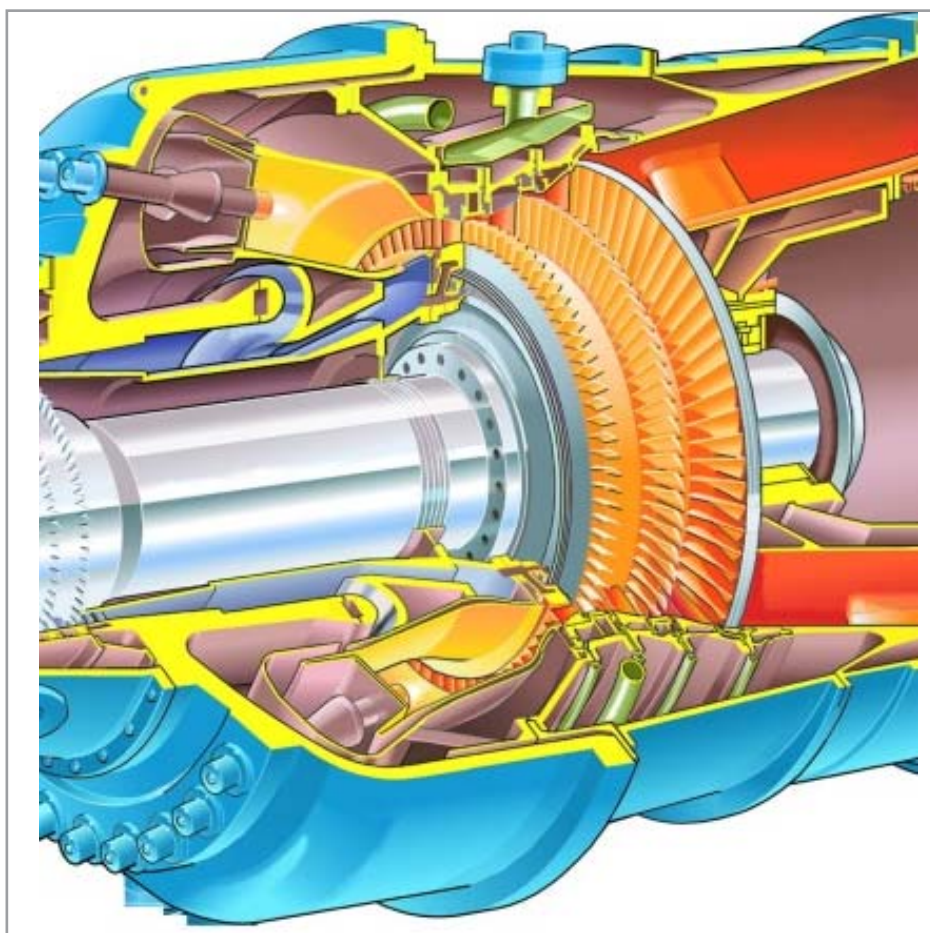


DURABLE MCrAlX COATINGS IN GASTURBINES

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Durable MCrAlX coatings

For demanding applications in gasturbines

KME 703

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Foreword

The project has been performed within the framework of the materials technology research programme KME, Consortium materials technology for thermal energy processes, period 2014-2018. The consortium is at the forefront of developing material technology to create maximum efficiency for energy conversion of renewable fuels and waste. KME has its sights firmly set on continuing to raise the efficiency of long-term sustainable energy as well as ensuring international industrial competitiveness.

KME was established 1997 and is a multi-cliental group of companies over the entire value chain, including stakeholders from the material producers, manufacturers of systems and components for energy conversion and energy industry (utilities), that are interested in materials technology research. In the current programme stage, eight industrial companies and 14 energy companies participate in the consortium. The consortium is managed by Energiforsk.

The programme shall contribute to increasing knowledge within materials technology and process technology development to forward the development of thermal energy processes for efficient utilisation of renewable fuels and waste in power and heat production. The KME goals are to bring about cost-effective materials solutions for increased availability and power production, improved fuel flexibility and improved operating flexibility, with low environmental impact.

KME's activities are characterised by long term industry and demand driven research and constitutes an important part of the effort to promote the development of new energy technology with the aim to create value and an economic, environmentally friendly and long term sustainable energy society.

Prof Ru Lin Peng, LiU, has been the project leader. The working group consisted of Dr Xin-Hai Li from SIT and Dr Kang Yuan (till 2015-02), PhD student Pimin Zhang (from 2015-05), Prof Ru Lin Peng and Prof Sten Johansson from LiU. Several other colleagues in SIT were also involved in tests and experiments. The reference group for the project consisted of Rikard Norling, Swerea KIMAB and Bo Jönsson from Kanthal/Sandvik Heating Technology AB. Siemens Industrial Turbomachinery AB has participated in the project through own investment (60 %) and the Swedish Energy Agency has financed the academic partners (40 %).

The report is a shorter version of the complete KME report.

Energiforsk would like to thank all the participants for a well performed project.

Bertil Wahlund, Energiforsk

These are the results and conclusions of a project, which is part of a research program run by Energiforsk. The author/authors are responsible for the content.

Sammanfattning

MCrAlX-ytbeläggningar (M = Ni, Co; X = mindre element) används i stor utsträckning för att skydda heta komponenter gjorda av superlegeringar, som används i gasturbinens högtemperatursektioner. Med den ökande andelen förnybar energi, har även behovet av industriella gasturbiner med bättre cyklisk tolerans och prestanda för att hålla en stabil elförsörjning, ökat. Huvudsyftet med detta projekt, som är en fortsättning av KME 503, är att utveckla nya MCrAlX-ytbeläggningar med egenskaper som är optimerade för tillämpning i cykliskt toleranta gasturbiner. Projektet genomfördes genom samarbete mellan avdelningen Engineering Materials vid Linköpings universitet och Siemens Industrial Turbomachinery AB i Finspång.

Tre ytbeläggningar (två Ni-baserade och en Co-baserade) från projektet KME 503 har optimerats vidare i projektet. Analys av resultaten i KME 503 samt termodynamisk simulering resulterade i en provmatris bestående av sju nya Ni-baserade, fyra nya Co-baserade och två Ta-innehållande kompositioner. Motståndet mot termisk cykling (TCF) och oxidation och korrosion vid höga temperaturer samt mekaniska egenskaper karakteriserades för de Ni-baserade och Co-baserade ytbeläggningarna. Effekterna av valda ytbeläggningar på termomekanisk utmattningsresistens hos superlegeringar studerades. De viktigaste resultaten från projektet är:

- Två Ni-baserade ytbeläggningar med förbättrad resistens mot hög temperaturoxidation och TCF
- Ytbeläggningsdata innefattande kort- och långtidsoxidation, termisk cykling, het korrosion, mekaniska egenskaper och etc. för ett antal Ni-baserade och Co-baserade ytbeläggningar
- Ett kriterium baserat på beräknade Al-aktivitet för mer exakt förutsägelse av oxidationslivslängd på MCrAlX ytbeläggningar
- Mer än tio vetenskapliga artiklar om korrosion, oxidation, effekter av X-element, effekter av ytbehandling, modellering och design av ytbeläggningar
- En licentiatavhandling.

Alla projektmål är uppfyllda förutom det komponenttest som återstår att slutföra.

Behandling för att skräddarsy och optimera beläggningens ytskikt samt effekterna av legeringsämnen på "rumpling" (förgrovnig av ytan) under termisk cykling kommer att studeras i fortsättning med avseende på motstånd mot oxidation och TCF. Dessa arbeten som kan bidra till förbättrad funktionalitet av MCrAlX-beläggningar som bindsikt, har stor betydelse för utveckling av cykliskt toleranta TBC-system. En annan intressant forskningsfråga att driva är Al-aktivitetens roll. Ökad förståelse för sambandet mellan Al-aktivitet och MCrAlX-ytbeläggningens oxidation och korrosionsbeteende kan leda till utveckling av bättre ytbeläggningar och livsmodeller.

Summary

MCrAlX (M=Ni, Co; X=minor elements) coatings are widely used for the protection of hot components made of superalloys, operating in the high temperature sections of gas turbines. With the increasing use of renewable energy sources, there has also been demand on industrial gas turbines with better cyclic tolerance and performance for use for example in sustainable energy systems to maintain a stable power supply. This project is a continuation of KME 503 project aiming at developing new MCrAlX coatings with properties optimized for applications in cyclically tolerant gas turbines. The project was carried out through collaboration between the Division of Engineering Materials of Linköping University and Siemens Industrial Turbomachinery AB in Finspång.

Three KME 503 coatings, two Ni-based and one Co-based, were further optimized. Refinement based on analysis of the results in KME 503 and with the help of thermodynamic simulation resulted in a sample matrix consisting of seven new Ni-based coating compositions, four new Co-coating compositions. Two Ta-containing coatings were also included for test. Resistance against oxidation and hot corrosion as well as mechanical properties were characterized for the Ni and Co-based coatings. The effect of selected coatings on the thermomechanical fatigue life of superalloys were studied. Main achievements from the project include:

- Two Ni-based coatings with improved oxidation and thermal cycling resistance after the optimization;
- Coating data for short and long term oxidation, TCF, hot corrosion, mechanical properties, and etc. for the Ni and Co-based coatings;
- An Al-activity base criterion for more accurate prediction of oxidation life of MCrAlX coatings;
- More than ten journal/conference papers on hot corrosion, oxidation, X-element effect, surface treatment effect, modelling and coating design;
- A licentiate thesis.

All project goals are essentially fulfilled except for the component test that remains to be completed.

Further research work on MCrAlX coatings has been planned. Among others, surface treatments to tailor and optimize a MCrAlX coating's surface condition or increased the coating's resistance against rumpling (surface roughening) during thermal cycling by alloying will be studied with regard to improved oxidation and thermal cycling resistance. Such improvement can also enhance the functionality of MCrAlX coatings as bondcoat in strain tolerant TBCs. Another interesting question to pursue is the role of Al-activity. A better understanding of the correlation between the Al-activity and MCrAlX coatings' oxidation and corrosion behavior can lead to development of better coatings and coating life models.

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1 Introduction

1.1 BACKGROUND

The efficiency and durability of modern gas turbine engines are dependent on the use of high temperature coatings, metallic overlay coatings and thermal barrier coatings (TBC) to protect superalloy components and allow them to operate at a high engine temperature. While metallic coatings provides oxidation and corrosion resistance, TBCs typically containing a ceramic top coat and a metallic coating as bondcoat also reduce superalloys' surface temperatures.

There has been an increased demand of industrial gas turbines with increased operational flexibility and performance for use for example in sustainable energy systems to maintain a stable power supply. This project (KME 703/713) is the continuation of KME 503/520 (Phase I) which aims at developing new durable MCrAlX (M=Ni, Co; X=minor elements) coatings optimized for applications in industrial gas turbines of medium size with requirement on operational flexibility. In KME 703/713 (Phase II), research work is continued on MCrAlX coatings with regard to further improving their properties and understanding correlation between coating performance and composition. Coating compositions developed in Phase I were further refined and evaluation of the coatings was done through various characterization tests and analyses. Study of the thermomechanical fatigue (TMF) behavior important for gas turbines operating in power grids utilizing wind power and solar energy was carried out. Continued work on the oxidation-diffusion model includes its adaption for coating design, chemical matching of coating and substrate for improved oxidation resistance, and establishment of an Al-activity criterion for accurate oxidation life prediction.

1.2 RESEARCH FIELD AND RESEARCH QUESTIONS

MCrAlX (also traditionally called MCrAlY) coatings are widely used as overlay coatings and as bondcoat in TBC. The main advantages of MCrAlX coatings over other high temperature metallic coatings such as PtAl diffusion coatings include better strain tolerance, flexibility for varying coating thickness or chemical composition to suit different applications, relatively good corrosion resistance and less dependence on base material's chemistry. In spite of their wide application, continued interests are directed toward research and development to improve existing MCrAlX coatings or to develop new MCrAlX coatings with better performance, as reflected in some recent review articles on high temperature coatings[1][2].

The oxidation and corrosion resistance of MCrAlX coatings relies on a continuous protective scale on the coating surface. MCrAlX coatings are designed to contain a certain amount of Cr and Al to form and maintain a scale of essential α -alumina[2]. Ni and Co are either added alone or together to form the desired microstructure typically consisting of intermetallic β -NiAl and γ -Ni solid solution. While Co-base MCrAlX coatings with a high Cr-content are often used in environments where hot corrosion resistance is important, Ni-base MCrAlY coatings with or without Co addition show better performance at higher service temperature for which

oxidation is the dominant degradation process. Reactive elements (REs) such as Y, Hf and Zr are important for the protective scale's formation, growth and adherence [3]. Literature and patent surveys carried out in KME 503 revealed that Hf, Si, Ta, Re, Ru, Zr, etc. are often considered in the development of new MCrAlY coatings to improve certain properties.

During gas turbine operation, coating degradation inevitably occurs and develops under the influence of environment, temperature and possible mechanical loading. A common mode of MCrAlY coating degradation is spallation of the protective oxide scale. Reducing the scale growth rate or increasing the scale-coating interfacial strength can effectively increase resistance to scale delamination. REs are conventionally added to reduce scale growth but later their effect of improving the scale adherence has been recognized [3]. The increased scale adherence is attributed to RE-oxide pegs formed in the scale and the removal of impurity species especially S at the scale/alloy interface [4][5]. As the presence of such non-alumina oxides in the protective scale can promote oxygen diffusion[6], overdoping by REs can accelerate scale growth. The total amount of Y in the coating is important for optimal scale adhesion and better adhesion can be achieved by co-doping with other REs [3][6]. In phase I of our project, co-doping of some REs was studied and the oxidation tests reveal no improvement in oxidation resistance. **In Phase II, the addition of Hf together with Y and Si is continued for its effect on thermal cycling resistance. In addition, the replacement of Y by Ce is investigated.**

Surface modification of deposited coating can provide a promising route for increasing MCrAlY coating's oxidation resistance. Their positive effects as reported in the literature can be attributed to improved surface morphology [7][8]. Novel surface treatment methods can also improve the oxidation resistance. For instance, sputtering a layer of Al_2O_3 on MCrAlY coating surface can reduce oxidation rate and also promote the adhesion between MCrAlY and top coat [8]. **In Phase II we also look at the effect of certain surface treatments on long term oxidation behavior and analyze the mechanisms behind.**

Another important degradation mode is the depletion of Al in the coating due to inward diffusion to the substrate of low Al-content during exposure to the service temperature. The importance of interdiffusion between the coating and substrate has long been recognized [9] and early experiments showed that Al-depletion rate varies between different coating/substrate systems. The work by Dahl et al. [10] showed that diffusion activity under a long term oxidation can be effectively studied by thermodynamic and kinetic simulations. Development and use of new superalloys also requires new MCrAlX coatings with matching composition and thermodynamic and kinetic simulation can facilitate coating design [1]. Interdiffusion can be reduced by alloying, introducing a diffusion barrier or be chemical matching of coating and substrate. In Phase I, alloying to reduce interdiffusion was studied for the effect of Ru, Mo and Ir [11] and an oxidation-diffusion model based on ThermoCalc software [12] [13][14] [15] aimed for eventual use for coating oxidation life prediction was established. **In phase II, the alloying effect including that of including Ru and Fe is further investigated. The condition for in situ formation of a continuous β -layer is exploited for the purpose of designing coatings with reduced inward diffusion of Al. Further,**

research in coating design/selection to match substrate for reduced interdiffusion is carried out. For the above mentioned work, the established oxidation-diffusion model from Phase I is an important research tool, which is further developed and used for understanding the effect of interactions between coating and substrate, for determining factors that control interdiffusion, and for designing coatings or for chemical matching between coating and substrate to gain better oxidation resistance.

For predicting oxidation life, a criterion marking the end of coating life, that is when the coating can no longer form a continuous scale of α -alumina, is needed. As Al is predominantly important, a critical Al-content is often used, although it is known that other alloying elements, especially the existence and level of Cr, can also affect the coating's ability of forming the protective scale. Another criterion similar to the critical Al-concentration, which we also used in Phase I, is the total depletion of β -phase which is considered to be coating's Al-reservoir. **In Phase II, in association with further development of the oxidation-diffusion model, the suitability of the Al-concentration criterion is investigated and efforts are directed towards establishing a criterion that can be used for more accurate prediction of coating's oxidation life.**

While the coating's ability to form and maintain a protective oxide scale is crucial for oxidation and corrosion resistance, the coating's microstructure and its stability during gas turbine operation are important for retaining the desired mechanical properties to resist coating cracking and spallation in association with thermomechanical cycling. However, damage development in coated system due to thermomechanical fatigue (TMF) can be complicated, affected by incompatible strains due to thermal and/or mechanical mismatch between the coating and the substrate, ductile-to-brittle-transition temperature (DBTT) of the coating, and relationship between the thermal and mechanical loading. For flexible gas turbine operations, the coated component resistance to TMF loading becomes even more important. Publications from experimental study of coated superalloys are rather limited, see however [16][17][18]. **In Phase II, the TMF behavior of selected coated systems and their DBTT are characterized and post analysis to gain insight into the damage process is performed.**

1.3 GOAL

Main project goals

- To develop new durable MCrAlX coatings with improved performance for applications in medium size industrial gas turbines operating on a flexible base in terms of fuel and operation mode,
- To contribute to increased understanding of correlations between chemical composition, coating process, microstructure and performance of MCrAlX coatings,
- To study the influence of applications of MCrAlX coatings on the behavior of superalloys.

Deliverables

- D1. Qualification of the Ni-base and Co-base MCrAlX coatings developed in Phase I which is required for implementation of the coatings on gas turbine components
- D2. Adaptability of two selected coatings from Phase I for applications on new base materials and other deposition methods
- D3. Three new coatings, one optimized for mechanical properties, one with a diffusion barrier, and one coating for cost reduction, and material data for oxidation and corrosion behavior
- D4. Improved oxidation-diffusion model which can be used for coating design and life modeling
- D5. At least 5 scientific publications in journals and international conferences
- D6. A licentiate and base for a PhD thesis
- D7. Bondcoat function of selected coatings
- D8. Report of component test

1.4 PROJECT PERIOD AND FUNDING

Siemens Industrial Turbomachinery AB in Finspång (SIT) and the Division of Engineering Materials of Linköping University (LiU) are the partners.

The project officially started in September 2014 and ended by April 2018.

The project has been conducted within KME and funded by the Swedish Energy Agency with in-kind contributions from Siemens Industrial Turbomachinery AB in Finspång.

2 Project Realization

2.1 PROJECT PARTICIPANTS

The working group consists of Dr Xin-Hai Li from SIT and Dr Kang Yuan (till 2015-02), PhD student Pimin Zhang (from 2015-05), Prof Ru Lin Peng and Prof Sten Johansson from LiU. Several other colleagues in SIT were also involved in tests and experiments.

The reference group for the project consists of Dr Rikard Norling from KIMAB and Bosse Jönsson from Sandvik Heating Technology AB.

SIT is responsible for providing all test samples, performing heat treatments, oxidation tests, TCF tests, part of the corrosion tests, DBTT tests, part of TMF tests, and etc.

LiU is responsible for most of the post-test analyses, some corrosion and TMF tests, modelling and model development, documentation and writing of publications, and etc.

Cooperation with University West allows a study of Fe-containing MCrAlX coatings as bondcoat for an emerging SPS TBC of strain tolerance.

2.2 PROJECT TASKS

Six project tasks were planned for KME 703:

- T1. *Fine-tuning and qualifying the selected surface coatings from Phase I*
Further refinement of coating composition with the help of simulation (for microstructure checking) and characterization by oxidation, corrosion and ductility tests on the selected coatings
- T2. *Adaptability of the selected coatings from Phase I*
Simulation and experiment to confirm the adaptability of the selected coatings through deposition by another method and on another substrate
- T3. *Thermomechanical Fatigue (TMF) test of superalloys with MCrAlX coating*
Thermal mechanical fatigue test on selected coating composition and substrate as well as post-test analysis to understand the failure mechanisms and coating influence on substrate
- T4. *New coatings developed in Phase I based on found improvement properties*
Refinement of coating composition by simulation, manufacturing of test samples and characterization by oxidation, corrosion and other tests.
- T5. *Microstructural characterization and analysis*
Post-test analysis of oxidized and corroded samples by SEM, XRD and possibly other method for understanding the different failure processes
- T6. *Modeling*
Further development of the oxidation-diffusion model for design and oxidation life prediction and validation

Two tasks were planned for KME 713:

- T7. *Bondcoat function of selected MCrAlX coatings*
Evaluation of the behaviour of selected coatings as bondcoat in thermal barrier systems by deposition samples and TCF test

T8. *Component test of selected MCrAlX coating and evaluation*

Selection and preparation of component and perform TCF tests on deposited components

2.3 METHODS

Coating deposition methods: HVOF (dominant), HVAF, VPS and LPPS

Characterization of coating properties includes:

- The oxidation behavior of the coatings was characterized by isothermal tests at 900, 1000 and 1100 °C, respectively;
- Resistance to thermal cycling by TCF test between 100 and 1100 °C with 1 hour dwell time at 1100 °C;
- Hot corrosion tests in three different environments;
- Mechanical tests at different temperatures to determine the ductile-to-brittle-transition-temperature (DBTT);
- Thermal mechanical fatigue tests to study the effect of the coatings on substrate materials.

Post-test analyses for increased understanding of coatings behaviour include:

- Scanning electron microscopy (SE/ECCI/EDS/WDS)
- X-Ray diffraction
- photostimulated luminescence
- Microhardness testing

Thermodynamic and kinetic modelling for coating composition design, composition refinement and coating degradation under thermal exposure

3 Results and Analysis

3.1 COATINGS FROM FURTHER REFINEMENT

Refinements of coating compositions from Phase 1 based on the obtained results and with help of microstructural simulation have resulted in 4 groups of coatings. The nominal compositions of the four coatings investigated in KME 703 are given in Table 1. Results from oxidation experiments are summarized in 3.2 and corrosion in 3.4. DBTT and TMF tests are summarized in 3.5. Test samples for the fourth group, Ta-containing have been manufactured and oxidation and TCF tests have started.

Table 1 Nominal composition of refined coatings and reference from Phase 1 (Al-content in parentheses is from measurement)

	Coating ID	Ni	Co	Cr	Al	Y	Si	Other X-elements
Ni-Base	D10, ref	43	28	15	11	0.4	0.25	Hf, Ru, Ta, Mo, and Ce
	D11-D12	43	28	15	11 (12.5-12.8)	0.4	0.25	
Co-Base	B10, ref	30	Bal	20	10	0.6	0.25	Hf, Ru, Ta, Fe
	B11-B14	30	Bal	20	11 (8-9.2)	0.6	0.25	
Fe-bearing	D70, ref	Bal	28	15	11	0.5	1	Hf, Ta, Mo, Fe
	D71-D75	Bal	28	15	11 (11-12.6)	0.5	1	
Ta-bearing	C1-C2	Bal	15	12.5	10	0.4	0.5	Ta, Fe

Table 2 Chemical composition of substrate alloys used in the project

Ni	Cr	Co	W	Ta	Ti	Al	Mo	Nb	C	Others
Bal.	12.5	9	4.2	4.2	4	3.4	1.9	-	0.08	B, Zr,...

Coatings used for oxidation and corrosion tests are deposited on IN-792 with a nominal thickness of 200 μm by the high-velocity oxy-fuel (HVOF) technique. The chemical composition of IN-792 can be found in Table 2. After the coating deposition, all the samples were heat treated to simulate the standard heat treatment typically for IN-792 components for strengthening. The coating microstructure consists of a Ni-base solid solution (γ) and an intermetallic NiAl (β). It should be noted that the measured chemical composition in as-deposited coatings deviates somewhat from those given in Table 1.

3.2 OXIDATION BEHAVIOUR

Characterization of oxidation resistance

The oxidation behavior of the different coatings were characterized by isothermal oxidation test at three different temperatures, 900, 1000, and 1100 $^{\circ}\text{C}$, and cyclic oxidation between 100 – 1100 $^{\circ}\text{C}$ with the longest oxidation time reaching 11 000

hours. The performance of the coatings was characterized first by evaluating microstructural changes due to surface oxidation as well as interdiffusion between coating and substrate. Both the processes result in loss of Al from the coating, leading to transformation of the aluminum rich β to aluminum poor γ and thereby the formation of an outer and an inner β depleted zone, respectively (Fig. 1, left). The oxidation resistance was then further analyzed by following the development of oxide scale growth. Example of the β -zone and TGO evolution are shown in Fig. 1 (right) and Fig. 2, respectively for the Ni-base coatings.

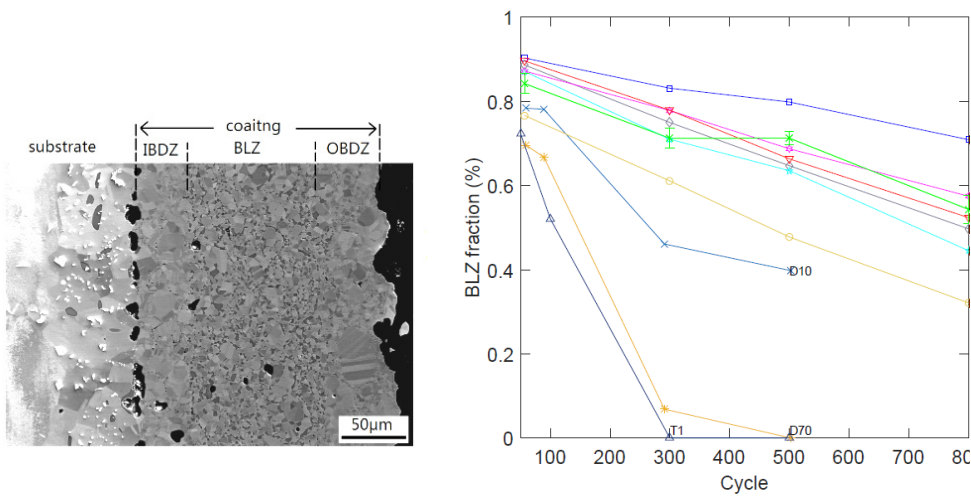


Figure 1 Definition of the different microstructural layer (left) and comparison of resistance to β -depletion between the Ni-base coatings under TCF (D1x and D7x). D10 and D70 are internal references while T1 is an external reference coating.

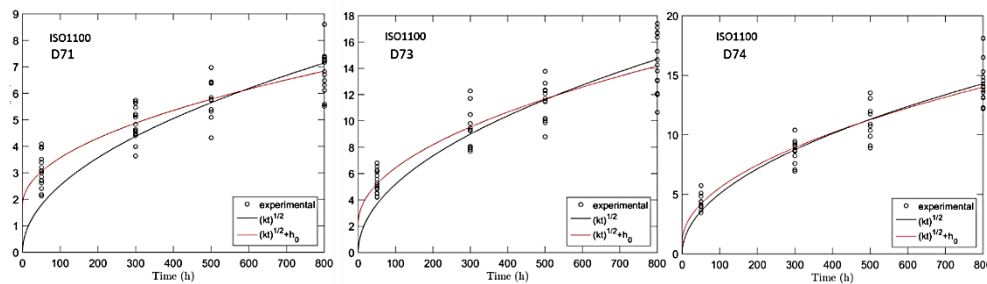


Figure 2 TGO thickness of three Fe-containing coatings during isothermal oxidation at 1100 °C.

From the analyses, oxidation behavior of the Ni-base coatings can be summarized below.

- The refined Ni-base coatings (D11-D12 and Fe-bearing D71-D75) have better resistance against total β -depletion from thermal cycling than the corresponding KME 503 coatings (D10 and D70) and external reference coating T1.
- D71 with an intermediate Fe level and without Hf addition has the best resistance to β -depletion and to scale growth in all oxidation test conditions.
- D11 with an increased Ru content shows improved resistance against inner β -depletion by interdiffusion than D10 [19].

- D12 with Ce in the place of Y shows the poorest resistance against cyclic oxidation and oxidation in the D1x group [20].

The refined Co-base coatings (B11-B14) have at the best similar but often lower resistance to oxidation in comparison with their corresponding coating in KME 503 (B10). Characterization of the as received samples discloses a large deviation in both chemical composition and coating thickness from the nominal values. Especially, the low Al-content (Table 1) and coating thickness Simulation was performed and the results show that the lower Al-content (Table 1) and a low coating thickness could indeed be the main cause of reduced resistance.

The effect of X-elements

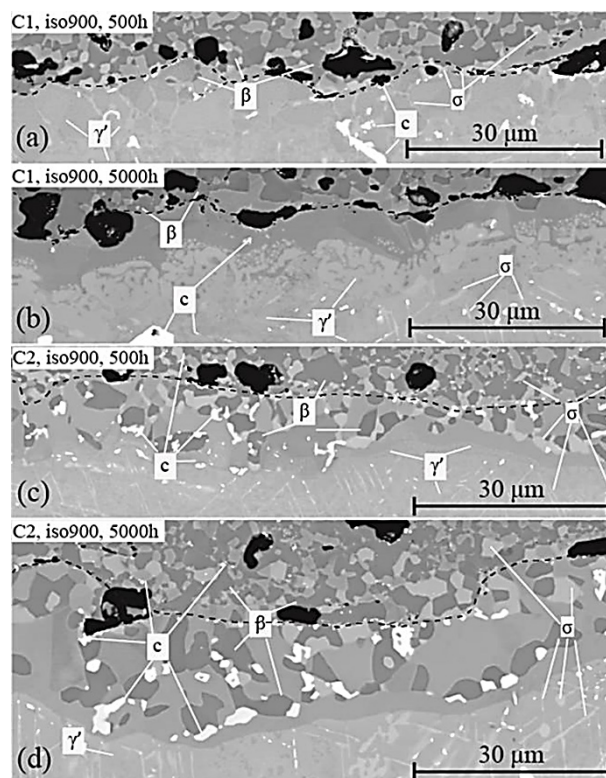


Fig. 3 Microstructure near the coating/substrate interface for D73 (c1) and D74 (c2).

- Post-test analysis indicates that the use of Ce in the place of Y seems to reduce the protective scale adherence but increase the scale growth rate, resulting in more severe scale spallation and rapid β -depletion below the oxide scale [20]. Root-like Ce-oxide observed in [21] that are considered to benefit TGO adherence also for TBC [22] have not been observed in the scale.
- Hf was observed in Phase I to precipitate in the scale that may increase scale growth rate of the doped Ni-base coating. The oxidation and TCF results in Phase II confirms reduced oxidation resistance due to a higher TGO growth rate but such a negative effect seems to be balanced by the Hf's positive effect on scale adherence.
- In addition to post analysis, thermodynamic and kinetic simulations were performed to understand the positive effect of increased Ru on oxidation

[23][24]. It can be concluded that Ru increases the solubility of Al in the β phase and the β phase fraction, thus reduces the removal of Al from the coating by interdiffusion.

- Fe also improves the oxidation resistance through interfering the coating-substrate interdiffusion, however likely by a different mechanism than Ru. Fig. 3 shows that the Fe-enrichment in the substrate due to interdiffusion promotes the formation of σ -phase. It was reported that a Cr(W)-rich σ layer can be a diffusion barrier for Al [24]. Further analysis is to be carried out.
- Simulations on the effect of Ta have been performed, which shows to have promoted the formation of γ' when Ta is over a critical value. Oxidation experiments are ongoing.

3.3 SURFACE TREATMENTS ON OXIDATION BEHAVIOUR

The effect of surface treatments, including shot-peening, mechanical polishing and surface laser melting on the oxidation behavior of a Ni-base coating were investigated. All the surface treatments have resulted in improved oxidation resistance with the shot peening being the most effective [25][26]. A detailed study with analyses using PSLS (photo stimulated luminescence spectroscopy) and SEM were carried out to compare shot peening, mechanical polishing and as-deposited coatings [25]. A brief summary is given below.

By the combination of SEM study of oxidized surface for morphology evolution, SEM cross-section observation for oxide scale structure and kinetics (example in Fig. 4), PSLS analysis for aluminum-oxide phase identification (example in Fig. 5, e.g.), PSLS for residual stress determination in the protective scale, and weight gain experiment, the oxidation mechanisms in the treated coatings were analyzed. The effect of surface treatments on the oxidation process can be summarized below with a schematic illustration in Fig. 6:

- a. A two-layer oxide scale, an outer α -Al₂O₃ layer transformed from outward grown transient alumina and an inward grown inner α -Al₂O₃ layer, forms on the coating surface. Fast growth of the transient alumina leads a thick outer layer. The rate of transformation of alumina controls the oxide growth at initial stage.
- b. Polishing and especially shot-peening treatment suppresses the fast growth of transient alumina by early formation of the protective inner α -Al₂O₃ scale, resulting in a much thinner outer α -Al₂O₃ layer.
- c. α -Al₂O₃ nucleation is aided by the presences of surface defects, such as splat boundary on polished coating surface and shot-peening induced defects. The high surface defect density in the shot-peened coating assists the fast formation of a continuous protective α -Al₂O₃ oxide layer at initial stage.
- d. Mechanical polishing reveals a β + γ two phase structure, transient alumina tend to form on the β - phase and grows fast on this region. The rate of $\theta \rightarrow \alpha$ -Al₂O₃ transformation is faster on polished coating than that of as-sprayed coating, resulting in a thinner outer α -Al₂O₃ layer than that of as-sprayed coating.

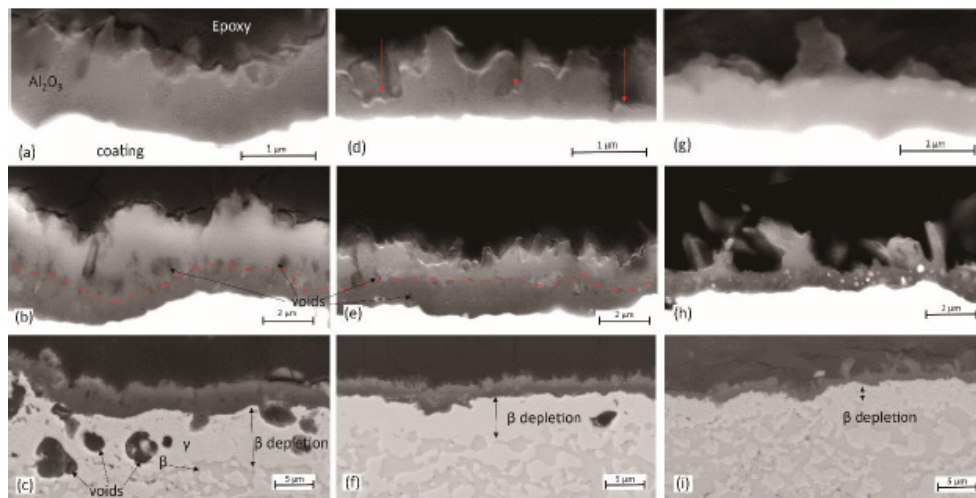


Figure 4 Back scatter electron images of cross-sections of oxide scale formed on a-c) as-sprayed, d-f) polished, g-i) shot-peened coatings. The first row: oxidation for 1 h and the second and third row: oxidation for 50 h. Red dash lines in b) and d) mark the inner and outer oxide layer interface. Note that the contrast and brightness of images were tuned to show details in the oxide scale.

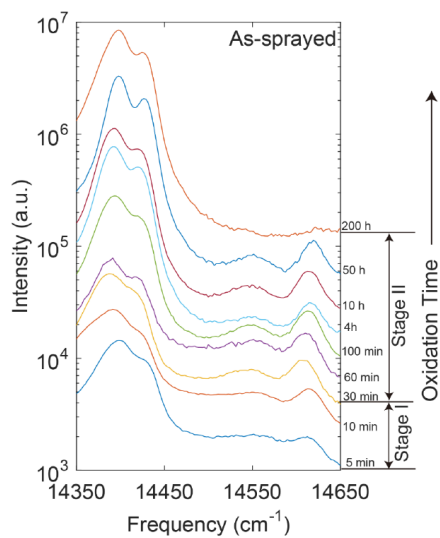


Figure 5 Evolution of photoluminescence spectra of the oxide scale formed on as-sprayed NiCoCrAlY coating at 1000 °C for various oxidation times, showing the transient oxidation development (14450-14650/cm).

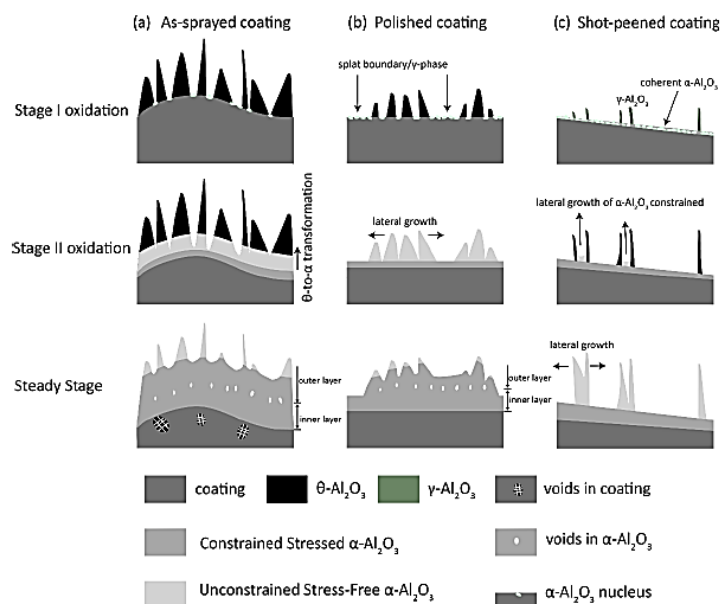


Figure 6 Schematic illustration of phase constitutions, transformation and growth of oxide scale formed on a) as-sprayed coating, b) polished coating and c) shot-peened coating at different oxidation stages.

3.4 HOT CORROSION BEHAVIOUR

Hot corrosion tests were carried out in Phase I to evaluate the corrosion resistance of Co-base coatings refined then. The same substrate, IN-792, was used. Post analyses involving XRD measurements for surface oxides and corrosion products and SEM for element distribution and microstructure have been continued in Phase II on two groups of corroded samples.

The first group was subjected to cyclic hot corrosion by salt solution of $(0.8\text{Na}, 0.2\text{K})_2\text{SO}_4$ for up to 480 hours. The process consists of solution spraying to obtain a salt concentration of $960 \mu\text{g}/\text{cm}^2$ at 100°C , heating at 900°C for 48 hours and then air cooling to 100°C for new salt deposition. It was revealed that the observed different hot corrosion behavior between the samples is more related to the different coating quality in terms of pores and splat boundaries than the difference in the addition of heavy element such as Ta, Ru and Ir [27].

The second group contains coating A (10 wt.% Al, 20 wt.% Cr) and B (7 wt.% Al, 28 wt.% Cr) which were tested using the same thermal cycles and same salt mixture as above but with three types of corrosion environments: air with salt deposition of $20\text{--}25 \mu\text{g}/(\text{cm}^2 \text{ h})$ (SD), air + 500 ppm SO_2 with salt deposition of $20\text{--}25 \mu\text{g}/(\text{cm}^2 \text{ h})$ (SS), and air + 500 ppm SO_2 (SA) [28]. Spectra from XRD measurements for understanding the development of surface damage are given in Fig. 7. It was found that corrosion in SD was by basic fluxing of alumina scale and Coating A with a higher amount of Al had better corrosion resistance. The existence of SO_2 reduced corrosion attack by the salt. A probable explanation is that the SO_2 could decrease the Na_2O activity and inhibit the basic fluxing of the oxide scale, however, sulfidation of Cr became a dominant corrosion process for Coating B. It can be concluded that for the investigated corrosive environments the coating with a higher Al/Cr ratio seems to perform better than a lower Al/Cr ration.

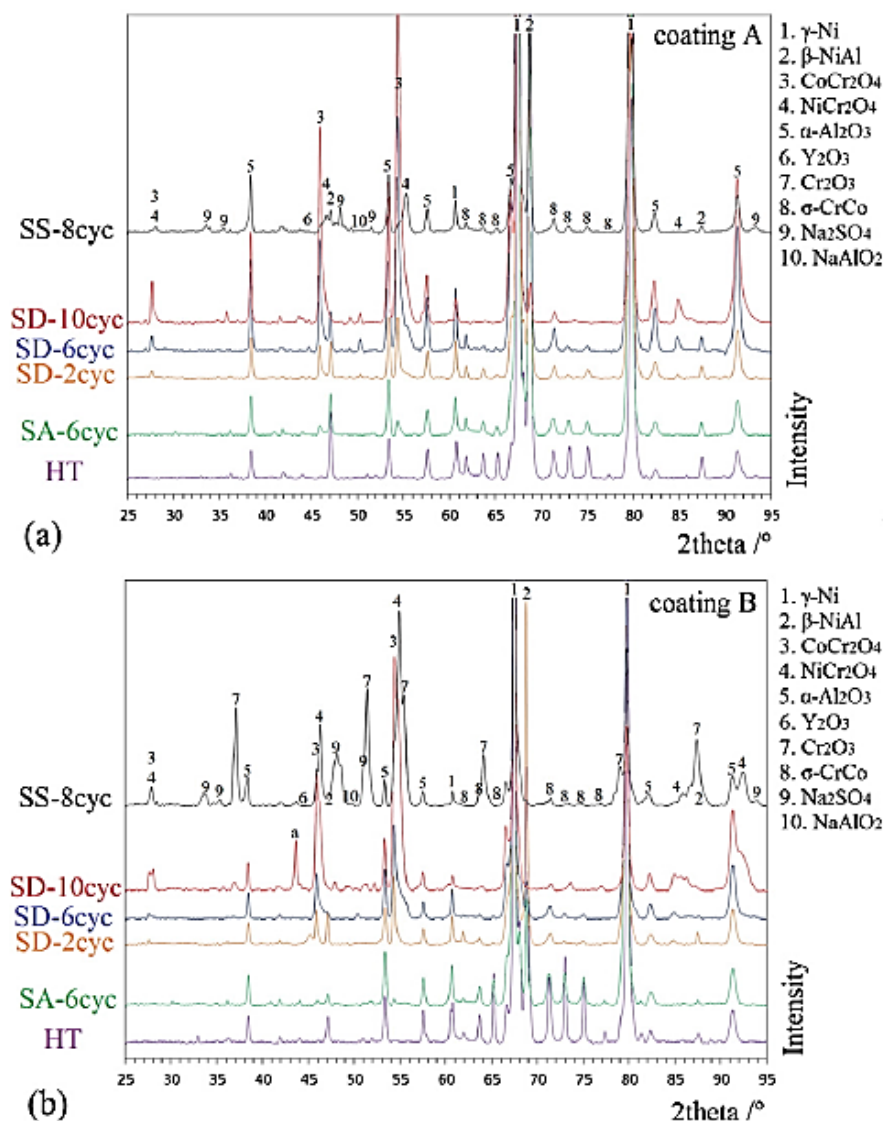


Fig. 7 XRD spectra obtained from coatings A and B after exposure in different corrosion environments.

The Co-base coatings and selected Ni-base coatings refined in Phase II were cyclic tested with a more aggressive but also frequently used salt mixture, 75 wt.% Na_2SO_4 +25 wt.% NaCl [29]. The salt deposition to obtain a concentration of 4 mg/cm² was done at room temperature and the samples were then exposed at 900 °C for 10 hours, followed by slow cooling to room temperature for start of a new cycle. As examples, A few SEM images are included to reveal the morphology of as corroded surface (Fig. 8, left) of refined Ni-base coatings or cross-sectional microstructure of refined Ni-base (Fig. 8, right) and Co-base coatings (Fig. 9).

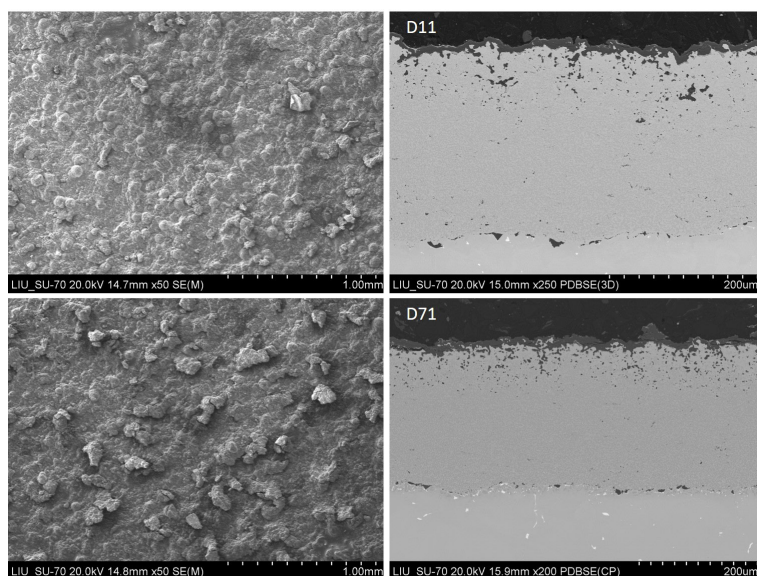


Fig. 8 Top view (left) and cross section microstructure (right) of selected Ni-base coatings after 27 cycles hot corrosion test.

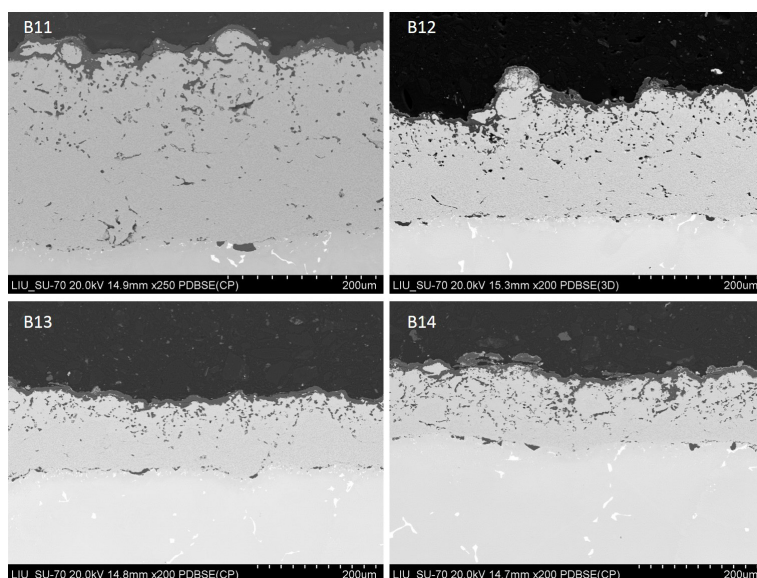


Figure 9 Cross-sectional microstructure for the refined Co-base coatings after 21 cycles hot corrosion test.

Main results from corrosion test on the refined Ni-base and Co-base samples are summarized as follows:

- A typical type I hot corrosion attack occurred, via the basic fluxing mechanism due to the lack of SO_3 gas. Al content is therefore the dominant factor controlling the hot corrosion resistance in current study. Minor Cr-depletion was detected, while Al depletion is readily observed in corrosion front.
- Internal oxidation was observed on all coatings and sulfur penetration was detected in those voids formed in coating.
- The addition of Fe up to 10 wt.% does not seem to have a negative effect on coating's hot corrosion resistance.

- Severe internal oxidation was observed in the Co-base coatings, attributed to the low Al content and thin coating thickness.

Edge attack is the major problem during hot corrosion for one-side coated samples. A stress-assisted mechanism has been proposed but is not presented here.

3.5 DBTT AND TMF BEHAVIOUR

The ductile-to-brittle transition temperature (DBTT) was determined for selected coatings on 3 substrates, polycrystalline In792, single crystal CMSX-4 and CM247LC. A uniaxial tensile load was applied on a coated test bar and the tensile strain at coating failure detected by an acoustic emission device was obtained. The DBTT was evaluated from the evolution of coating failure strain with testing temperature. Details of the test set-up can be found in [30]. The coating that performs best in oxidation test, D71, has the highest DBTT temperature, which may limit its application. In general, better ductility is observed with a substrate of single crystal.

OP-TMF tests were performed on D10 and D11 coated Stal15 test bars. Mechanical polishing was performed after the coating deposition to reduce the coating surface roughness to below 1 μm . The test spectrum follows SIT's standard and 3 strain levels were used. It can be seen in Fig. 10 that D10 and D11 coated samples show a similar TMF life. Video recording of the coating surface during TMF test reveal a high density of microcracks developed with increasing TMF cycles (Fig. 11). Damage curves are therefore derived from the measured number of microcracks and their length (Fig. 12). In general, both the crack density and the length increase with increasing TMF cycle. However, the crack number density in D10 is much higher than in D11 samples.

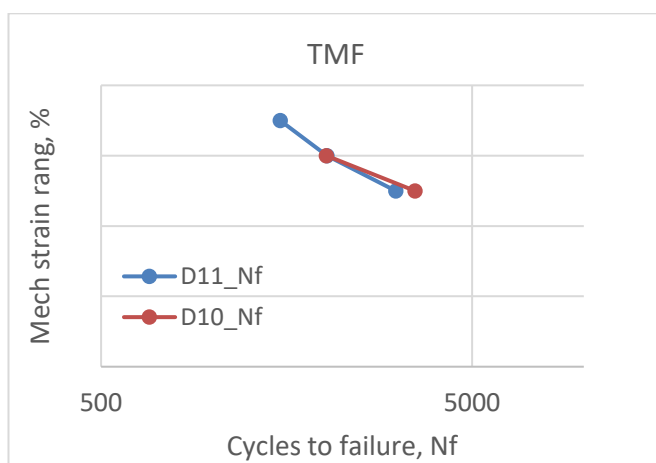


Figure 10 TMF results for two Ni-base coatings

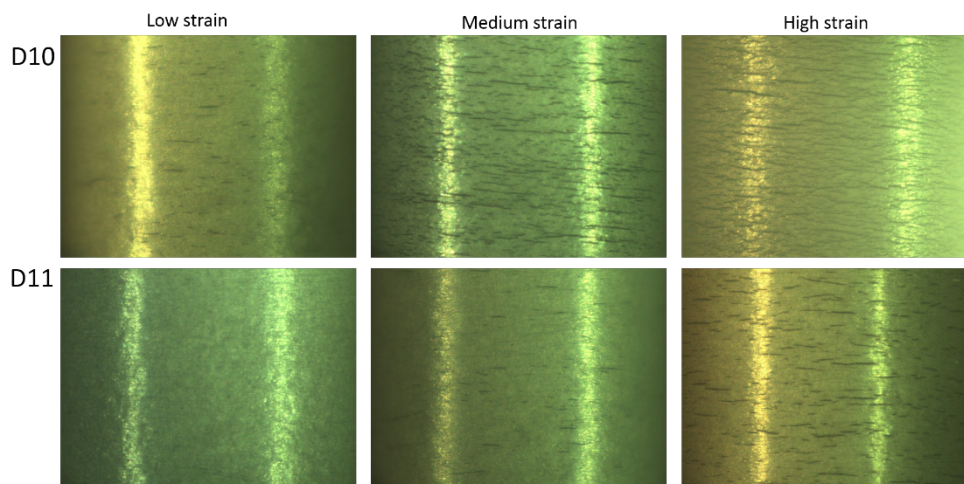


Fig. 11 Surface defect distribution in D10 and D11 coated Stal15 samples.

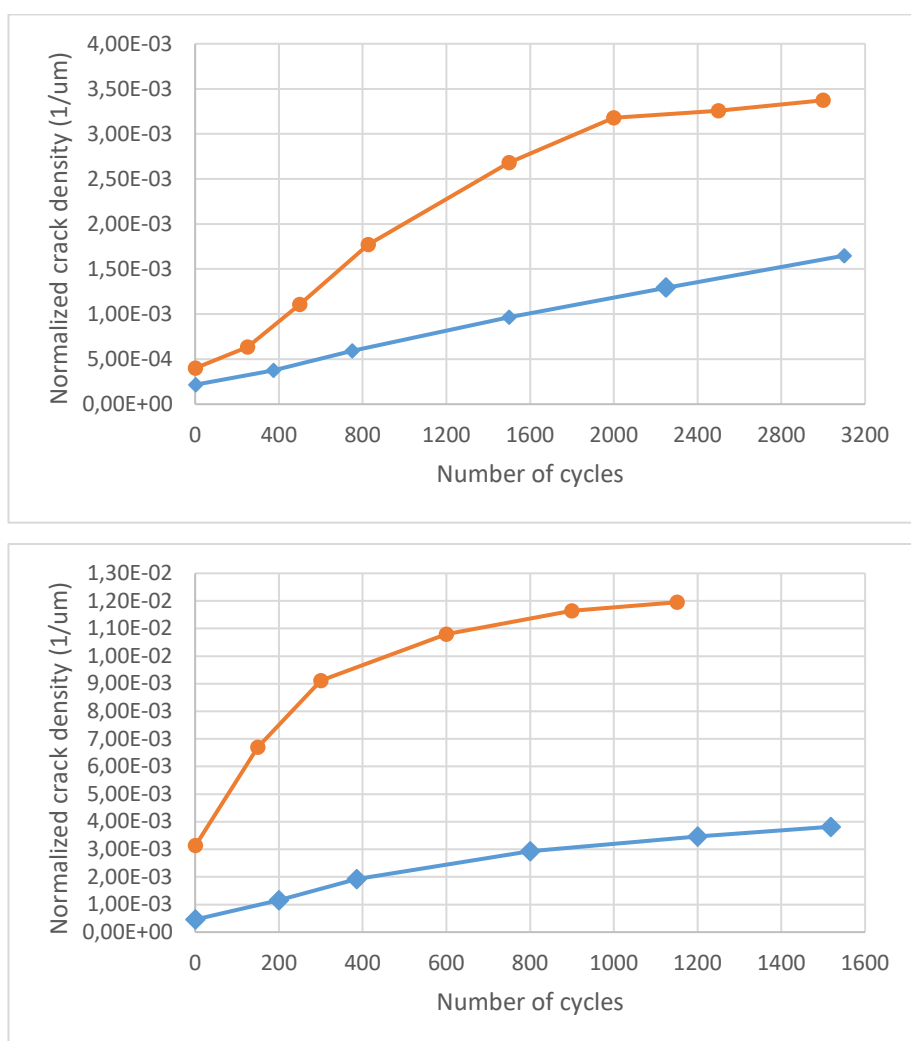


Figure 12 Development of crack number density with increasing TMF cycles for D10 (dots) and D11 (diamonds) coated sample at low (upper) and high (lower) strain level.

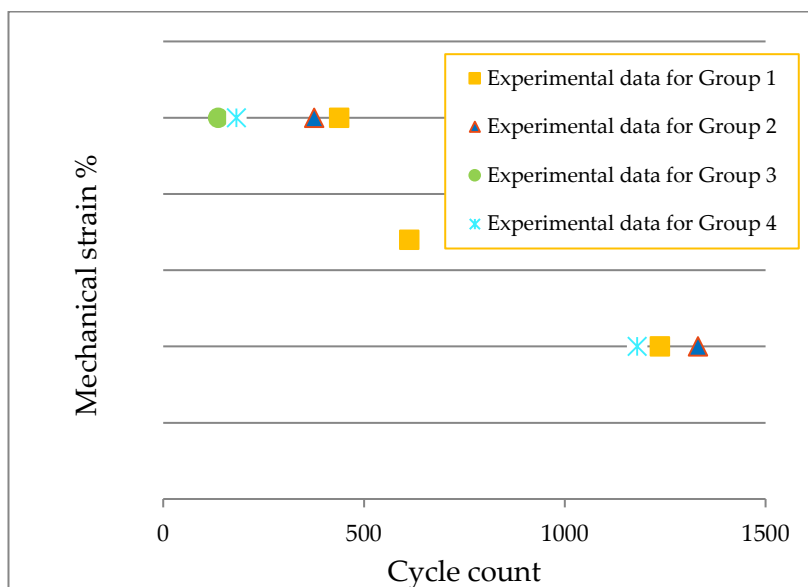


Figure 13 Comparison of TMF life between D10, D11, and D71 (Group 1, 2, and 3 respectively) coated IN792. Group 4 is the uncoated IN792.

Fig. 13 summarizes TMF results from OP-TMF tests on D10, D11 and D71 coated IN792 bars together with those uncoated IN792 (a polycrystalline superalloy). Few cracks in the coatings were observed. Also two different failure mechanisms could be derived from analysis of the fractured surface. For D71, it seems that necking of substrate induced contraction has resulted in delamination of the coating. For D10 sample, the failure of coating is accompanied by failure of substrate from cyclic softening.

The TMF results show that the surface crack propagation and failure mode depended on coating compositions as well as substrate property. However, the application of the coatings does not seem to have a significant effect on the TMF life of the substrate alloys.

3.6 BONDCOAT FUNCTION

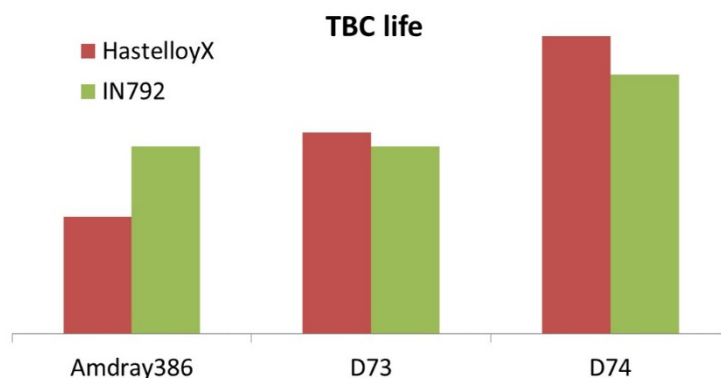


Figure 14 Comparison of TCF life between different TBC systems.

D73 and D74 coating were used as bondcoat in APS TBC as well as in SPS TBC system. Thermal cyclic fatigue (TCF) testing is performed to characterize the cyclic performance of TBCs at high temperatures. On two different substrates, the SPS TBCs with D74 show better coating life than the D73 and the commercial bond coat (Fig. 14). The higher aluminium activity of D74 that may promote early formation of a protective alumina could have been the explanation[31][26].

3.7 MODELLING

Formation of secondary β -layer

A continuous secondary β -layer was observed in the coating near the MCrAlY/CMSX-4 substrate interface after being exposed to a certain number of TCF cycles, as shown in Fig. 15. Considering possible benefit of such a structure layer for oxidation through reducing interdiffusion as well as providing Al for surface scale repairing, the oxidation-diffusion model was adapted to exploit the condition for its formation. The results presented in Fig. 16 indicate that in order to obtain such a layer during oxidation, the Al-activity in both the coating and substrate have to reach their respective critical values [32][33]. The benefit of formation of such a layer for oxidation resistance was also confirmed by simulation.

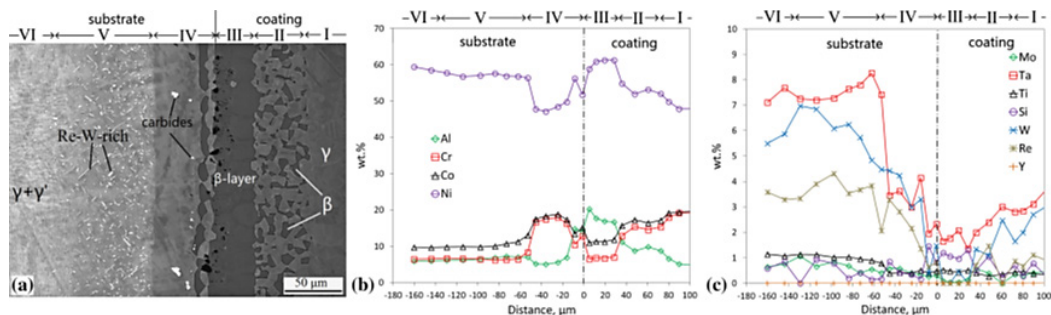


Figure 15 Cross section of c1-s1 sample after the TCF test (a) microstructures, (b) and (c) concentration profiles of elements across the cross section in (a).

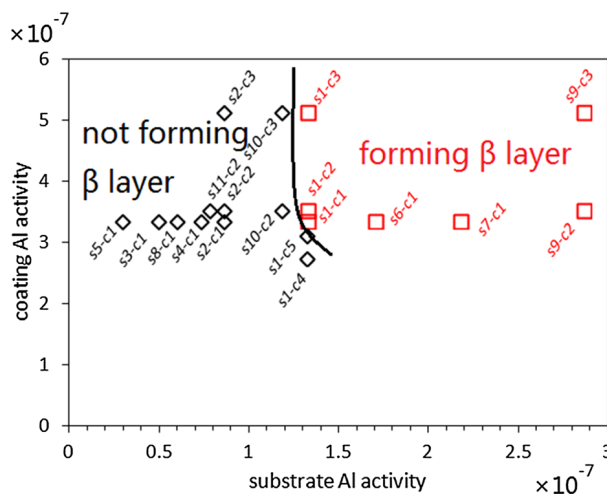


Figure 16 The activity-activity map of Al showing the region for the formation of a continuous β -layer at coating-substrate interface based on simulations in different substrate-coating systems

The formation of the β -layer requires both Al and Ni accumulation near the interface. Simulation using a 1-D diffusion model on Ni shows that its uphill diffusion could indeed occur (Fig. 17).

Isothermal and TCF experiments on a coated system that was predicted the formation of a β -layer showed the formation of a discontinuous β -layer. More experiments on for example other coating/substrate systems need to be done.

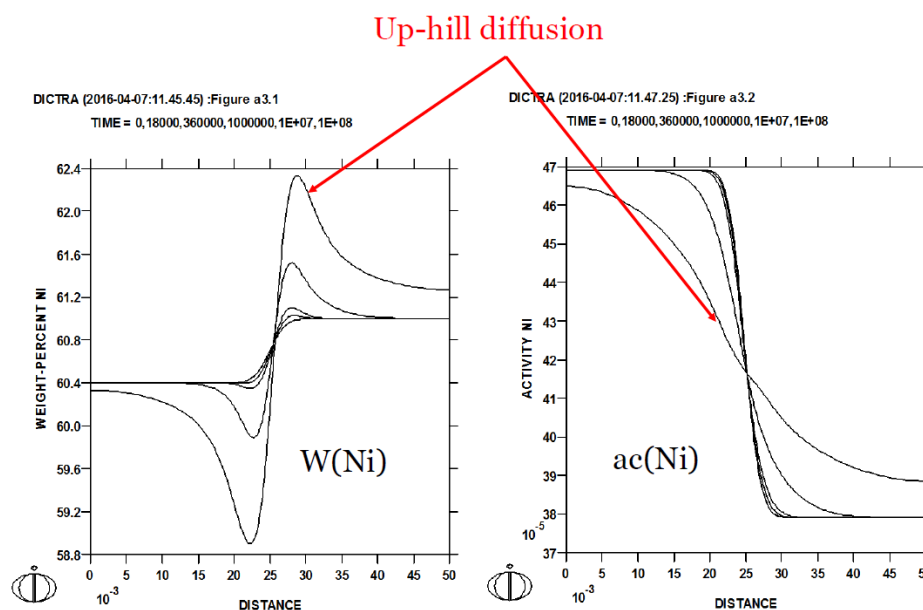


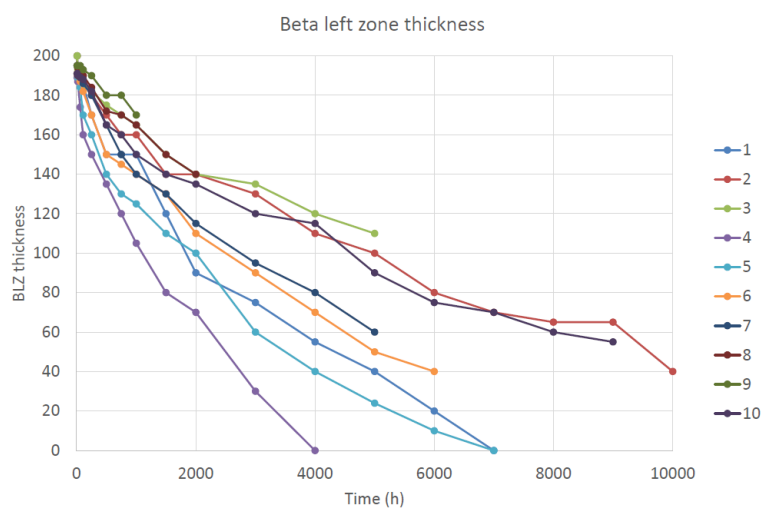
Figure 17 Simulation results of Ni-concentration profile (left) and Ni-activity (right) showing increase of Ni concentration near the coating-substrate interface with time through uphill diffusion.

Chemical matching of coating/substrate

Simulation of coating and substrate matching was performed to investigate the role of substrate during oxidation. The simulation test matrix is listed in Table 4. Figure 18 shows calculated β -phase left zone thickness at 950 °C oxidation. It can be seen that a high Al content of substrate shows the best resistance against β -depletion, and the matching of high Al activity of both coating and substrate showed the best performance. In the light of the work on oxidation life criterion to be described below, new analysis will be carried out to analyze the oxidation behaviour with regard to Al-activity of the coating and substrate.

Table 4 Simulation test matrix.

No	COAT	SUB	SUB	COAT	Al_sub	Al_coat
1	high c(AL)	Low	Rene 41	Armdry365	1.5	12.5
2		Medium	In-792	Armdry365	3.4	12.5
3		High	CMSX-4	Armdry365	5.6	12.5
4	low c(AL)	Low	Rene 41	Armdry997	1.5	8.5
5		Medium	In-792	Armdry997	3.4	8.5
6		High	CMSX-4	Armdry997	5.6	8.5
7	high ac(AL)	Low ac	Rene 41	HighacAl	1.5	10.5
8		Medium ac	In-792	HighacAl	3.4	10.5
9		High ac	CMSX-4	HighacAl	5.6	10.5
10			HX	Armdry365	0.15	12.5

**Figure 18 Results of β -phase left zone thickness for coating systems in Table 3.**

An Al-activity based criterion for oxidation life prediction

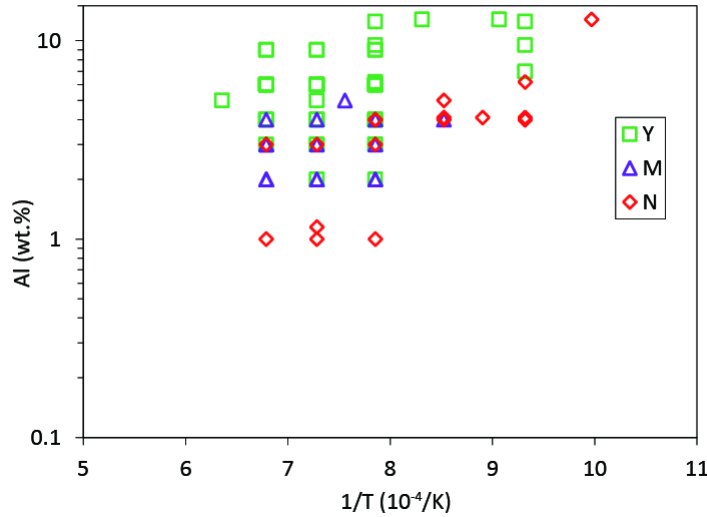


Figure 19 A summary of literature data relating showing the Al-concentration and oxidation stage. □ Y: alloys able to form a single scale of alumina, △ M: alloys able to form an alumina scale with other oxides outside, ◇ N: alloys unable to form a continuous alumina and severe internal oxidation occurs.

In association with the development of the diffusion-oxidation model for coating life prediction in the project, efforts have also been directed on finding a parameter to replace the Al-concentration for more accurate prediction of MCrAlX coatings' life [34]. A survey of published oxidation data for the correlation between the formation of TGO and the Al-level below it were made on Ni-Al and Ni-Cr-Al alloys. The collected data in Figure 18 do not reveal a determined Al-concentration boundary for the formation of a continuous α -alumina. Reanalysis of the data leading to Fig. 19 eventually results in the appearance of Fig. 20 with the Al-activity instead of the Al-concentration as an important parameter. The Al-activity was calculated from the chemical composition below TGO using the Thermo-Calc software and database, which is largely affected by the chemical composition (Fig. 21). A clear boundary separates those compositions giving a continuous protective scale from those that cannot retain such a scale. Fitting to the boundary data points thus produces a temperature dependent oxidation life criterion.

$$a_{Al,cri} = \exp(-10235/T - 11.1)$$

In which $a_{Al,cri}$ is the critical Al-activity and T the temperature. Application of the criterion on a couple of coatings in the project shows that the calculated oxidation life agrees with the experimental observation.

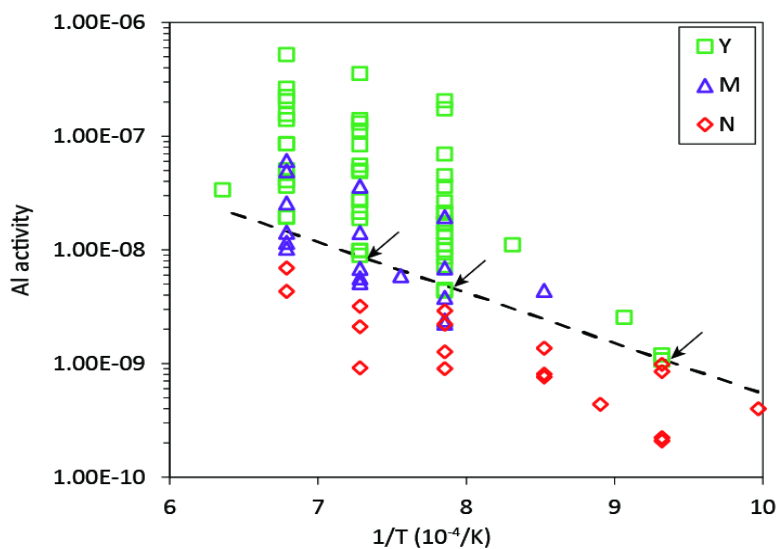


Figure 20 Calculated Al-activity for alloys in Fig. 19 plotted as a function of inverse of temperature.

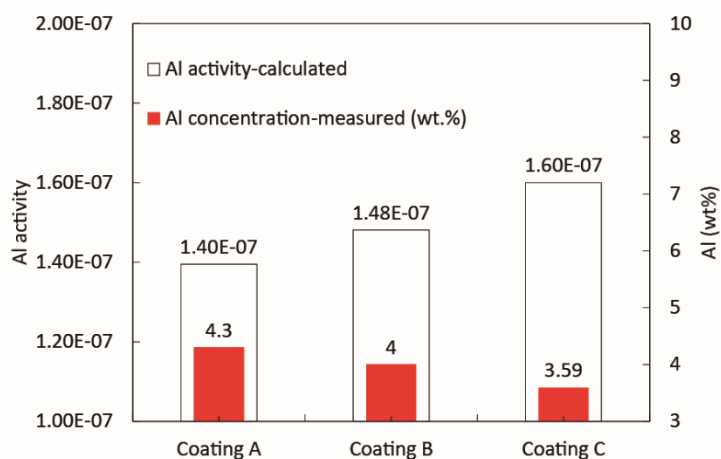


Figure 21 Comparison of the calculated Al activity and Al concentration in coatings under alumina scales after oxidation for 10,000 hours.

4 Discussion and Conclusions

Based on analysis of the results from Phase I and with the help of thermodynamic simulation, chemical composition refinement was carried out on two Ni-based and one Co-based MCrAlX coatings from Phase I. Seven new Ni-base coating compositions and four new Co-coating compositions were studied in Phase II. The refinement on the Ni-base coatings has in general improved their oxidation resistance. However, no improvement or even reduced oxidation resistance was observed for the refined Co-base coatings. This could be attributed to the observed deviation of coating composition especially the Al content and thickness from the nominal values in the received test samples. Limitation in budget and time did not allow for manufacturing and testing of new Co-coatings samples with better composition and thickness control.

The addition of a certain amount of Fe and Ru has contributed to better oxidation resistance, mostly derived from interference with interdiffusion. Both elements reduce the loss of Al due to its inward diffusion towards the substrate but by different mechanism. Replacement of Y by Ce or the addition of Hf does not seem to have positive effect on oxidation resistance.

For the corrosion environments investigated in the project, the coatings seem to provide good protection. Much severe corrosion occurs at the sample edge where the substrate was naked than in the center of the samples. The Ni-based MCrAlX coatings perform in general better than the Co-base coatings. Further, a higher Al/Cr ratio gives a better resistance.

Surface treatments including shot peening, mechanical polishing and laser remelting improve oxidation resistance. Modification of the surface condition affects the initial oxidation stage, leading to a slow scale growth rate. A scale of low growth rate can be especially beneficial for MCrAlX as bondcoat in strain tolerant TBC systems.

TMF behavior of coated bars depends on both the coating composition and the substrate material. However, the application of the coatings seems to have an insignificant or no effect on the substrate TMF life.

A temperature dependent criterion based on the estimation of Al-activity has been established and verified against long time oxidation tests. The Al-activity, considering the collective effect of composition in the coating surface on its ability to form the protective scale is more realistic than the oversimplified criterion of the minimal Al-concentration or complete β -depletion. In addition, changes in the coating induced by interdiffusion between coating-substrate seems to be also more dependent on the Al-activity than the Al-concentration. Further study of the correlation between Al-activity and coating behaviour may increase understanding of the coatings composition effect, which can lead to the development of better coatings and coating life models.

The coating systems studied in the project contain over 10 elements. Correlation of the high temperature behaviour to the chemical composition is often challenging without a substitution composition matrix. The project has shown that

thermodynamic and kinetic simulations can be effective tools for coating design and development, selecting of coatings with regard to substrate composition, evaluating of coating system degradation under exposure to high temperatures, etc.

5 Literature references

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6 Publications

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5. K. Yuan, R. Lin Peng, X.-H. Li, S. Johansson, Y.-D. Wang: "Some aspects of elemental behaviour in HVOF MCrAlY coatings in high-temperature oxidation" (2015) Surface and Coatings Technology, 261, pp. 86-101.
6. K. Yuan, R. Lin Peng, X.-H. Li: "Formation of a continuous β -NiAl layer near the interface in a γ + β MCrAlY" (2015) Proc ITSC 2015, 1, pp. 54-61.
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7 Appendices

Svensk titel på projektet	
Nya ytbeläggningar av MCrAlX-typ för krävande gasturbintillämpningar	
English project title	
Durable MCrAlX coatings for demanding application in gasturbines	
Universitet/högskola/företag	Avdelning/institution
Linköping University (LiU)	Department of Management and Engineering
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Universitet/högskola/företag	
Siemens Industrial Turbomachinery AB in Finspång	
Adress	
Linköping University (LiU)	
Ru Lin Peng	
Namn på ev övriga projektdeltagare	
Pimin Zhang (LiU), Kang Yang (LiU), Xin-Hai Li (SIT), Sten Johansson (LiU), several other colleagues at SIT	
Nyckelord:	
MCrAlX coatings, oxidation, corrosion, oxidation life criterion, TMF	

DURABLE MCrAlX COATINGS IN GASTURBINES

Improved oxidation and thermal cycling resistance has been achieved for two Ni-based MCrAlX coatings after the further refinement. Coating data for short term and long term oxidation behaviour, thermal cycling resistance, corrosion behaviour and mechanical properties have been obtained for a number of Ni-based and Co-based coatings. An Al-activity base criterion has been established for more accurate prediction of oxidation life of MCrAlX coatings. More than ten journal/conference papers based on the project work were published, which covers coating design and life prediction, oxidation, corrosion, and etc. Eventual implementation of the coatings/results can contribute to increased gas turbine performance and operational flexibility.

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