



HIGH TEMPERATURE OXIDATION BEHAVIOUR OF PLASMA SPRAYED NiCrAlY COATINGS ON Ni-BASED SUPERALLOYS IN AIR

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ABSTRACT

Plasma spray coating technologies are capable of depositing a very wide range of compositions without significantly heating the substrate. In the present investigation NiCrAlY metallic coatings were deposited on two Ni-based Superalloys, Superni 75 and Superni 600 by plasma spray process. Oxidation studies were conducted on uncoated as well as plasma spray coated superalloys in air at 900°C under cyclic conditions. Each cycle consisted of one hour heating followed by 20 minutes of cooling in air. The thermogravimetric technique was used to approximate the kinetics of oxidation. All the coated as well as uncoated superalloys nearly followed parabolic rate law. The uncoated Superni 600 showed a minimum weight gain, whereas the NiCrAlY coated Superni 75 a maximum after 50 cycles of exposure among the cases studied. X-Ray Diffraction, SEM/EDAX and EPMA techniques were used to analyse the oxidation products. The main phases analysed for the coated superalloys are oxides of nickel, chromium and aluminium and spinel of nickel and chromium, which are suggested to be responsible for developing oxidation resistance at high temperatures.

Keywords: Superalloys, NiCrAlY coatings, plasma spray coatings, high temperature oxidation.

1. INTRODUCTION

Metal and alloys get oxidised when they are heated to elevated temperatures in air or highly oxidising environments, such as a combustion gas with excess of air or oxygen. They often rely on the oxidation reaction to develop a protective oxide scale to resist corrosion attack, such as sulphidation, carburisation, ash/salt deposit corrosion etc. That is why oxidation is considered to be the most important high-temperature corrosion reaction. Further the rate of oxidation for metals and alloys increases with increasing temperature ¹.

Superalloys are frequently used in high temperature applications such as gas turbine and energy conversion systems etc. During operation, blades and vanes of gas turbine are subjected to high thermal stresses and mechanical loads. In addition, they are also attacked chemically by oxidation and/or high temperature corrosion ². According to Eliaz et al ³ the superalloys at gas turbine engines serve most of the time in an oxidising environment. Therefore high temperature oxidation of superalloys has attracted the attention of many researchers in the past as well as in the recent times. Further, the superalloys are not able to meet both the high-temperature strength and the high-temperature corrosion/oxidation resistance simultaneously so protective coatings on superalloys are used to counter the latter. Thus, the oxidation behaviour is very important for high-temperature coatings ⁴.

Although protective surface treatments are widely used at low temperature, the use of these at elevated temperature is relatively more recent. High temperature applications are limited largely

to the aerospace industry. An enormous challenge exists to develop and apply these techniques to other high temperature applications⁵. Among the various coating methods, advanced plasma spray technique has many advantages such as high productivity for thick coating films of more than 100 μ m and good applicability for a wide range of coating materials including ceramic powder. Furthermore, the process does not cause degradation of the mechanical properties of the alloy substrate^{6,7}.

Overlay coatings include a family of corrosion resistant alloys specially designed for high temperature surface protection. They are often referred to as MCrAlY coatings, where M is the alloy base metal (typically nickel, cobalt, or combination of these two)³. Furthermore MCrAlY overlays are also used bond coats in thermal barrier coatings and oxidation of the bond coat remains an important issue⁸. Various researchers have reported oxidation studies on thermal sprayed MCrAlY coatings at elevated temperatures in the recent past⁹⁻²⁰.

Objective of the present work is to study the oxidation behaviour of two Ni-based superalloys with and without plasma spray NiCrAlY coatings under cyclic conditions at a temperature of 900°C in air. Knowledge of the reaction kinetics and the nature of the surface scales formed during oxidation are important for evaluating the alloys for their use and degradation characteristics in high-temperature applications²¹. The Ni-base superalloys under study were procured from Mishra Dhatu Nigam Limited, Hyderabad (India), first being Superni 75 (Similar grade Nimonic 75) and the second Superni 600 (Similar grade Inconel 600). Superni 75 finds applications in Gas Turbine and boiler parts, whereas Superni 600 is being utilised for Furnace parts and heat treatment jigs. NiCrAlY alloy powder was deposited on these superalloys by plasma spray process from the point of view of its assessment in high temperature oxidising environments.

2. EXPERIMENTAL PROCEDURE

2.1. Development of coatings

Ni-based superalloys namely Superni 75 and Superni 601 were obtained in the rolled sheet form. The chemical composition of Superni 75 is 3.0 Fe – 19.5 Cr - 0.3 Ti -0.1 C – Bal Ni, whereas Superni 600 has 10.2 (max) Fe – 15.5 Cr - 0.5 Mn -0.2 C – Bal Ni. Specimens each measuring approximately 20mmX15mmX5mm was cut from the sheets. The specimens were polished and grit blasted with alumina powders (grit 60) before being plasma sprayed. NiCrAlY coatings were then formulated on the Ni-base superalloys specimens with 40kW Miller Thermal Plasma Spray Gun. All the process parameters including the spray distance were constant throughout coating process with an arc current of 700A, arc voltage 35 V, powder flow rate 3.2 rev. /min, spraying distance 90-110 mm, the plasma arc gas (Argon) and powder carrier gas (Argon) pressures of 59 and 40 psi respectively. The details of coating powder and porosity (2.88-4.42%) measurement of as-sprayed coatings are reported in the earlier publication of the authors²². The average thickness of the coatings as measured from Back Scattered Electron Images (BSEI) was around 228 μ m.

2.2 High temperature oxidation tests

Cyclic oxidation studies were performed in static air for 50 cycles. Each cycle was consisting of one hour heating at 900°C in Silicon Carbide tube furnace followed by 20 minutes cooling at room temperature. The specimens were kept in alumina boats and then the boats were inserted in the furnace. Aim of the cyclic oxidation was to create accelerated conditions for testing. The studies were performed for uncoated as well as plasma spray coated specimens for the purpose of comparison. The specimens were polished down to 1 μ m alumina wheel cloth polishing

before oxidation run. The weight change measurements were made at the end of each cycle with help of Electronic Balance Model 06120 (Contech) with a sensitivity of 1 mg. The spalled scale was also included at the time of measurements of weight change to determine total rate of corrosion. Efforts were made to formulate the kinetics of corrosion. The samples after oxidation were analysed by XRD, SEM/ EDAX and EPMA.

Scanning Electron Microscope [JEOL (JSM-5800)] with EDAX attachment of Oxford (Model-6841) made in England was used for surface SEM/EDAX analysis of the oxidised specimens. The equipment can directly indicate the phases (oxides) present alongwith their compositions based on built-in EDAX software, which is a patented product of Oxford ISIS300. XRD analysis of some of the oxidised specimens was carried out with Philips X-ray diffractometer, model PW 1140/90 using Cu target, whereas that of others was done with Bruker AXS D-8 Advance Diffractometer (Germany) with CuK_α radiation. The oxidised specimens were then cut across the cross-sections with ISOMET 1000 Precision Diamond Cutter. These cross-sections were then mounted in transoptic mounting resin, mirror-polished and carbon coated to obtain X-ray mappings of the various elements present across scale with JXA-8600M electron probe micro analyser. X-ray mappings were obtained for all the elements of the substrate as well as of the coatings, but only those important from the point of view of discussion are reported to limit the size of the paper.

3. RESULTS

3.1 Cyclic oxidation in air

Colour of oxide scale formed on the uncoated superalloys Superni 75 and 600 after air oxidation for 50 cycles at 900°C was dark grey, in general, with some brownish spots. In case of Superni 75, greenish tinges were also observed in the scale in the early cycles of study. Few white spots could also be seen on the scale of Superni 75 from 22nd cycle onwards, which vanished afterwards towards the end of cycles. Lustrous scales were formed upto mid cycles of the study, which eventually went on becoming dull with increasing number of cycles. The scales of the Superni 75 and 600 showed no spalling of the scales and scales were very intact.

Whereas the plasma spray NiCrAlY coated superalloys have shown the formation of smooth scales without the presence of cracks, when subjected to cyclic oxidation for 50 cycles at 900°C . Colour of the oxide scales for the coated superalloys at the end of the study was observed to be greenish grey, near to dark green. The colour of scale changed from grey with minor green tinges to greenish grey with the progress. The scales were found to be lustrous, with no tendency shown for spalling. The NiCrAlY coating showed very good adherence to both the superalloys over most of the surface area of the specimens, during the course of cyclic oxidation studies, except at the edges. The development of cracks in the coatings at the edges was observed after the very first 1-2 cycles in both the cases. This cracking resulted in spalling of coating from one or two edges in form of flakes during the subsequent cycles. These flakes looked like as if were consisting of some outer layers of the coating, as it has been observed that the edges after spalling were still containing the dark green coating on them. This spalling of the coating from edges stopped in 37th and 42nd cycle respectively for the coated Superni 75 and 600 respectively.

Weight change (mg/cm^2) versus number of cycles plots for the uncoated as well as plasma spray NiCrAlY coated superalloys Superni 75 and 600 oxidised at 900°C in air upto 50 cycles are shown in Fig. 1. It can be inferred from Fig. 1 that the oxidation rate can be divided into two stages, one being the transient oxidation stage which shows initial rapid oxidation rate and the other is the steady state oxidation stage. In the steady state oxidation stage the oxidation rate has become nearly constant. The total weight gain at the end of 50 cycles for the uncoated

superalloys Superni 75 and 600 is 3.501 and 0.200 mg/cm² respectively, whereas corresponding values for the NiCrAlY coated Superni 75 and 600 are 4.388 and 3.815 mg/cm², refer Fig. 2. This shows that the weight gain in case of oxidised Superni 600 is lowest, whereas it is highest in the case of NiCrAlY coated Superni 75. In Fig. 3, the (weight gain/unit area)² versus number of cycles plot are shown for all the cases to ascertain conformance with the parabolic rate law. Although some scatter in the data can be observed in the plots, but it is apparent that these data can be approximated by a parabolic relationship. Ignoring this scatter in the data, values of the parabolic rate constant (K_p) are calculated as 27.30 and 6.11×10^{-12} g² cm⁻⁴ s⁻¹ for the uncoated Superni 75 and 600 respectively, whereas for the NiCrAlY coated superalloys Superni 75 and 600 the values are evaluated as 4.67 and 4.21×10^{-11} g² cm⁻⁴ s⁻¹ respectively.

3.2. X-ray diffraction analysis

XRD diffractograms for the uncoated and coated superalloys after exposure to air at 900°C for 50 cycles are depicted in Fig. 4 and Fig. 5 on reduced scales. As is clear from the diffractograms in Fig. 4 NiO, Cr₂O₃ and NiCr₂O₄ are the phases which are found to be present in both the oxidised superalloys Superni 75 and Superni 600, whereas Fe₂O₃ is revealed in Superni 600 case only. Further, it can be seen from the diffractograms, Fig. 5 that both the NiCrAlY coated superalloys have indicated the formation of NiO, Al₂O₃ and NiCr₂O₄ as main phases after oxidation alongwith some relatively weak peaks of Cr₂O₃.

3.3 SEM/EDAX analysis

SEM/EDAX micrograph of the scale formed after 50 cycles of oxidation in air 900°C for Superni 75 superalloy indicates mainly white crystalline phase rich in NiO, irregularly dispersed in the black phase (matrix) rich in Cr₂O₃, Fig. 6 (a). While SEM micrograph of top surface of the scale in case of Superni 600 superalloy indicates intergranular cracks, which mainly contain Cr₂O₃ and NiO, point 1 in Fig. 6 (b). Fine globules (white) are mainly dispersed in the black matrix, alongwith the presence of some coarse globules (Cr₂O₃-rich). The matrix is mainly rich in NiO (59%) as indicated by EDAX analysis at point 2. Further, SEM micrographs showed in Fig. 7 point out similar morphologies of the oxide scales for both the NiCrAlY coated superalloys. SEM/EDAX shown in Fig. 7 (a) for the coated Superni 75 indicates that the oxide scale contains mainly NiO with little amount of oxides of Cr and Al. Whereas in case of Superni 600 coated specimen, white (NiO and Cr₂O₃ rich) areas can be observed to be dispersed in the scale. The presence of small amounts of Fe₂O₃ (2%) and SiO₂ (1%) at point 2, Fig. 7 (b) indicates the probable diffusion of Fe and Si from the base superalloy.

3.4 EPMA analysis

BSEI and X-ray mappings for a part of oxide scale of Superni 75 superalloy after oxidation in air for 50 cycles at 900°C are shown in Fig. 8. The micrographs indicate a dense scale, which mainly contains chromium and titanium. Whereas BSEI and X-ray mappings for the oxidised Superni 600, Fig. 9 show dense but thinner scale as compared to that of Superni 75. The scale consists of two layers; the upper layer is rich in nickel and iron while the lower layer is mainly having chromium and manganese with traces of silicon.

Further corresponding analysis for the NiCrAlY coated superalloy Superni 75 (Fig. 10) indicate that the scale contains nickel in high concentrations with substantial amounts of chromium and aluminium and at most of the places Ni and Al exist alternatively in layers. Yttrium and titanium are found to be uniformly distributed in the scale. The presence of titanium in the upper scale indicates diffusion of the same from the substrate superalloy into the scale. A scale consisting mainly of nickel with uniform distribution of chromium, which also contains aluminium, could be seen for the coated Superni 600 as depicted in Fig. 11.

Aluminium has shown its presence in high concentrations at the places where nickel is absent. Yttrium has shown tendency towards clustering, while iron has diffused into the scale, which is very intensive near the coating/substrate interface.

4. DISCUSSION

The weight change plots for the NiCrAlY as well as uncoated superalloys Superni 75 and 600 indicate that the oxidation behaviour has shown conformance to parabolic rate law. The small deviations from the parabolic rate law have also been observed by Levy et al²³ during studies on the oxidation and hot corrosion of some Ni-base advanced superalloys at 704 to 1093°C. They attributed these deviations to cracking and spalling of the oxide scales. Greene and Finfrock²⁴ have also reported some scatter in the mass gain per unit area versus time of oxidation data in the early stages of exposure for Inconel 718. XRD analysis for the oxidised superalloys has revealed mainly NiO, Cr₂O₃ and NiCr₂O₄ phases, which is supported by EDAX and EPMA analysis. Deb et al²⁵ have also reported the formation of phases like NiO and Cr₂O₃ during their oxidation studies on a cast Ni-base superalloy and have opined that the scale will have a continuous outer layer of Cr₂O₃. Li et al²⁶ have also reported NiO, Cr₂O₃ and NiCr₂O₄ for a single crystal Ni-base superalloy oxidised at 900°C in air.

Further, it is well known fact that the superalloys rely on selective oxidation approach to obtain oxidation resistance, which consists of oxidising essentially one element in the superalloy and relying upon this element's oxide for protection. Moreover in case of Ni-base superalloys, chromium or aluminium is the element, which provides required protection by selective oxidation²⁷. In the superalloys under study this element is chromium. It can be seen in Fig. 8 and 9 that the chromium has formed a continuous and adherent layer in both the uncoated superalloys Superni 75 and 600 cases, after 50 cycle oxidation. This chromia layer in turn blocks the diffusion of oxygen through it to reach the substrate superalloys and hence provided an excellent oxidation resistance to the superalloys under study.

During investigations on the oxidation behaviour of Inconel 740, Zhao et al²⁸ have suggested that in the initial oxidation stage, some kinds of oxides, (Ni, Co) O and Cr₂O₃ etc. were formed on the surface of specimens and internal oxidation reaction of some minor elements took place, which led to a rapid mass gain. But during subsequent exposure formation of a continuous Cr₂O₃ film inhibited further oxidation of nickel, cobalt and other elements. (Ni, Co) O particles were surrounded with Cr₂O₃ and the solid-state reaction occurred to form (Ni, Co)Cr₂O₄ spinel in the scale gradually and dispersed in the external oxide layer. Formation of the spinel NiCr₂O₄ as indicated by XRD analysis for the superalloys under study might be indicative of the same phenomenon.

Further on the basis of cumulative weight gain at the end of 50 cycles of oxidation, it can be inferred that the uncoated superalloy 600 has shown best oxidation resistance whereas the NiCrAlY coated Superni 75 has shown the minimum resistance to the oxidation at an elevated temperature of 900°C. Moreover it is seen that the weight gains by the plasma spray NiCrAlY coated superalloys are greater than those by their uncoated counterparts under the given conditions of oxidation. The coatings have shown high weight gains in the initial cycles of the study, which might also be contributed by the porosity of the coatings to some extent. In the initial cycles more and more oxygen enters through the pores to oxidise the coating and contribute to the weight gain. As the study progresses the pores in the coating are filled by the oxides and the weight gain only becomes limited to that incurred by the surface scale. This situation is favourable for the development of oxidation resistance provided the growth of this oxide scale is becomes constant. In the present investigation the same phenomenon has been observed as the weight gains has shown tendency to become even with the progress of study.

From this it can be inferred that the coatings under study have provided necessary protection to the base superalloys.

Wahl and Nicoll²⁹ suggested that in the service environment, the coating forms an oxide surface layer, which ideally inhibits corrosion. Thus the coating is designed to serve as a reservoir for the element forming the surface oxide. The oxide required for the purpose of corrosion and oxidation resistance are Al_2O_3 , Cr_2O_3 and SiO_2 and coating compositions are selected accordingly to allow oxides to form in service. In the present investigation, XRD analysis has confirmed the formation of protective oxides such as NiO , Cr_2O_3 and $\alpha\text{-Al}_2\text{O}_3$ in case of NiCrAlY coated specimens. EDAX as well as EPMA analysis further support the formation of these phases. According to Toma et al⁹ the oxidation resistance of MCrAlY coating is based on the formation of $\alpha\text{-Al}_2\text{O}_3$ in the steady state of oxidation as $\alpha\text{-Al}_2\text{O}_3$ grows very slowly and is thermodynamically stable. In addition to the oxides of Ni, Cr and Al, presence of the spinel NiCr_2O_4 also develops oxidation resistance. Chatterjee et al³⁰ have explained that the better protection given by the spinel phases is due to the fact that the spinels of the mixed oxides of the general composition AB_2O_4 (A and B represent two metallic components) have much smaller diffusion coefficients of the cations and anions than those in their parent oxides. Besides, the coatings have been found to be successful in providing the base alloys with the reservoir of elements like Ni, Cr and Al, which form the protective oxide scales. This further reduces the depletion of these elements from the base alloy and hence increases the life of the substrate alloy.

Wang et al³¹ and Wang et al³² have also reported the formation of Cr_2O_3 and Al_2O_3 phases for the arc ion plated NiCrAlY coating, when oxidised at 900°C , whereas Toma et al⁹ have reported similar phases for oxidised VPS MCrAlY coatings and Choi et al¹⁹ for oxidised air plasma spray NiCrAlY coatings. Further MCrAlY coating has been reported to follow the parabolic rate law similar to the current study by Li et al² and Wang et al³¹. Moreover the minor deviations from the parabolic rate law as shown by the coated specimens are in agreement with the findings of Choi et al¹⁹ and Liu and Gao³³.

During the cyclic testing cracks were observed along the coatings near the edges only and some spalling of the coatings was observed. This may be attributed to the different values of thermal coefficients for the coating and the substrate as has been reported in the literature by the authors²², Evans & Taylor⁸, Wang et al³² and Niranatlumpong et al³⁴. Niranatlumpong et al³⁴ were of the opinion that spallation could be initiated by the rapid growth of void-like defects lying adjacent to coating protuberances, at which tensile radial stress developed during cooling as a result of the thermal contraction mismatch between the oxide and the coating is maximum. The formation of cracks in the coating originates from stresses developed in the deposit or at the coating-base metal interface³⁵. Through these cracks the corrosive environment can quickly reach the base metal and cut its way under the coating to result in adhesion loss and spalling, whereas some elements may diffuse outwards through these cracks to form their oxides or spinels. The greenish colour of the scale in case of NiCrAlY coating might be attributed to the presence of NiO as a dominating phase in the scale identical to the findings of Singh²⁰ and Bornstein et al³⁶.

5. CONCLUSIONS

1. The two Ni-base superalloys Superni 75 and Superni 600 have shown very good oxidation resistance when exposed to cyclic oxidation for 50 cycles at 900°C in air. A continuous chromia scale could be seen on the surface of the oxidised superalloy specimens, which is supposed to impart oxidation resistance to the base superalloys by reducing oxygen activity.

2. The plasma spray process provides the possibility of developing coatings of NiCrAlY powder on the Ni-based superalloys Superni 75 and Superni 600. The plasma sprayed NiCrAlY coatings when subjected to cyclic oxidation in air at 900°C, have shown fairly good adherence to the substrate superalloys and developed protective oxide scales to protect the substrate from oxidation. The main oxides formed were NiO, Cr₂O₃, Al₂O₃ alongwith a spinel NiCr₂O₄.
3. The weight gain by Superni 600 after 50 cycles of oxidation in air is negligible, while that by other superalloy Superni 75 is also not significant. After plasma spray NiCrAlY coating, the overall weight gain is slightly increased in case of Superni 75. Whereas the increase seems to be large in case of Superni 600. But the overall weight gain by the coating is not significant. The substrate has not been affected and it can be inferred that coating has provided required protection.
4. In all the cases of uncoated as well NiCrAlY coated Superni 75 and Superni 600 superalloys, oxidation behaviour could be approximated by parabolic rate law.

REFERENCES

1. Lai G Y, 'Oxidation-Chapter 3', *High Temperature Corrosion of Engineering Alloys*, Pub. ASM International (1990) 15.
2. Li M H, Zhang Z Y, Sun X F, Li J G, Yin F S, Hu W Y, Guan H R, and Hu Z Q, *Surf. Coat. Technol.* **165** (3) (2003) 241.
3. Eliaz N, Shemesh G, and Latanision R M, *Engineering Failure Analysis* **9** (2002) 31.
4. Liu P S, Liang K M, and Gu S R, *Corrosion Science* **43** (2001) 1217.
5. Stroosnijder M F, Mevrel R, and Bennett M J, *Mater. High Temp.* **12** (1) (1994) 53.
6. Gill B J, and Tucker Jr. R C, *Mater. Sci. Technol.* **2** (1986) 207.
7. Nicoll A R, Grruner H, Wuest G, and Keller S, *Mater. Sci. Technol.* **2** (1986) 214.
8. Evans H E, and Taylor M P, *Oxid. Met.* **55** (1-2) (2001) 17.
9. Toma D, Brandl W, and Koster U, *Surf. Coat Technol.* **120-121** (1999) 8.
10. Czech N, Schmitz F, and Stamm W, *Mater. Man. Proc.* **10** (1995) 1021.
11. Singheiser L, Grunling H W, and Schneider K, *Surf. Coat. Technol.* **42** (1990) 101.
12. Longa Y, and Takemoto M, *Oxid. Met.* **41** (1994) 301.
13. Kishitake T, Era H, Otsubo F, and Sonoda T, *J. Therm. Spray Tech.* **7** (1998) 64.
14. Tawancy H M, Abbas N M, and Bennett A, *Surf. Coat. Technol.* **68-69** (1994) 10.
15. Lelait L, and Alperine S, *J. Mater. Sci.* **27** (1992) 5.
16. Brandl W, Grabke H J, Toma D, and Kruger J, *Surf. Coat. Technol.* **86-87** (1996) 41.
17. Brandl W, Toma D, Kruger J, Grabke H J, and Matthauss G, *Surf. Coat. Technol.* **94-95** (1997) 21.
18. Brandl W, Toma D, and Grabke H J, *Surf. Coat. Technol.* **108-109** (1998) 10.
19. Choi H, Yoon B, Kim H, and Lee C, *Surf. Coat. Technol.* **150** (2002) 297.
20. Singh B, *Ph.D. Thesis*, Met. & Mat. Engg. Deptt., IITR, Roorkee, INDIA (2003).
21. Hussain N, Shahid K A, Khan I H, and Rahman S, *Oxid. Met.* **41** (3/4) (1994) 251.
22. Singh H, Puri D, and Prakash S, *Surf. Coat. Technol.* *in press*.
23. Levy M, Huie R, and Pettit F, *Corrosion* **45** (8) (1989) 661.
24. Greene G A, and Finfrock C C, *Oxid. Met.* **55** (5/6) (2001) 505.
25. Deb B, Iyer S R, and Radhakrishnan V M, *Mater. Letters* **29** (1996) 19.
26. Li, M H, Sun, X F, Li, J G, Zhang, Z Y, Jin, T, Guan, H R, and Hu, Z Q, *Oxid. Met.* **59** (5/6) (2003) 591.
27. Pettit F S, and Meier G H, *Superalloys*, Gell M, Kartovich C S, Bricknel R H, Kent W B, Radovich J F, (Eds.), The Met. Soc. of AIME, Warrendale, Pennsylvania, (1984) 651.

28. Zhao S, Xie X, and Smith G D, *Surf. Coat. Technol.* **185** (2004) 178.
29. Wahl G, and Nicoll A.R, ICMS, San Diego, *Thin Solid Films* **5** (1983) 35.
30. Chatterjee U K, Bose S K, and Roy S K, *Environmental Degradation of Metals*, Pub., Marcel Dekker, 270 Madison Avenue, New York (2001).
31. Wang B, Gong J, Sun C, Huang R F, and Wen L S, *Mater. Sc. Engg.* **A357** (2003) 39.
32. Wang B, Gong J, Wang A Y, Sun C, Huang R F, and Wen L S, *Surf. Coat. Technol.* **149** (2002) 70.
33. Liu Z, and Gao W, *Scripta Materialia* **38** (7) (1998) 1057.
34. Niranatlumpong P, Ponton C B, and Evans H E, *Oxid. Met.* **53** (3-4) (2000) 241.
35. Heath G R, Heimgartner P, Irons G, Miller R, and Gustafsson S, *Mater. Sci. Forum* **251-254** (1997) 809.
36. Bornstein N S, Decrescente M A and Roth H A, Proc. of Conf. on Gas Turbine Mater. in the Marine Environment, MMIC-75-27, Columbus, Ohio, USA, (1975), 115.

FIGURES

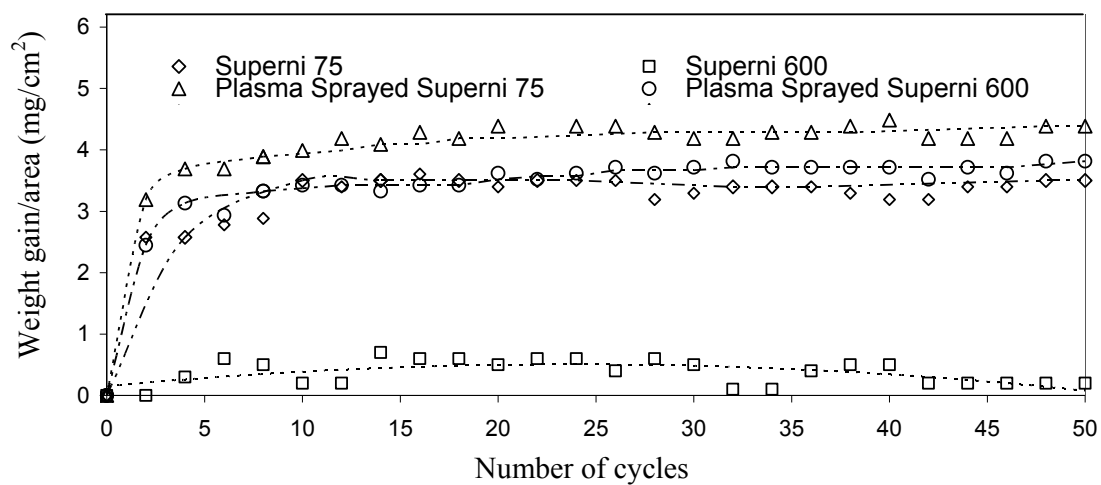


Fig. 1 Weight gain vs. number of cycles plot for the NiCrAlY coated and uncoated superalloys Superni 75 and Superni 601 subjected to cyclic oxidation for 50 cycles in air at 900°C.

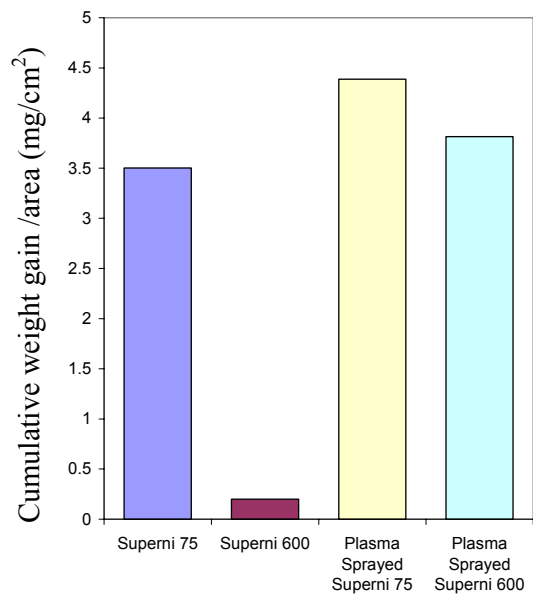


Fig.2. Bar chart showing cumulative weight gain per unit area for the NiCrAlY coated and uncoated superalloys Superni 75 and Superni 600 subjected to cyclic oxidation for 50 cycles in air at 900°C.

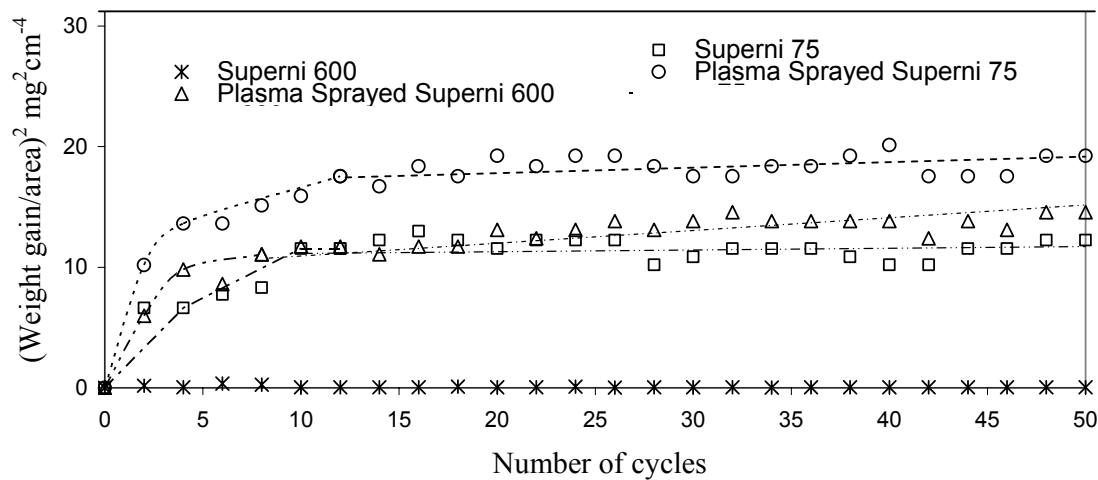


Fig.3. (weight gain/area)² vs. number of cycles plot for the NiCrAlY coated and uncoated superalloys Superni 75 and Superni 600 subjected to cyclic oxidation for 50 cycles in air at 900°C.

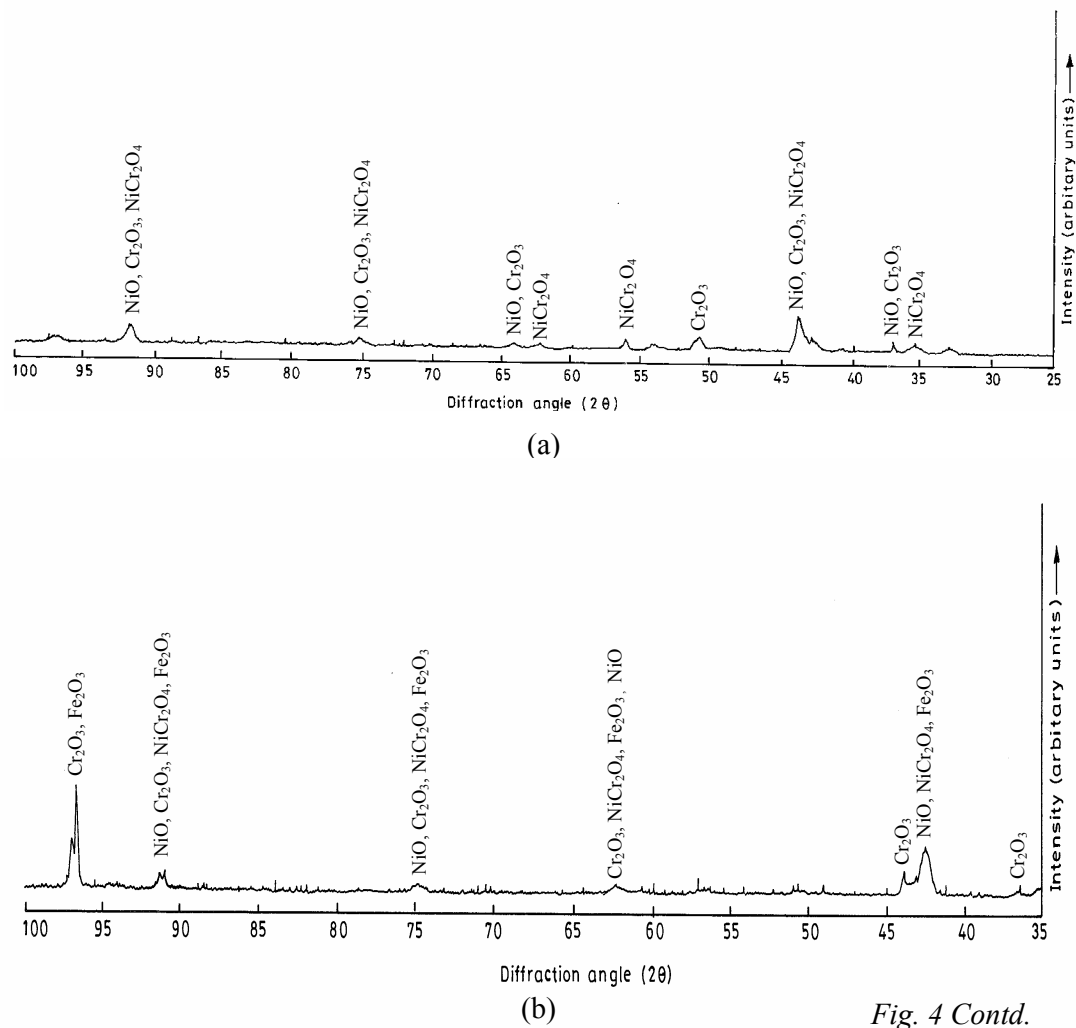


Fig. 4 Contd.

Fig. 4 X-ray diffraction patterns for the superalloys subjected to cyclic oxidation in air at 900°C after 50 cycles.

- (a) Superni 75
(b) Superni 600.

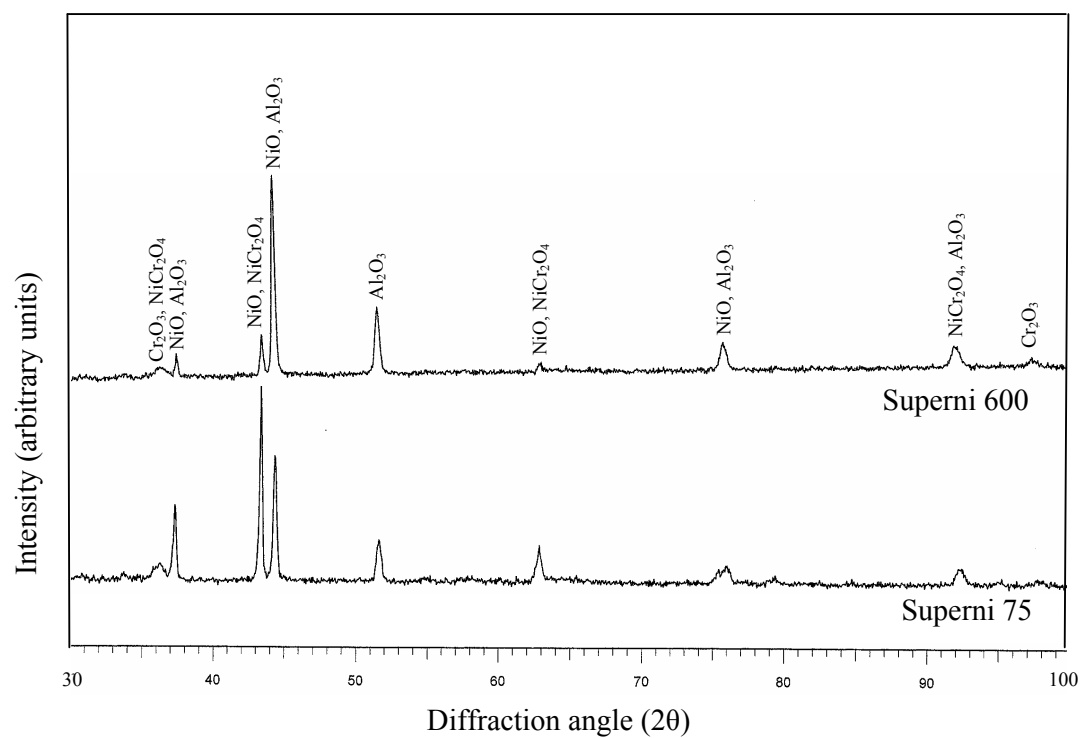


Fig. 5 X-ray diffraction patterns for the NiCrAlY coated superalloys Superni 75 and Superni 600 subjected to cyclic oxidation in air at 900°C after 50 cycles.

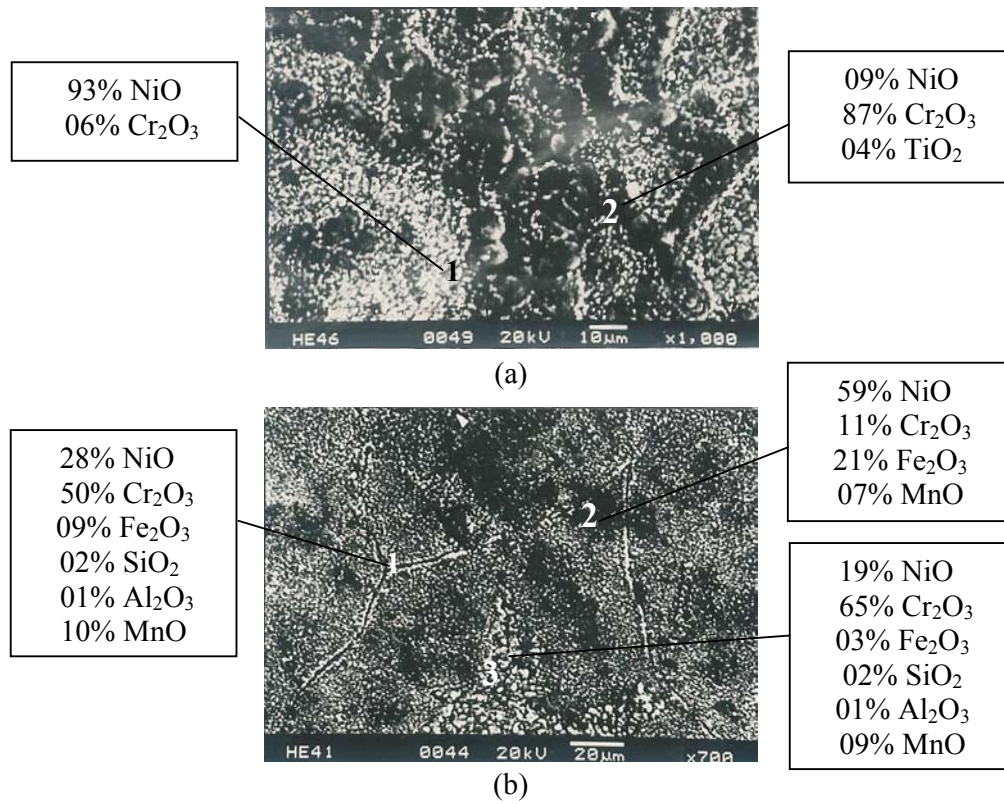


Fig. 6 Surface scale morphology and EDAX analysis for the uncoated superalloys subjected to cyclic oxidation in air at 900°C for 50 cycles (a) Superni 75 (b) Superni 600.

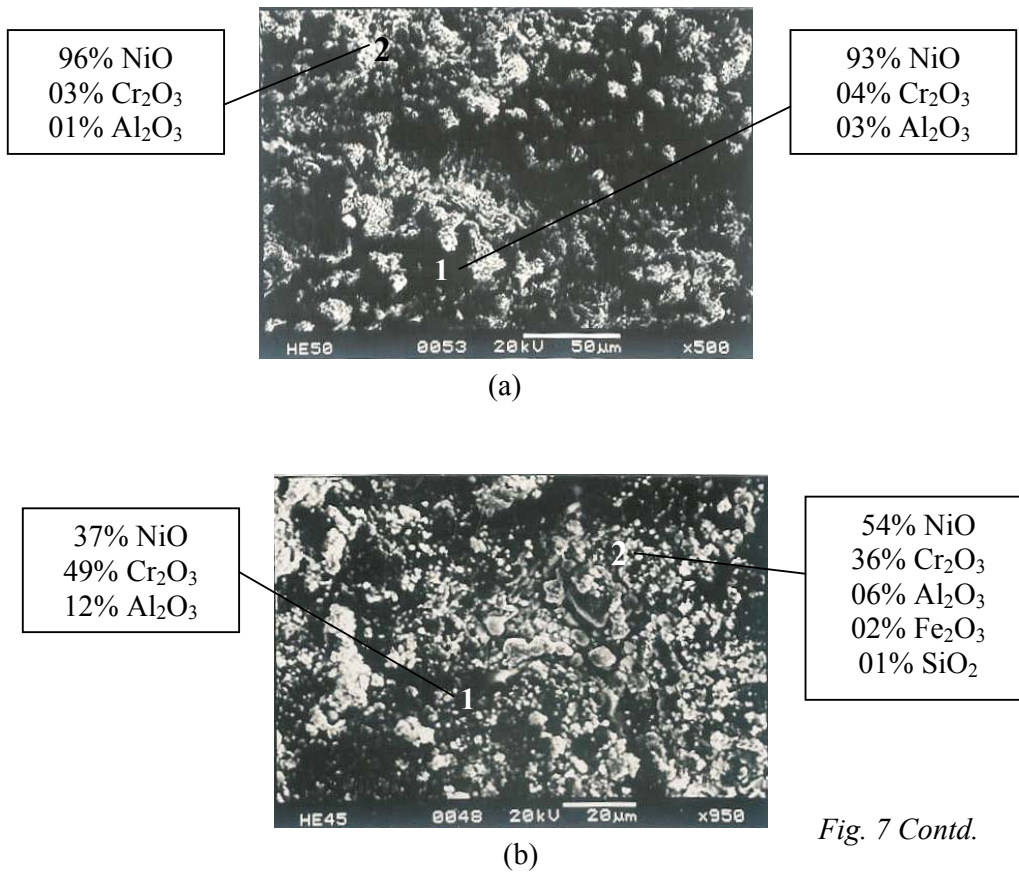


Fig. 7 Surface scale morphology and EDAX analysis for the plasma sprayed NiCrAlY coated superalloys subjected to cyclic oxidation in air at 900°C for 50 cycles (a) Superni 75 (b) Superni 600.

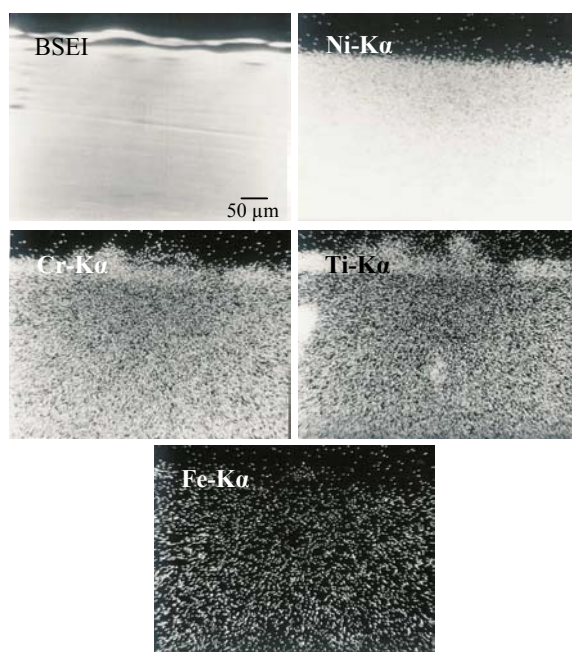


Fig. 8 Composition image (BSEI) and X-ray mappings of the cross-section of Superni 75 subjected to cyclic oxidation at 900°C in air after 50 cycles.

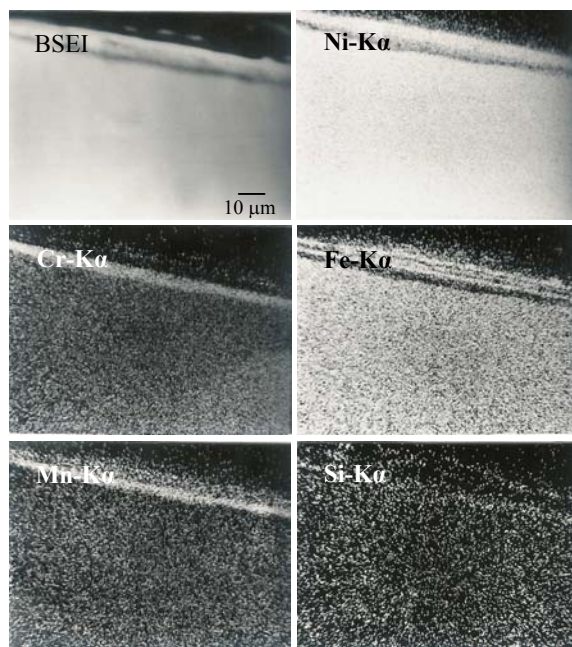


Fig. 9 Composition image (BSEI) and X-ray mappings of the cross-section of Superni 600 subjected to cyclic oxidation at 900°C in air after 50 cycles.

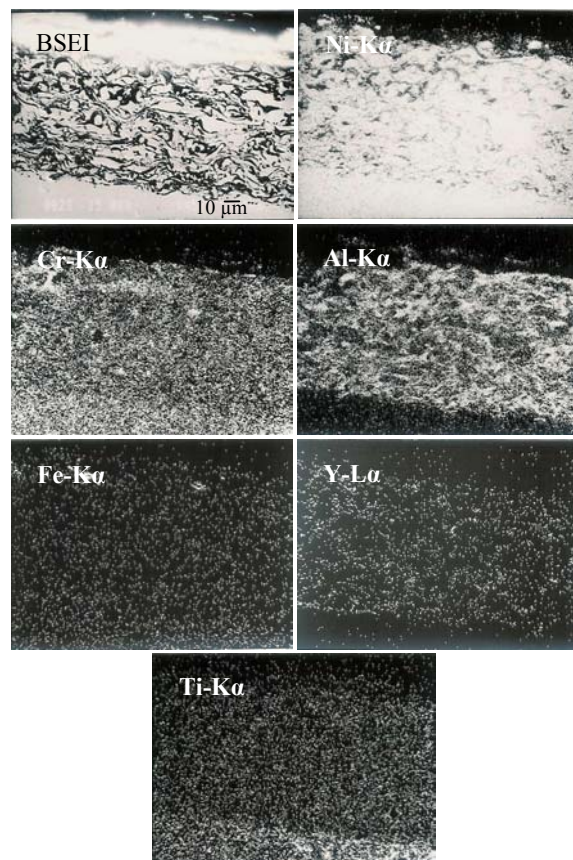


Fig. 10 Composition image (BSEI) and X-ray mappings of the cross-section of NiCrAlY coated superalloy Superni 75 subjected to cyclic oxidation at 900°C in air after 50 cycles.

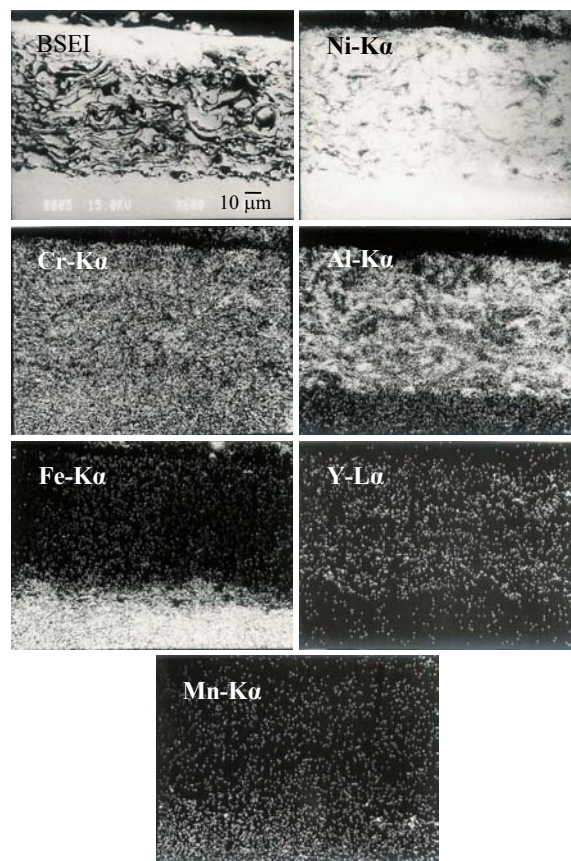


Fig. 11 Composition image (BSEI) and X-ray mappings of the cross-section of NiCrAlY coated superalloy Superni 600 subjected to cyclic oxidation at 900°C in air after 50 cycles.