text{C}$ . These pieces were taken from the furnace in graduated time intervals which provided possibilities for microstructural analyses and assessment of hot corrosion behaviour in progress of test. After that, the samples were cut and electron microanalyses were performed for cross-sections. In this paper, the samples, before and after 64 hours of exposition in the furnace, were compared. Fig. 1 represents the sample with tablet of  $\text{Na}_2\text{SO}_4$ .



Fig. 1. Sample with tablet of  $\text{Na}_2\text{SO}_4$  before testing.

## 3. Results

### 3.1. Results from cyclic oxidation test

Gravimetric mass changes of tested superalloys with coatings have been made from the aspect of the kinetics cyclic oxidation description and pursuing of mass changes during oxide formation. The results of this investigation could be used for comparison of oxide resistance of tested superalloy/coating systems as well as for selection of the most suitable combination for applying in the practice. The resulting graph of all tested samples is shown in Fig. 2.

From this picture, it is clear that IN 713 LC with unmodified aluminide coating had lower life-time than without coating and it is very surprising finding. Mutual comparison of all gravimetric curves revealed that MAR-M247 without coating could not be used for practice application in the oxidation environment. Samples from MAR-M247 superalloy with aluminide Al coating appears to be the most acceptable selection of combination relating to superalloys/coating.

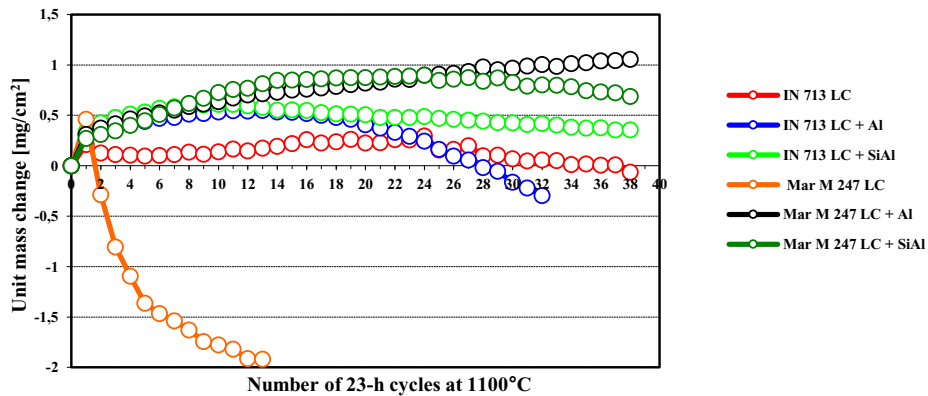


Fig. 2. Weight changes after each oxidation cycle for all tested samples.

### 3.2. Results from hot corrosion test

Cross-sections of samples were investigated by the help of scanning electron microscopy (SEM) and chemical microanalyses (EDS). View on the samples with aluminide and Si modified aluminide coating after testing and their microstructures are compared on Figs. 3–7.

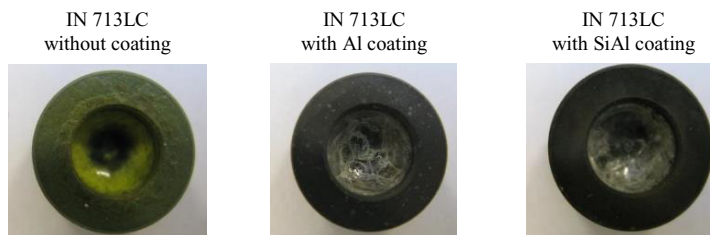


Fig. 3. Samples from IN 713 LC after 64 hours of testing.



Fig. 4. Samples from MAR-M247 after 64 hours of testing.

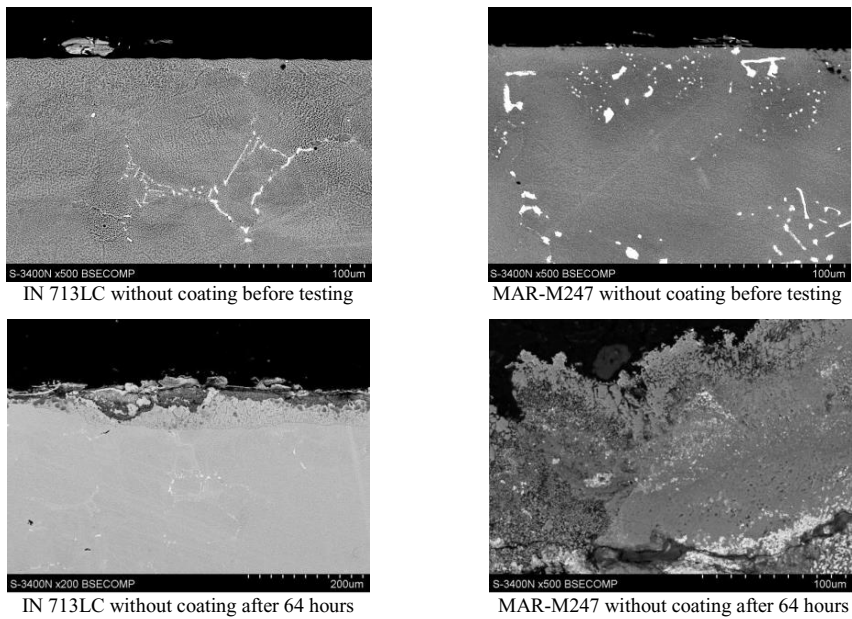


Fig. 5. Microstructures of samples without coating before and after 64 hours of testing.

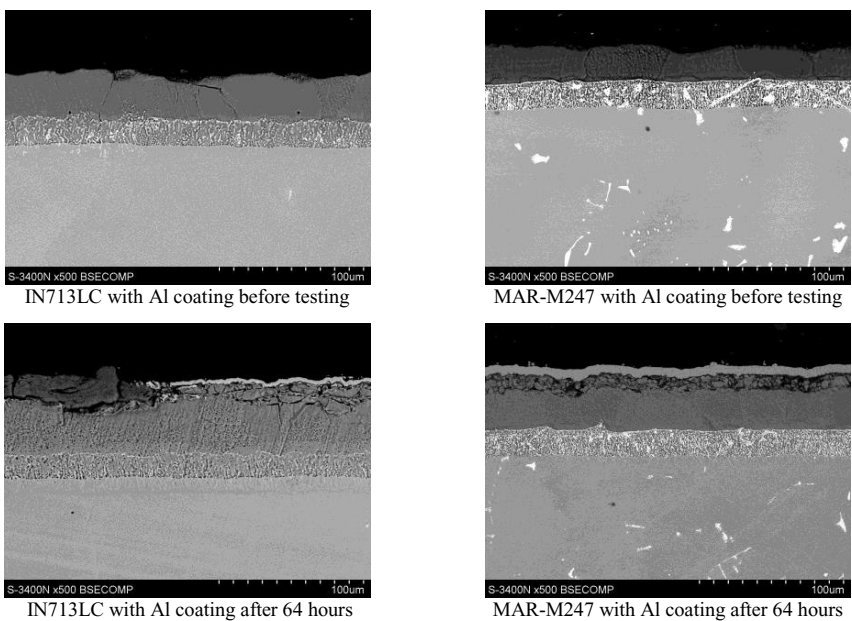


Fig. 6. Microstructures of samples with Al coating before and after 64 hours of testing.

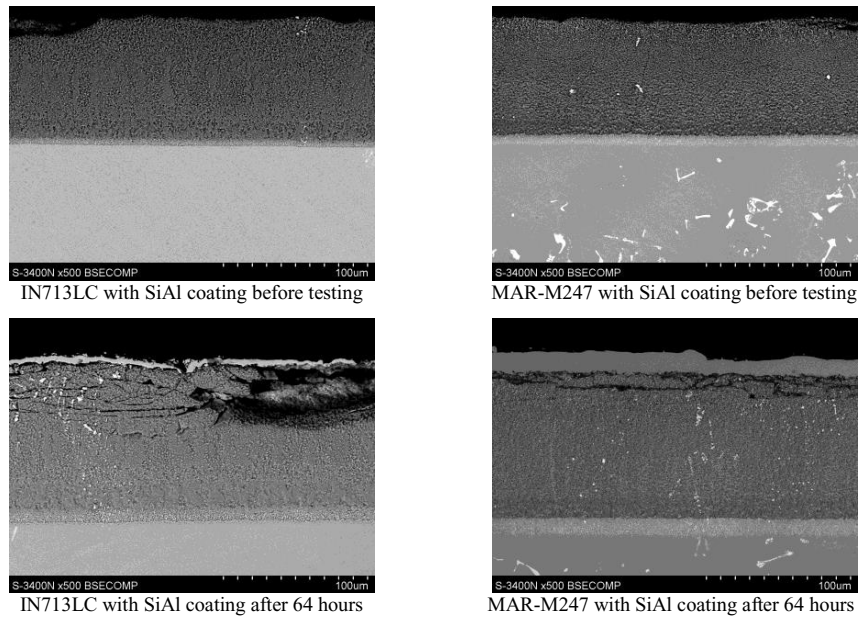


Fig. 7. Microstructures of samples before test and after 64 hours of testing.

#### 4. Conclusion

The main goal of the paper was to compare the cyclic oxidation and hot corrosion resistance of protective aluminide coatings deposited on two types of nickel superalloys, namely, Inconel 713LC and MAR-M247.

In the case of cyclic oxidation resistance, these conclusions are clear referring to Fig. 2:

1. System MAR-M247 with aluminide unmodified coating have the better resistance to cyclic oxidation because its gravimetric maximum was even not reached after 38 cycles, while MAR-M247 without coating showed the lowest resistance to cyclic oxidation.
2. IN 713LC without coating exhibited high resistance and its weight fell under original weight as far as 37 cycles which was very surprising finding.
3. Si modified coating on IN 713LC has been found as the most suitable system for this type of degradation.
4. Microstructure of IN 713LC without protective coatings did not satisfy requests of creep resistance in the environment of high temperatures and from this reason they are not appropriate for this type of degradation mode.

In the case of hot corrosion resistance we can summarize:

1. The largest attack by corrosion salts was observed in the case of MAR M247 without coating.
2. Superalloys with aluminide coatings had greater resistance to hot corrosion than without coatings, aluminide diffusion coating on the surface of superalloys made an effective barrier inhibiting input of corrosion salts.
3. Among all tested samples, Si modified coating revealed the smallest corrosion attack after 80 hours of testing.
4. Tested superalloys without coatings achieved the second advanced stage (propagation stage), while samples with coatings achieved the first stage – initiation stage of hot corrosion.

Using of diffusion coatings depends on coating – superalloy system and environment conditions. Considering only these two degradation modes (cyclic oxidation and hot corrosion), the best results were obtained for Si modified coatings on MAR M247.



## Acknowledgement

The authors acknowledge the project VEGA 1/0385/14 of Ministry of Education, Science, Research and Sport of the Slovak Republic.

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