

Appendix B

Oxidation of Heat-Resistant Alloys

Addendum to Chapter 4

Abdulaziz I. Al-Meshari
Hughes Hall, Cambridge

University of Cambridge
Department of Materials Science and Metallurgy

A supplementary material to the dissertation entitled
“Metal Dusting of Heat-Resistant Alloys”
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1 Introduction

This appendix must be read in conjunction with Chapter 4. It is intended to report extra and detailed results concerning the oxidation of the heat-resistant alloys HP, 35Cr-45Ni, and UCX at the test temperatures 650, 750, and 850°C for 100 and 1000h. The temperature profiling across the tube furnace as well as heating and cooling intervals are presented. Weight change measurements are also illustrated. Furthermore, extra surface images of the oxidised samples are enclosed in addition to EDX and XRD charts.

2 Results

2.1 Short-Term Experiments

2.1.1 Temperature Profiling

Temperature profiling, at 650 and 750°C, was conducted to locate the hot zone. A marked thermocouple was gradually inserted into the tube and the corresponding temperature was recorded. The profiling was obtained with and without the use of ceramic fibre sealing in order to understand the airflow effect on temperature.

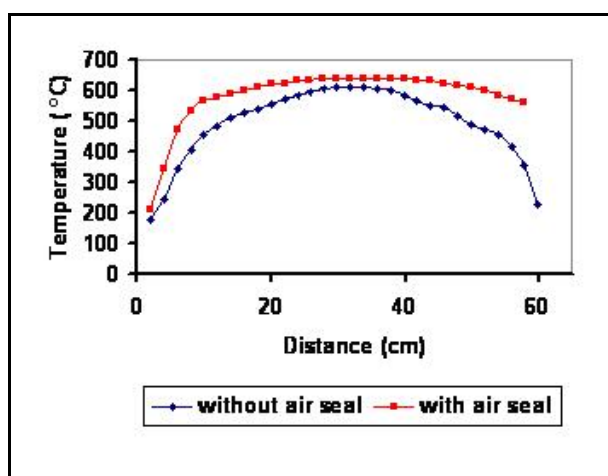


Figure 2.1 Temperature profile of the furnace at 650°C.

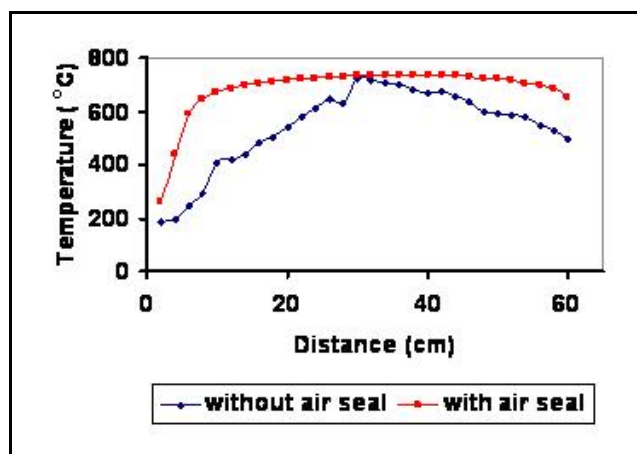


Figure 2.2 Temperature profile of the furnace at 750°C.

2.1.2 Furnace Heating and Cooling

The heating and cooling rates were recorded and plotted in Figures 2.3-2.6. The furnace heated up to 650, 750, and 850°C in 28, 30, and 38 minutes respectively. However, it took much longer time for it to cool down.

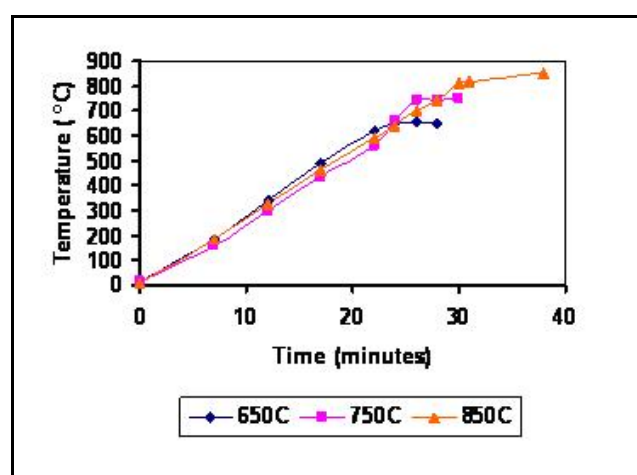


Figure 2.3 Heating to 650, 750, and 850°C.

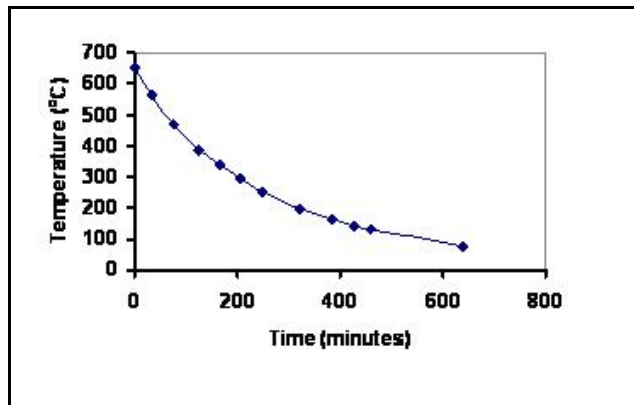


Figure 2.4 Cooling down from 650°C.

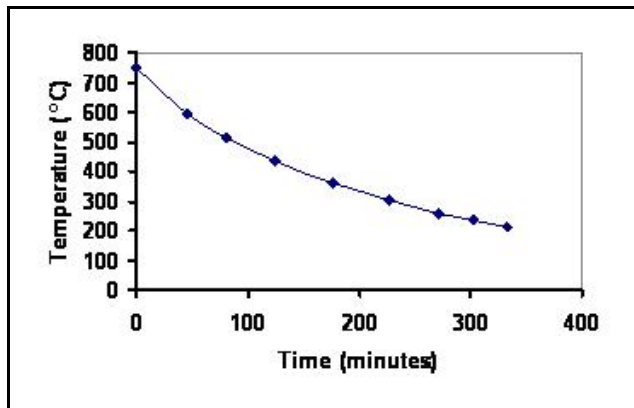


Figure 2.5 Cooling down from 750°C.

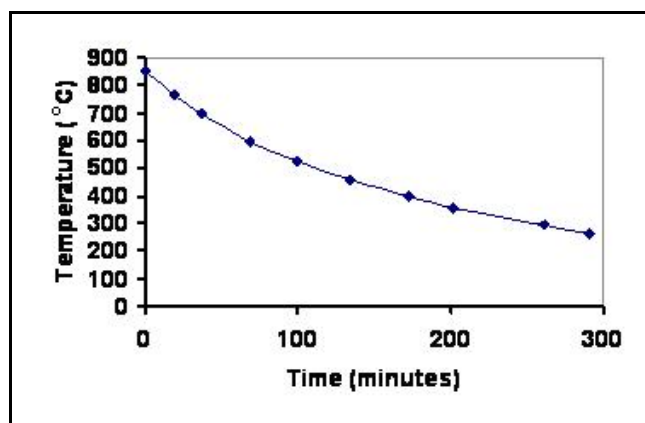


Figure 2.6 Cooling down from 850°C.

2.1.3 Weight Change Measurements

A four decimal, digital balance (Mettler AT261 DeltaRange) was used for weight measurements, Table 2.1. Each sample dimension was measured using digital callipers and a micrometer.

Table 2.1 Weight change measurements after the exposure to air at 650, 750, and 850°C for 100h. The sample actual surface areas were used in the calculations.

Alloy	T, °C	W _{before} , g	W _{after} , g	A, cm ²	ΔW/A, mg/cm ²
HP	650	15.6000	15.6000	12.0440	0.0000
35Cr-45Ni	650	15.5973	15.5976	11.9544	0.0251
UCX	650	15.5669	15.5681	11.9492	0.1004
HP	750	15.6171	15.6184	12.0756	0.1077
35Cr-45Ni	750	15.6219	15.6237	11.9474	0.1507
UCX	750	15.4645	15.4662	11.9213	0.1426
HP	850	15.7116	15.7033	12.5092	-0.6635
35Cr-45Ni	850	15.7300	15.7228	12.4713	-0.5773
UCX	850	14.9771	14.9722	12.1851	-0.4021

2.1.4 SEM/EDX and XRD Analyses

A Oxidation of Alloy HP at 650°C for 100h

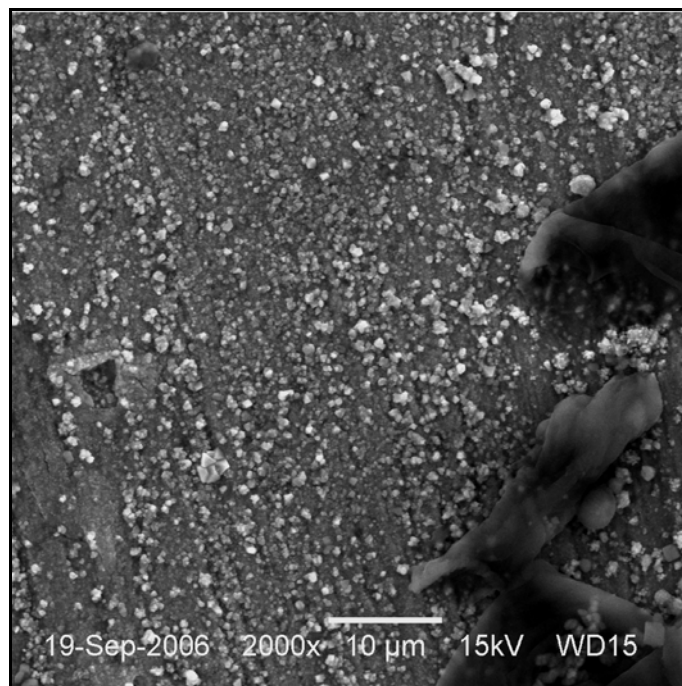
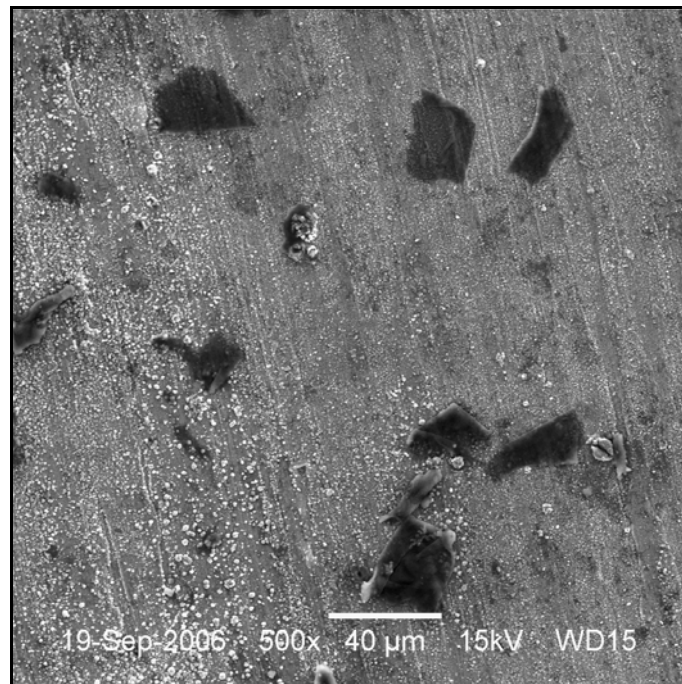


Figure 2.7 SEM/SE images of alloy HP surfaces exposed at 650°C.

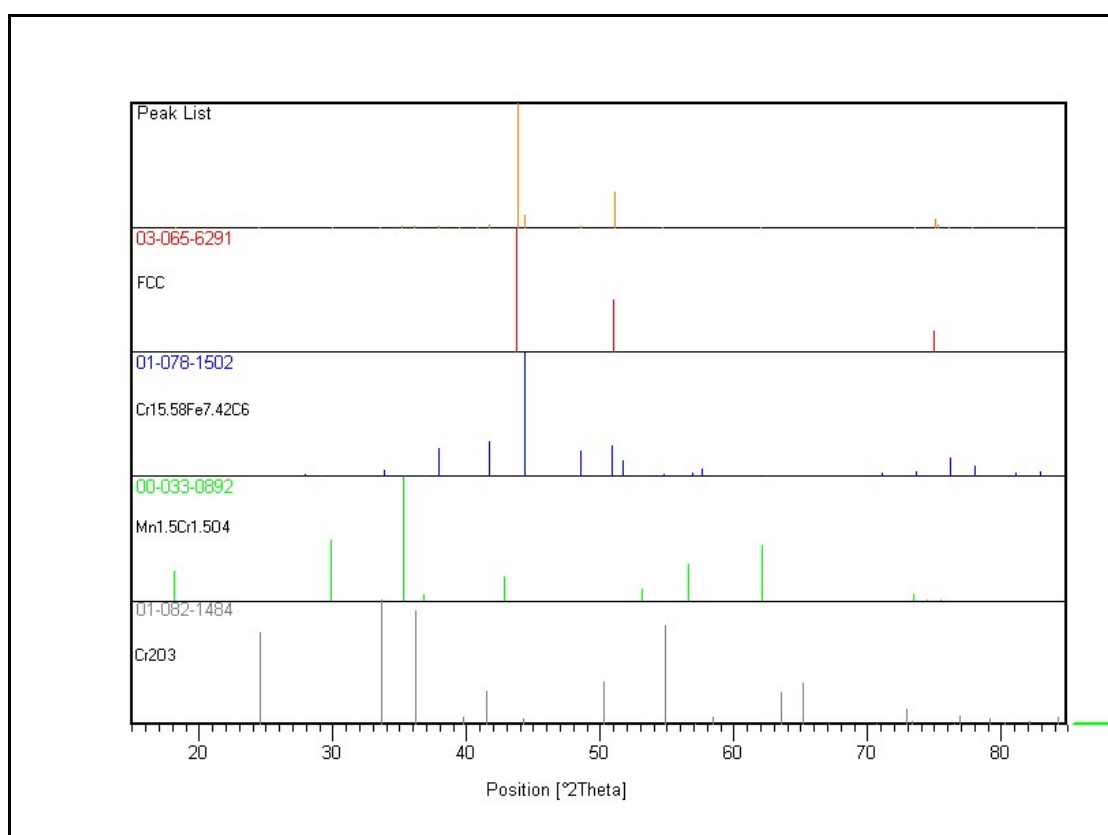
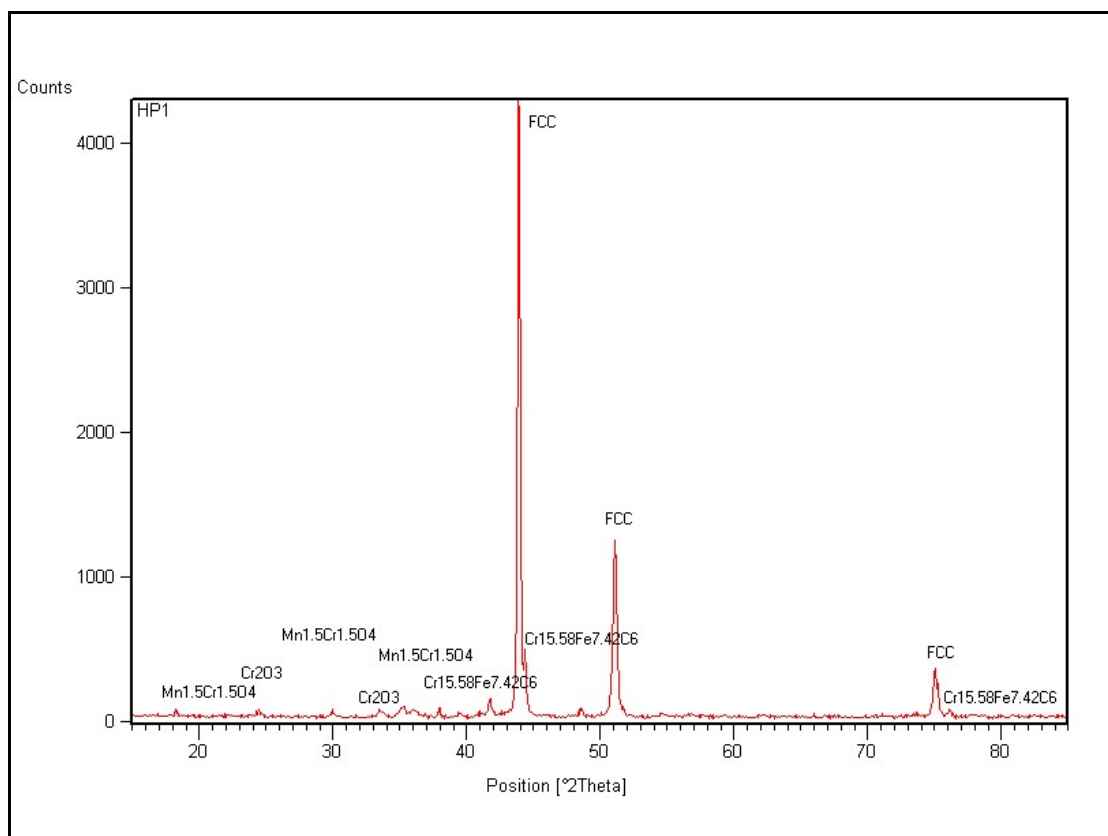
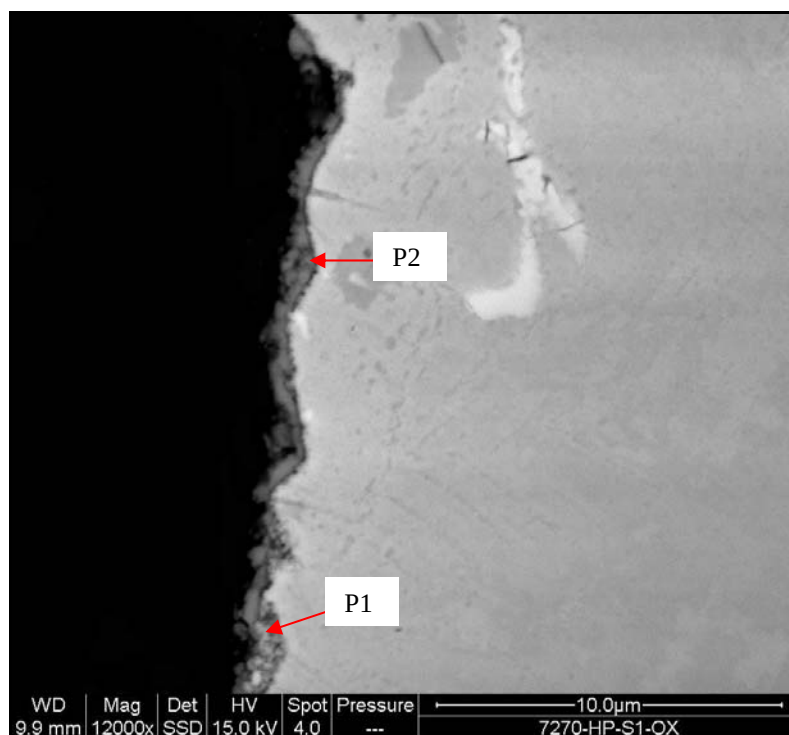
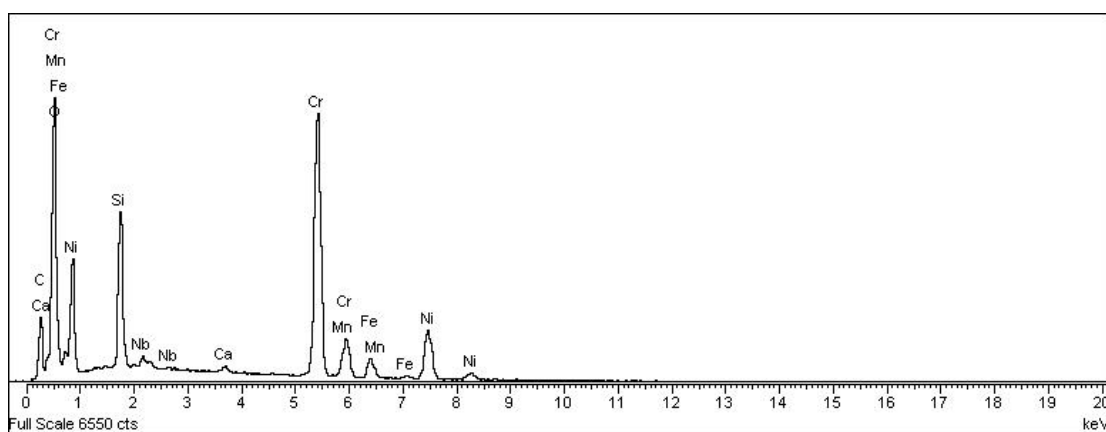


Figure 2.8 XRD pattern recorded from the alloy HP after exposure at 650°C.



P1



P2

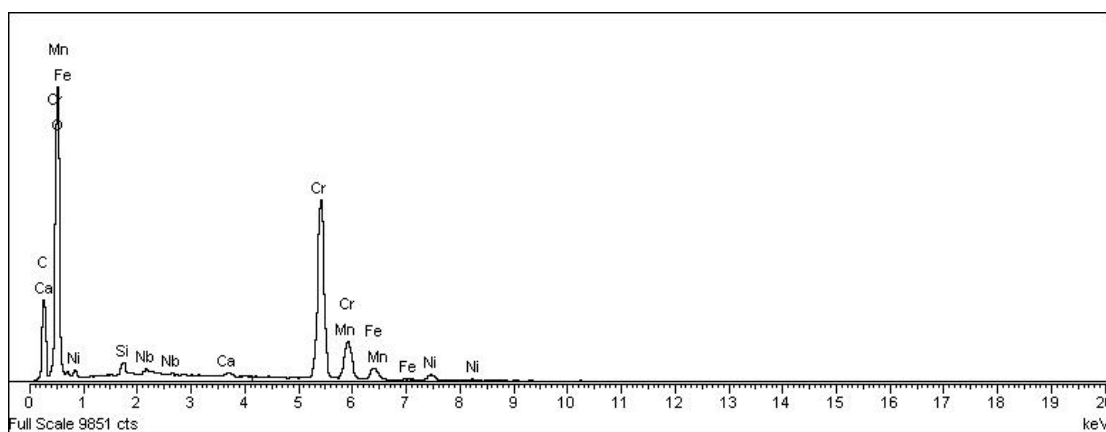


Figure 2.9 EDX analyses of the oxides formed on alloy HP after the 650°C experiment.

B Oxidation of Alloy HP at 750°C for 100h

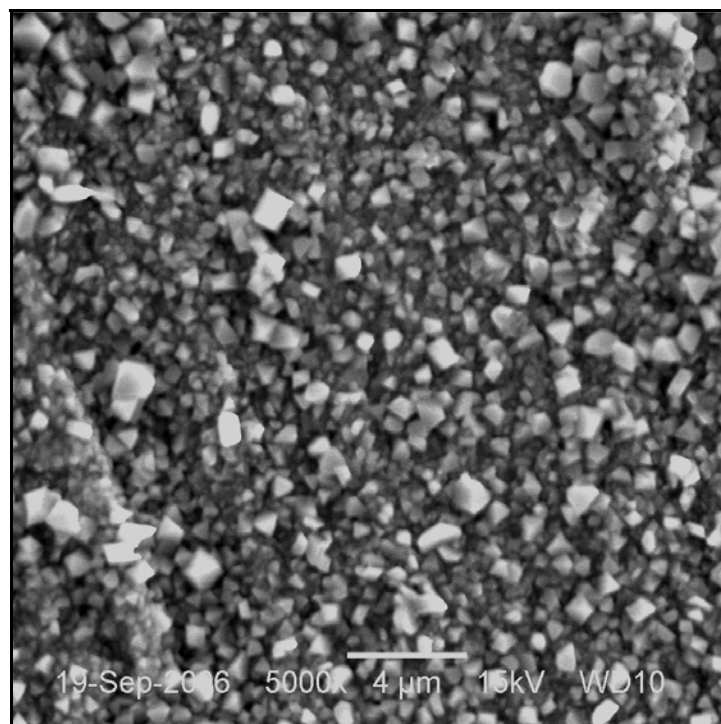
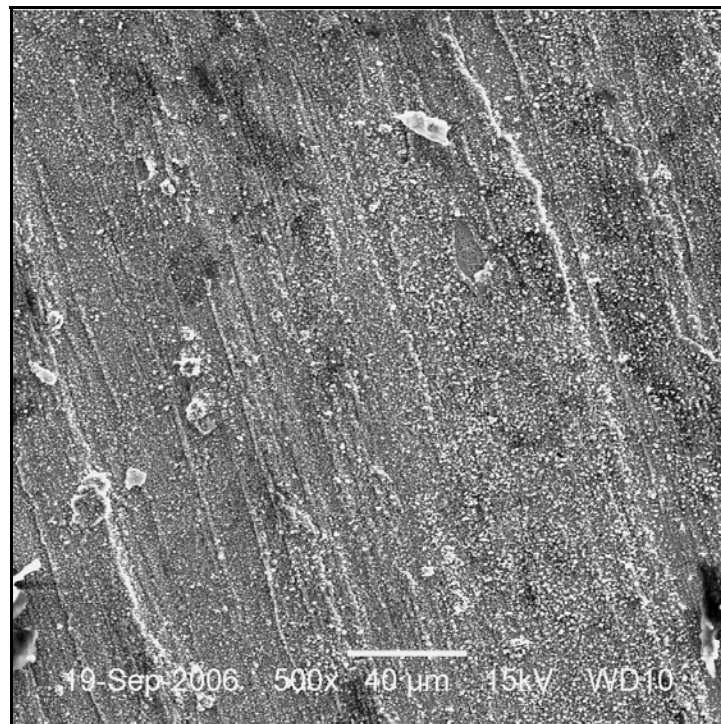


Figure 2.10 SEM/SE images of alloy HP surfaces exposed at 750°C.

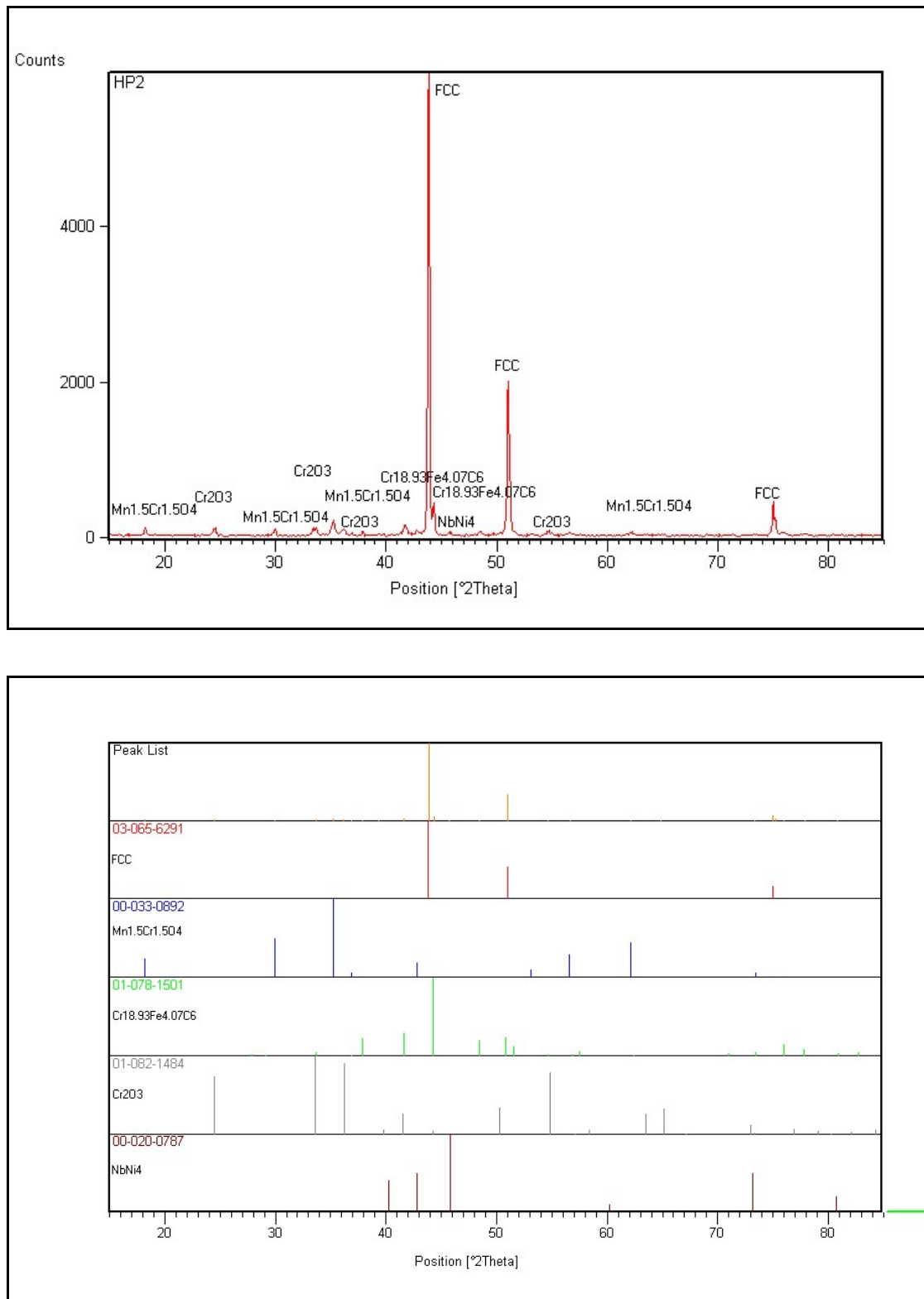
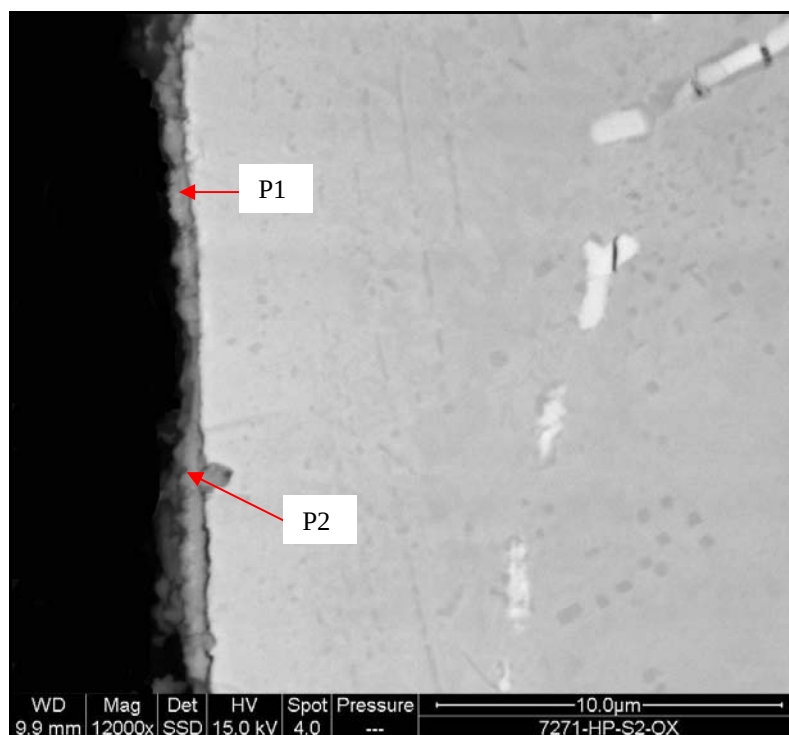
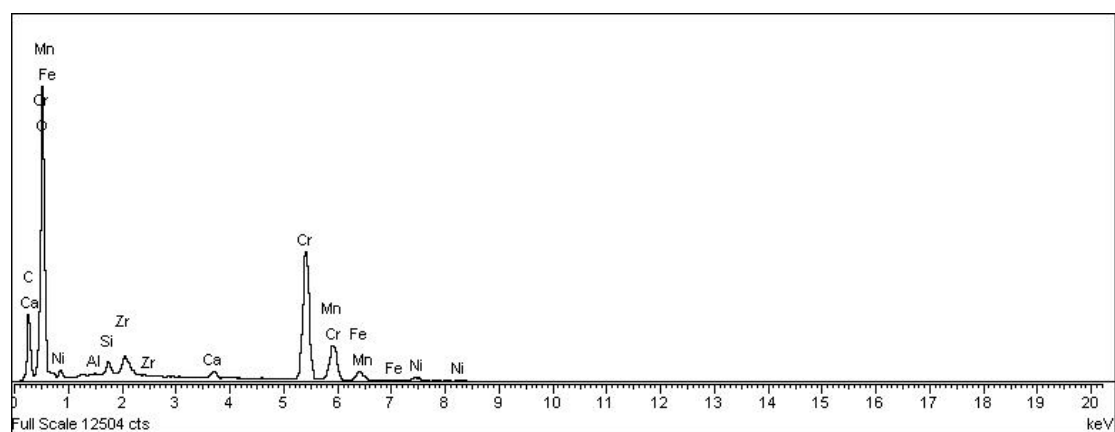


Figure 2.11 XRD pattern recorded from the alloy HP after exposure at 750°C.



P1



P2

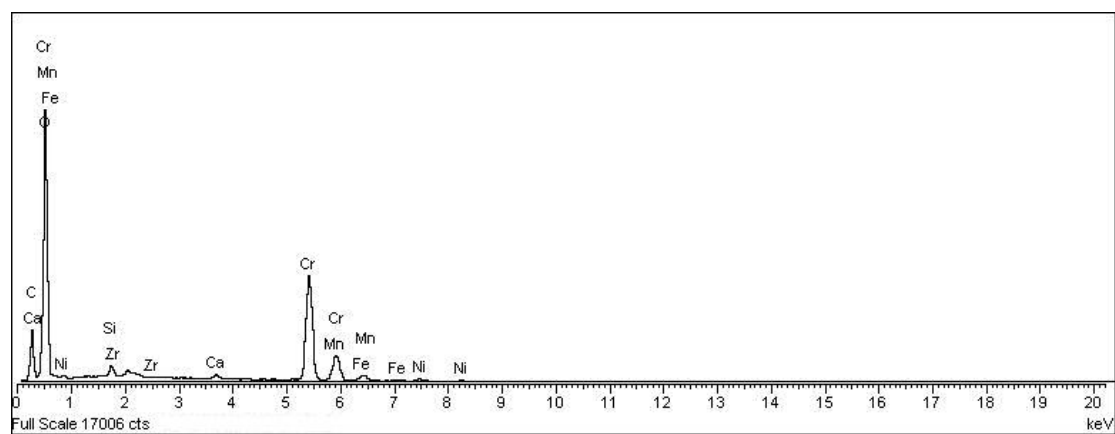


Figure 2.12 EDX analyses of the oxides formed on alloy HP after the 750°C experiment.

C *Oxidation of Alloy HP at 850°C for 100h*

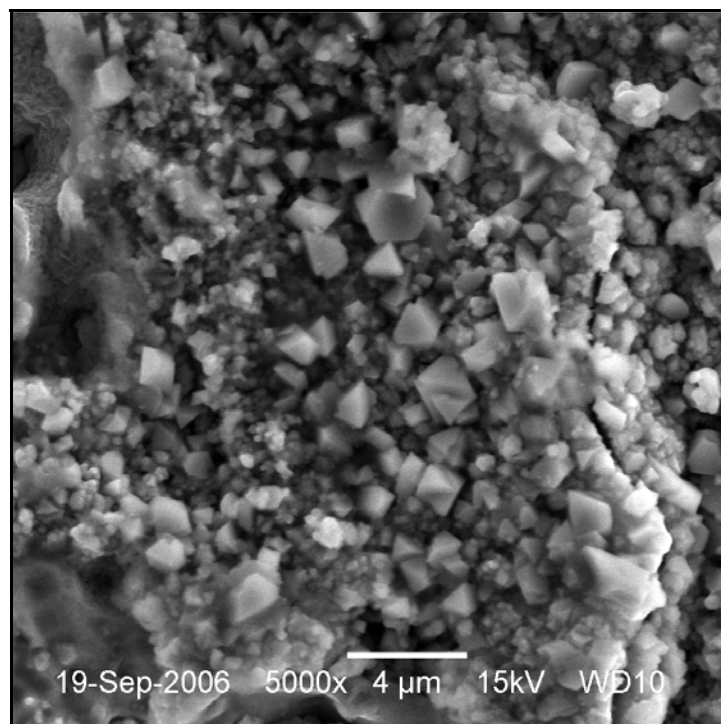
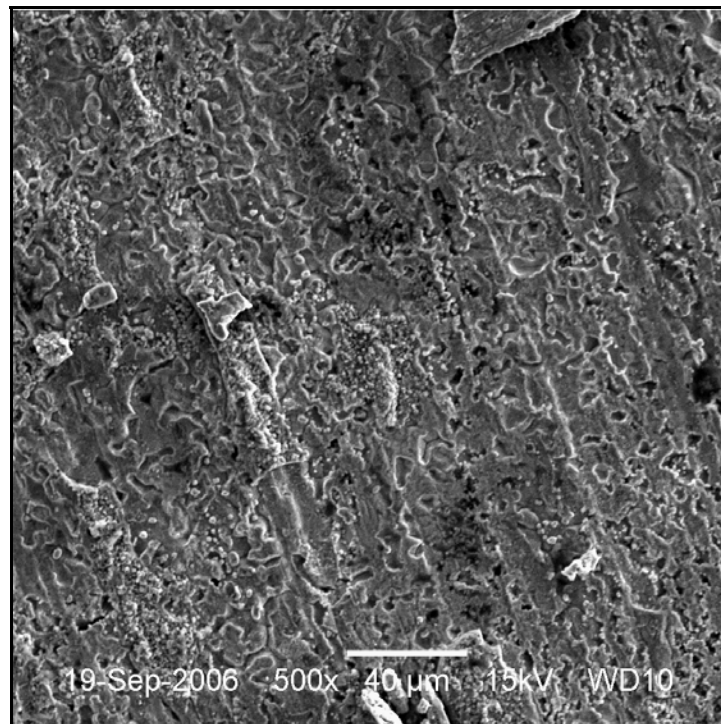


Figure 2.13 SEM/SE images of alloy HP surfaces exposed at 850°C.

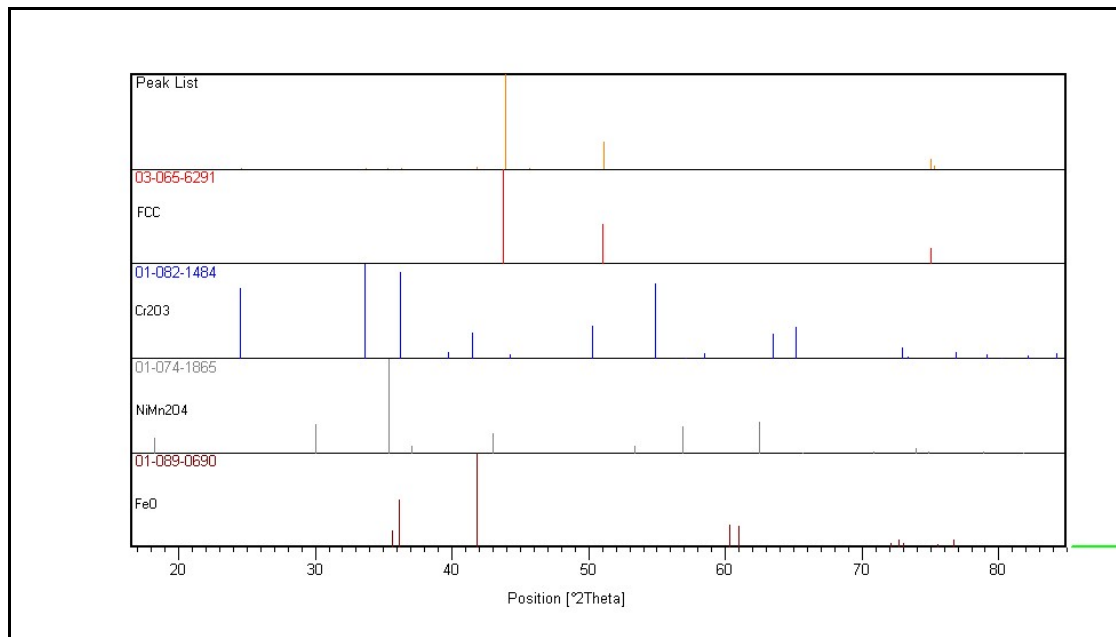
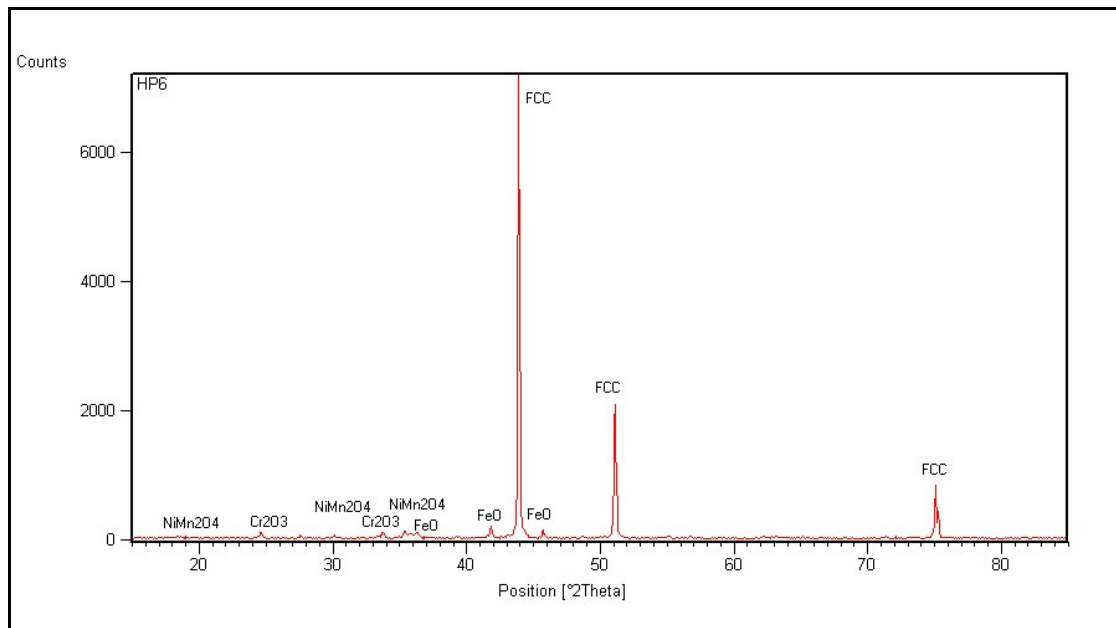
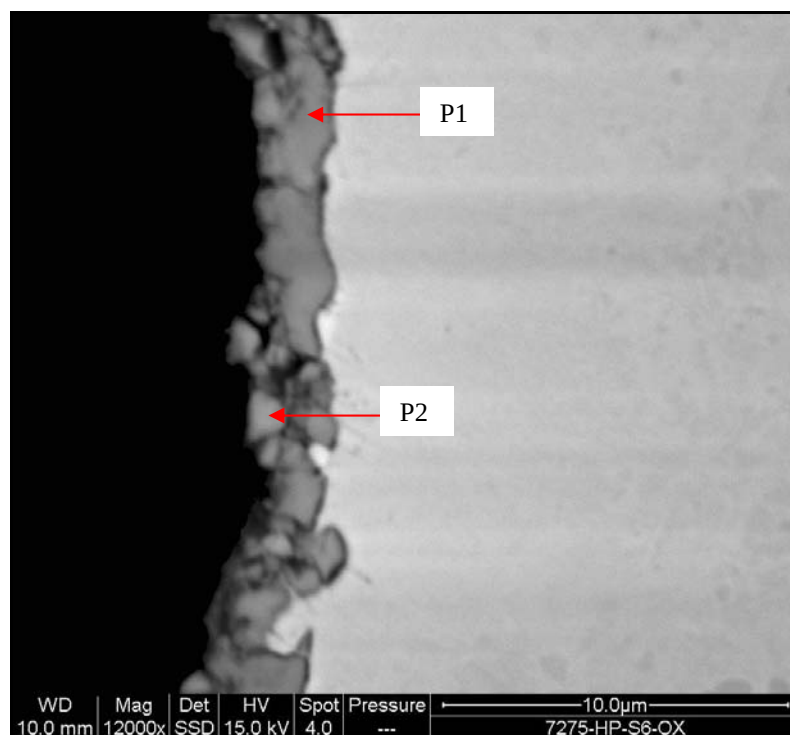
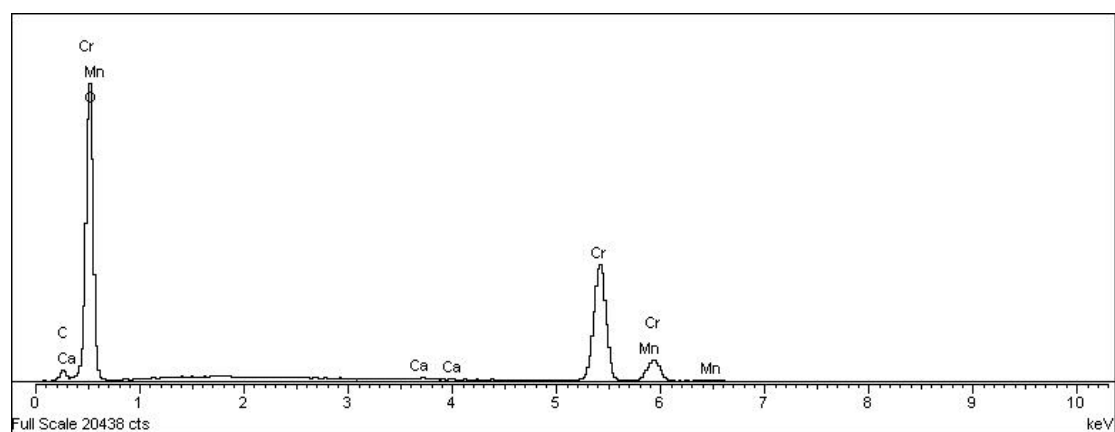


Figure 2.14 XRD pattern recorded from the alloy HP after exposure at 850°C.



P1



P2

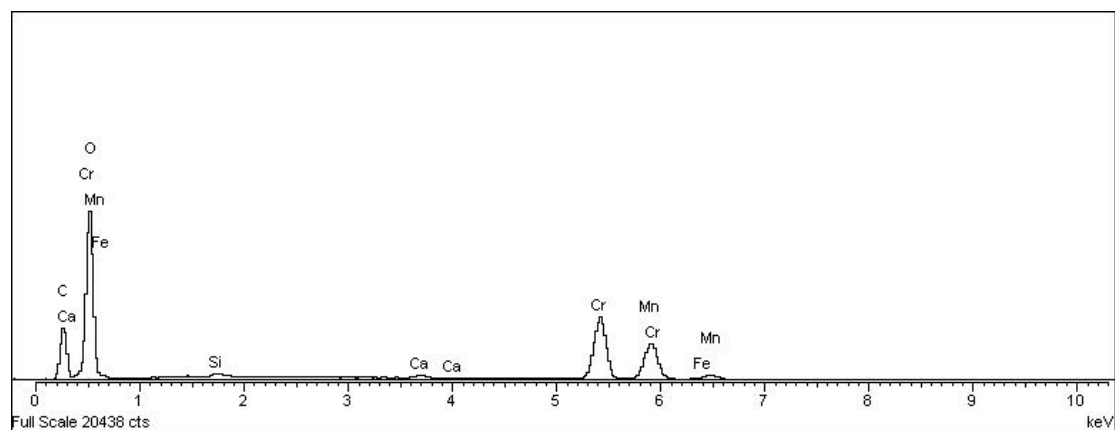


Figure 2.15 EDX analyses of the oxides formed on alloy HP after the 850°C experiment.

D Oxidation of Alloy 35Cr-45Ni at 650°C for 100h

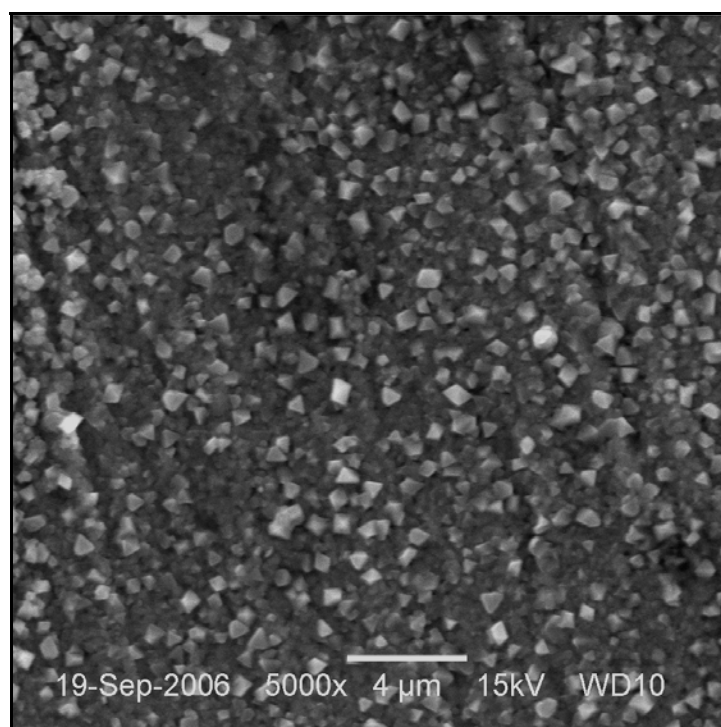
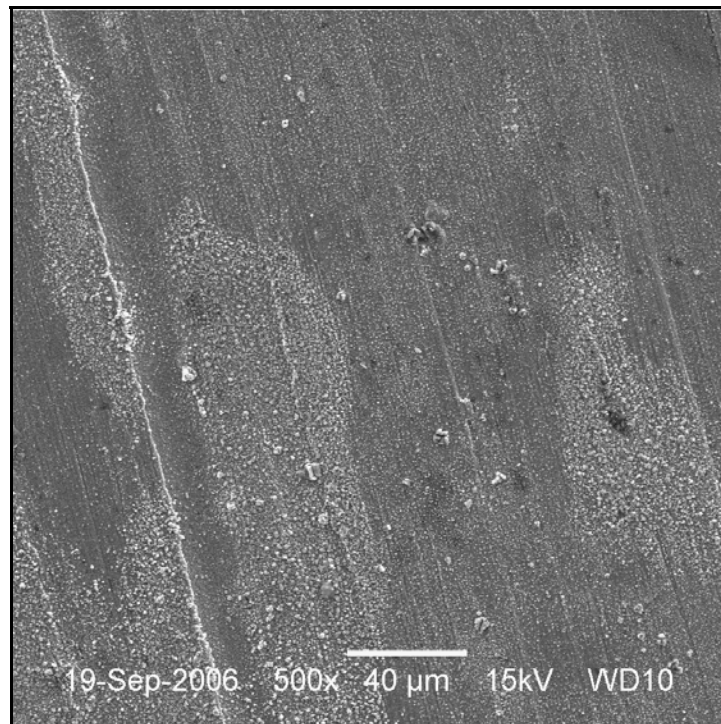


Figure 2.16 SEM/SE images of alloy 35Cr-45Ni surfaces exposed at 650°C.

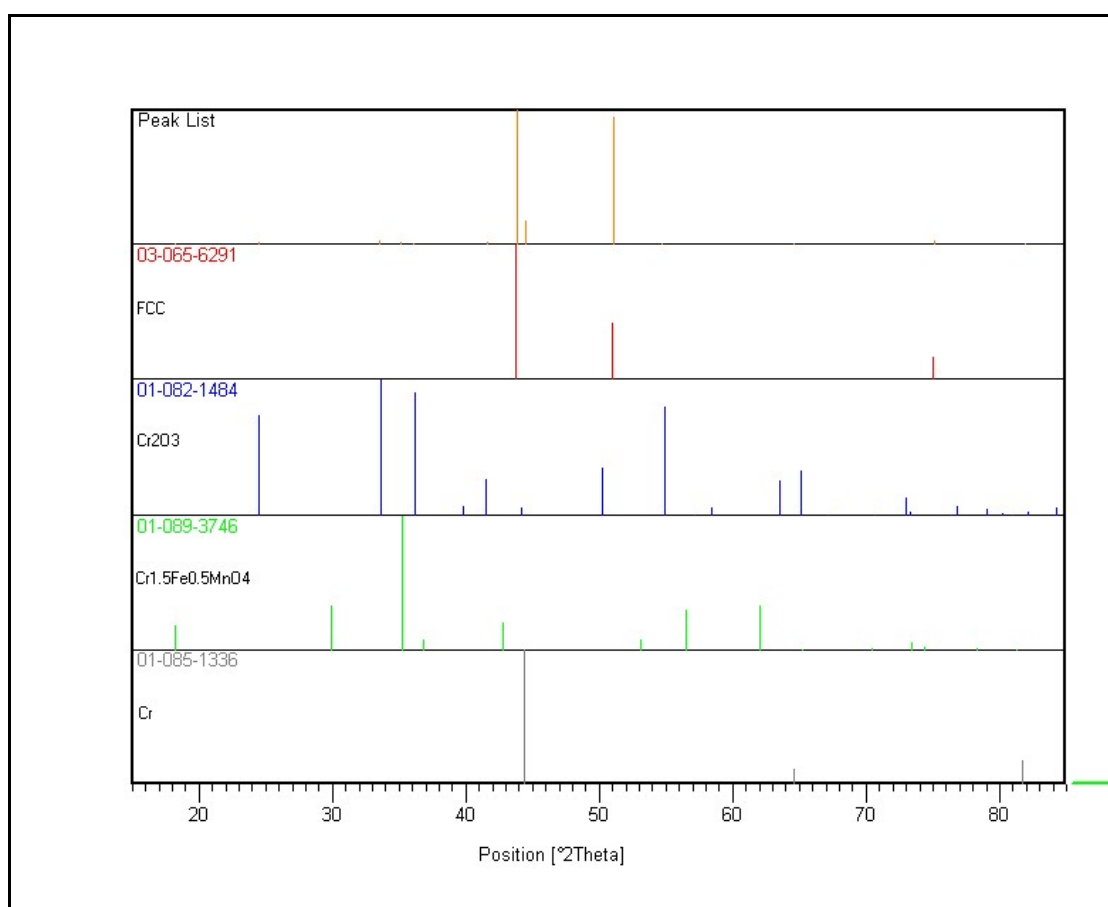
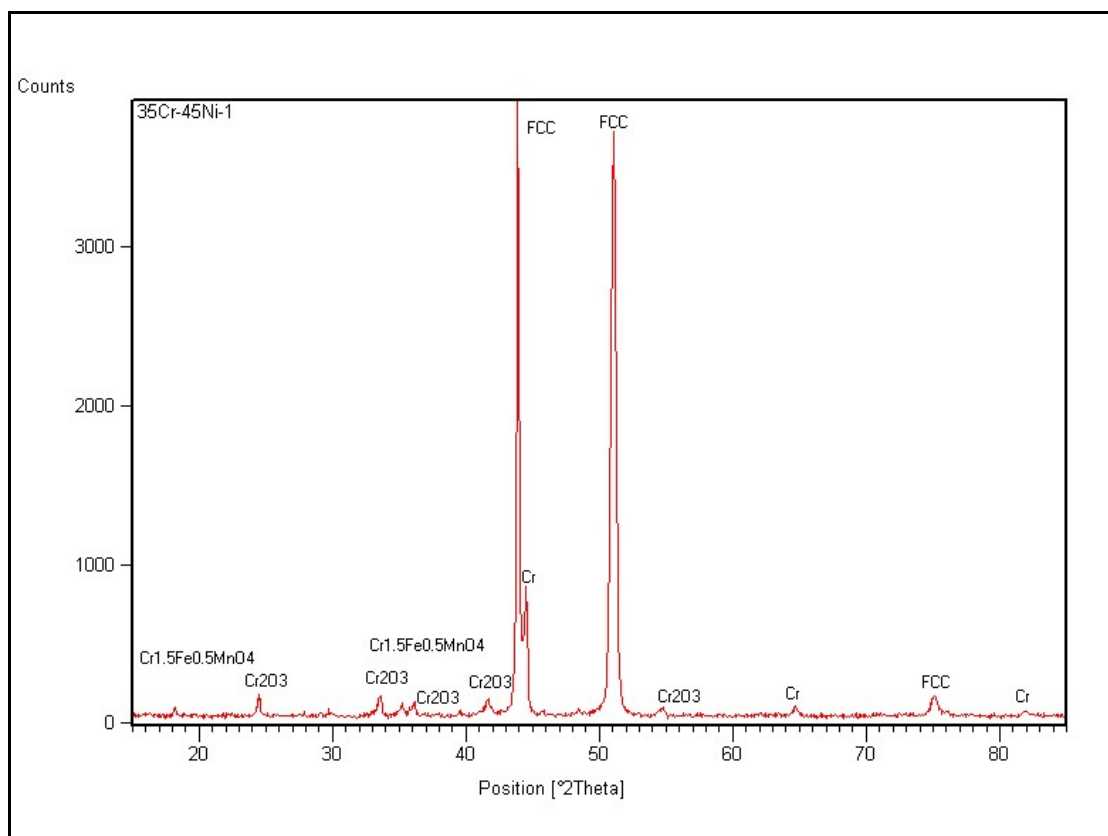
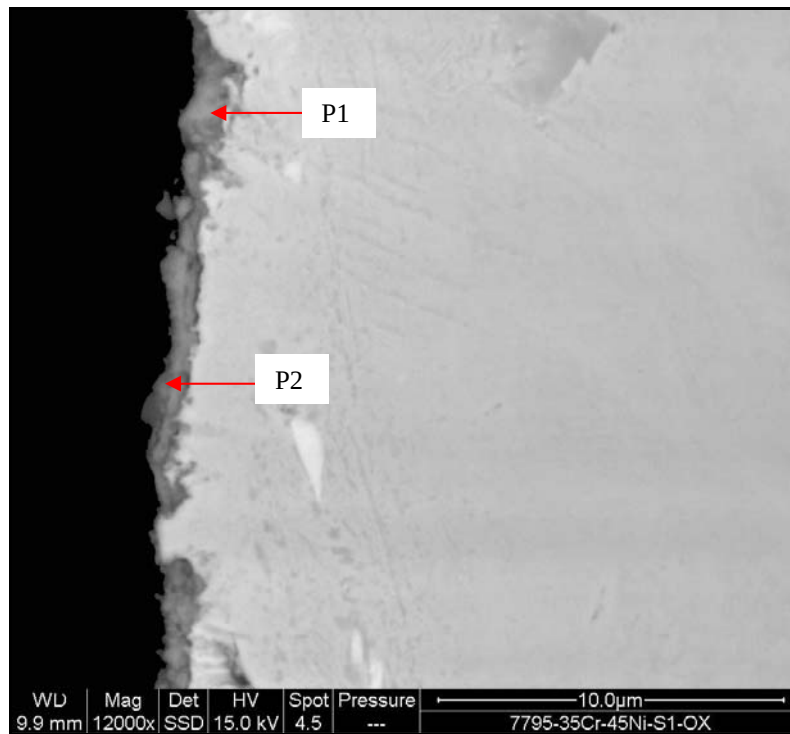
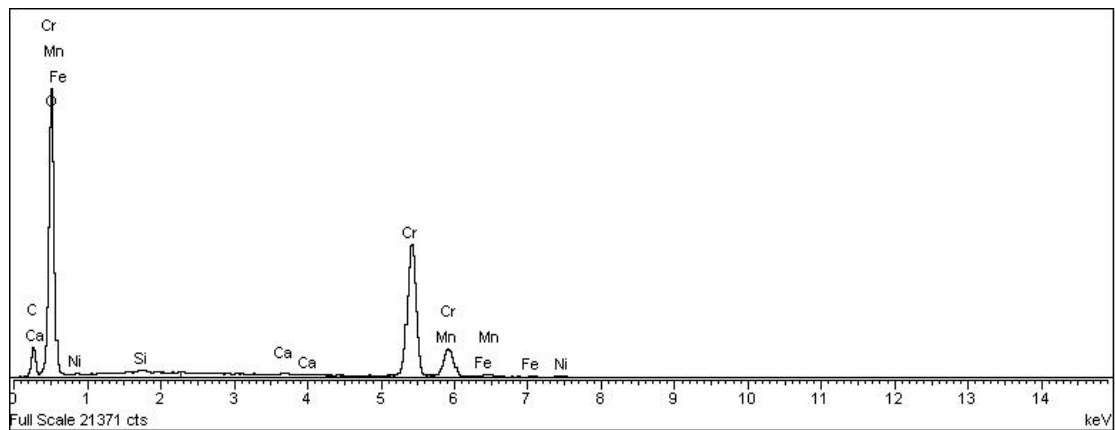


Figure 2.17 XRD pattern recorded from the alloy 35Cr-45Ni after exposure at 650°C.



P1



P2

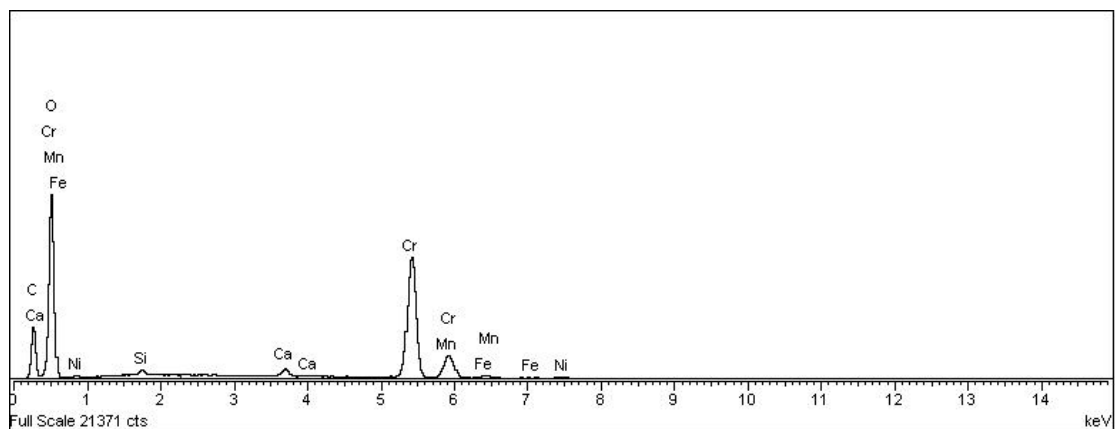


Figure 2.18 EDX analyses of the oxides formed on alloy 35Cr-45Ni after the 650°C experiment.

E Oxidation of Alloy 35Cr-45Ni at 750°C for 100h

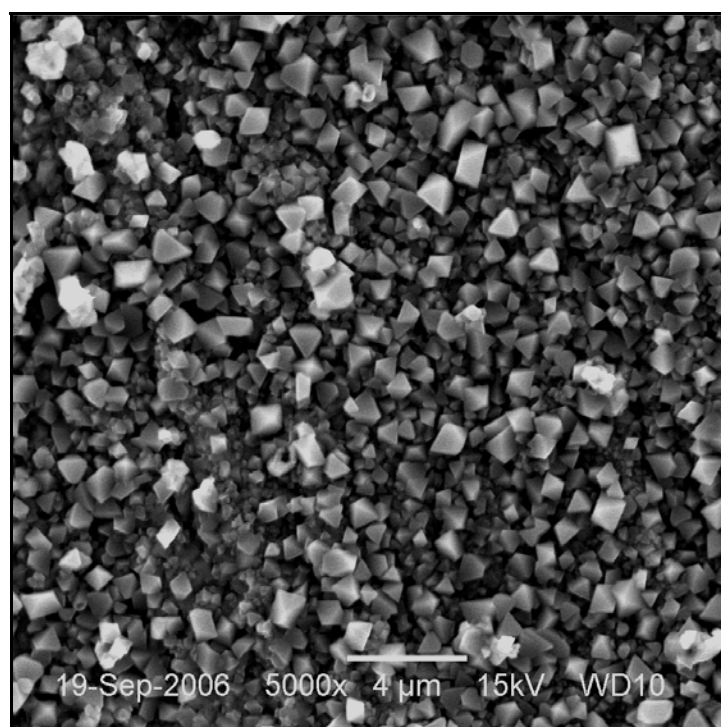
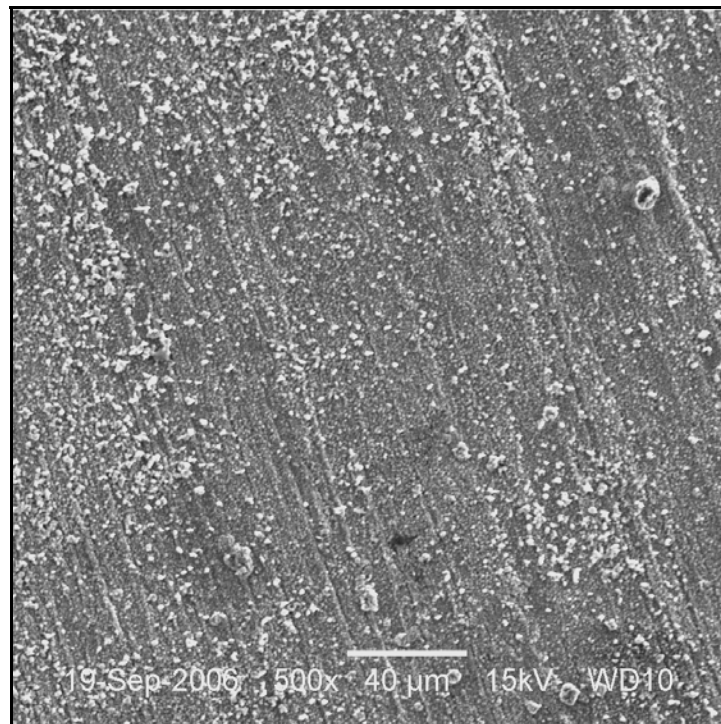


Figure 2.19 SEM/SE images of alloy 35Cr-45Ni surfaces exposed at 750°C.

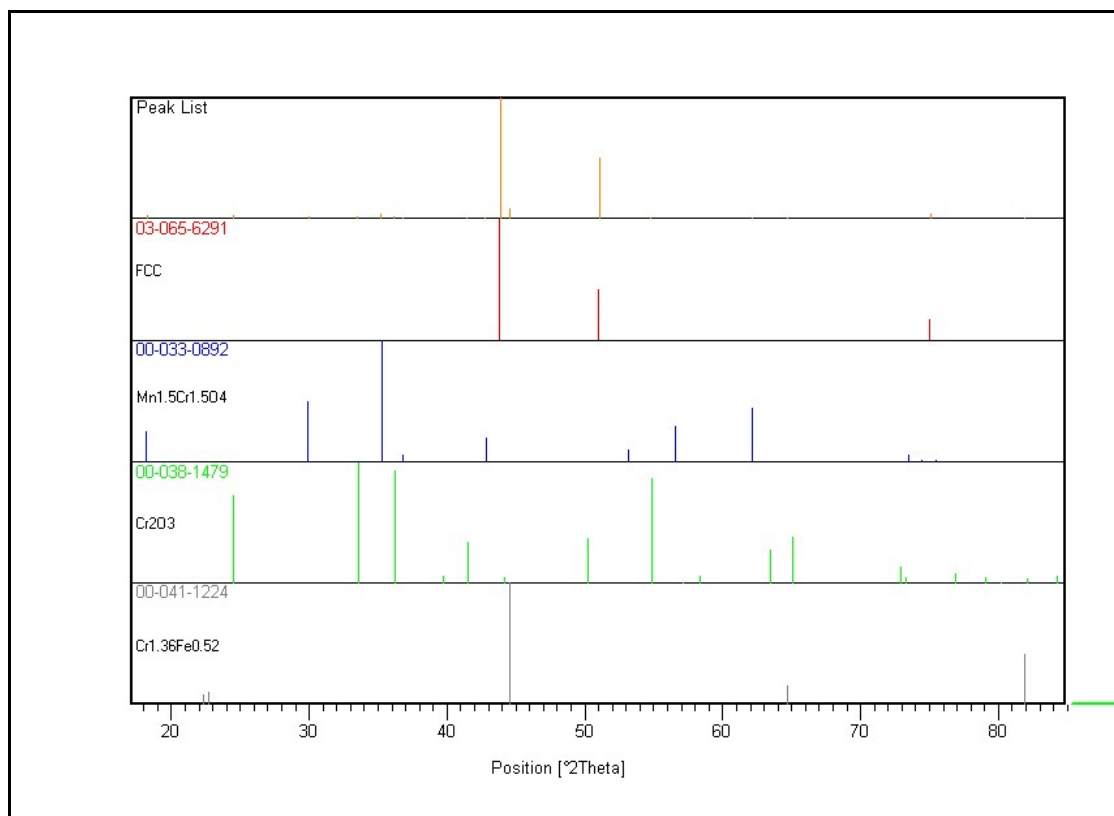
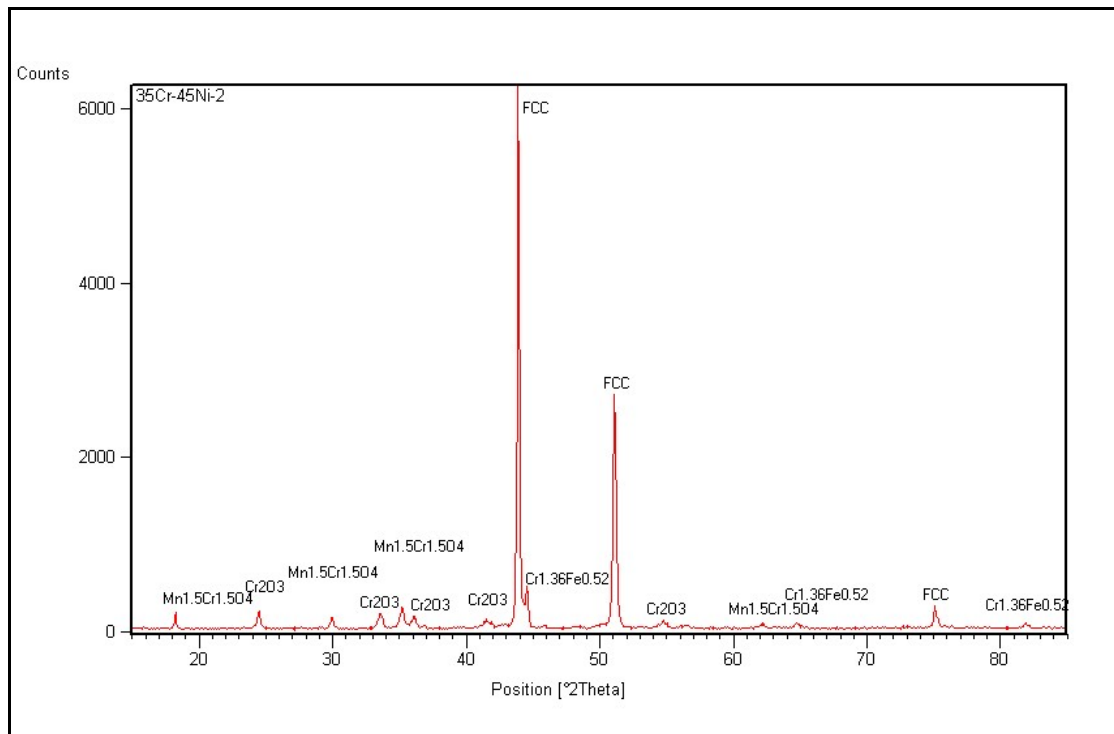
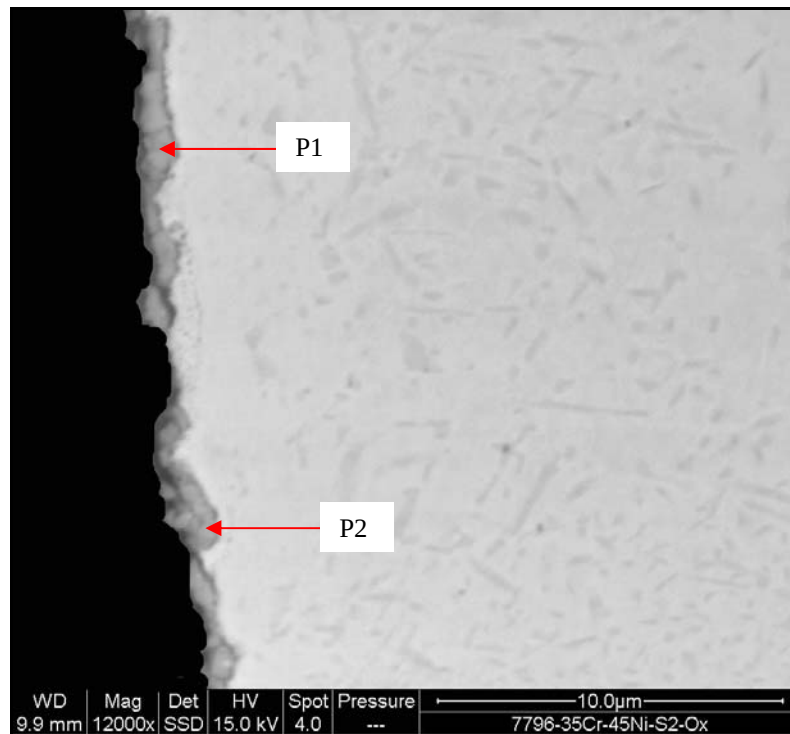
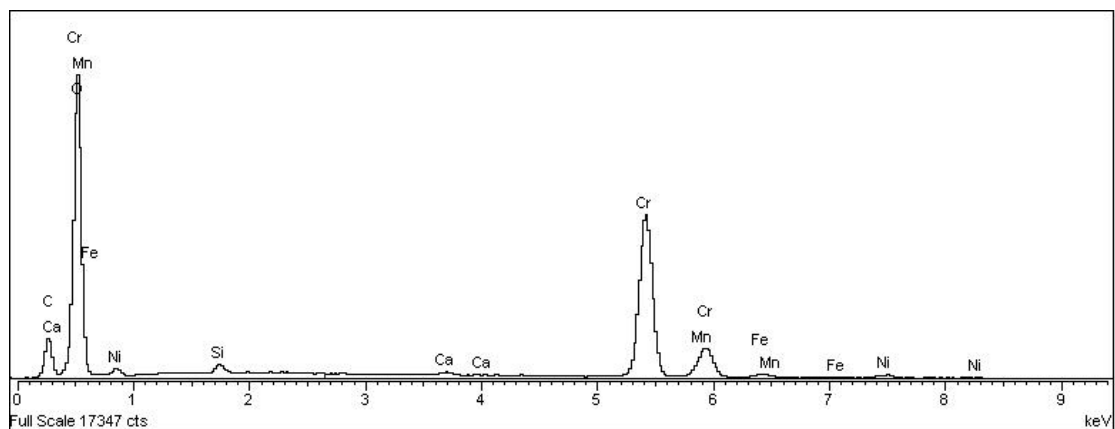


Figure 2.20 XRD pattern recorded from the alloy 35Cr-45Ni after exposure at 750°C.



P1



P2

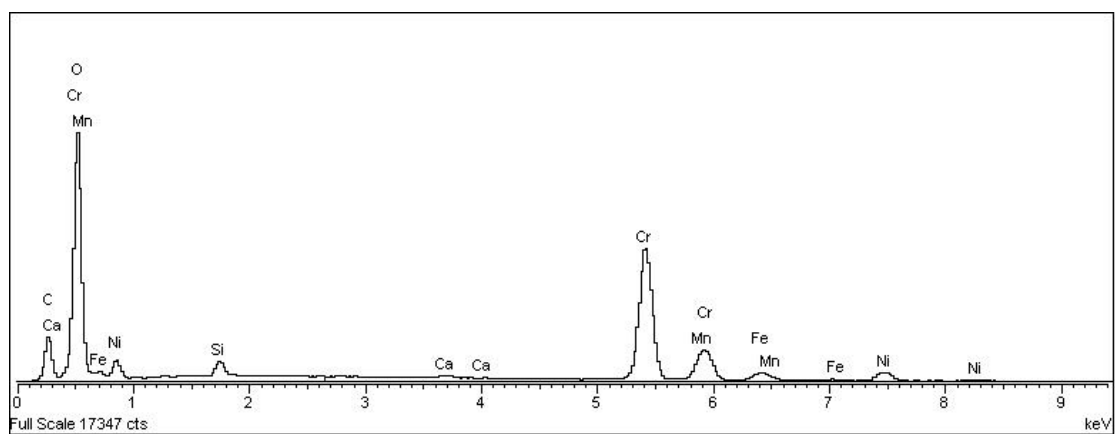


Figure 2.21 EDX analyses of the oxides formed on alloy 35Cr-45Ni after the 750°C experiment.

F Oxidation of Alloy 35Cr-45Ni at 850°C for 100h

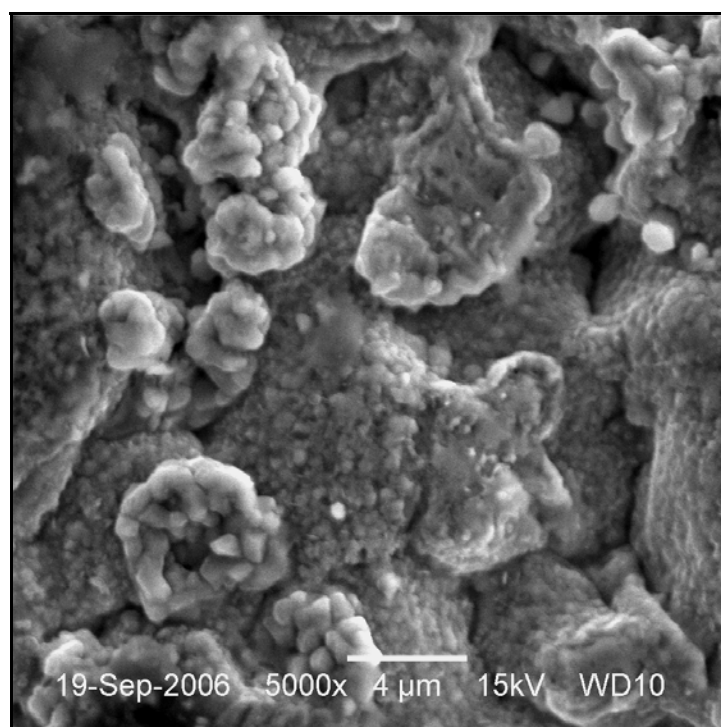
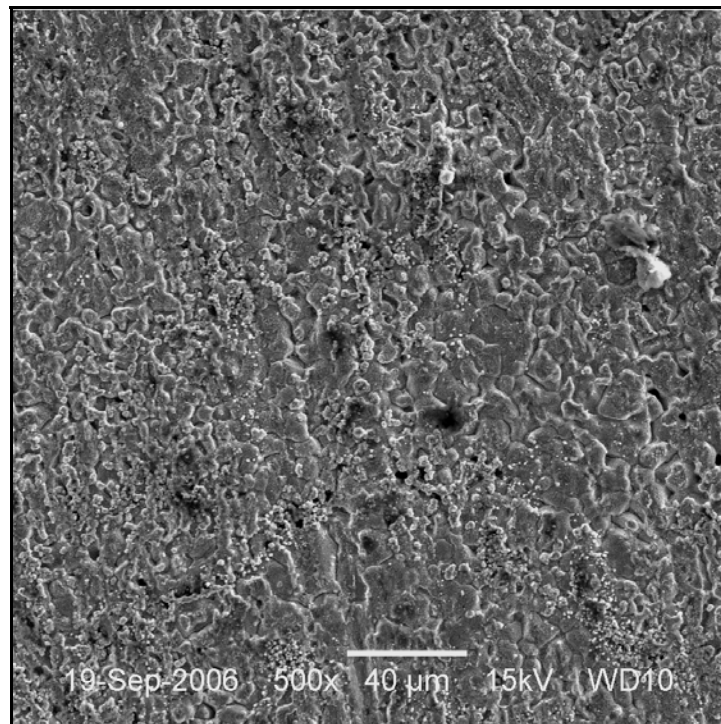


Figure 2.22 SEM/SE images of alloy 35Cr-45Ni surfaces exposed at 850°C.

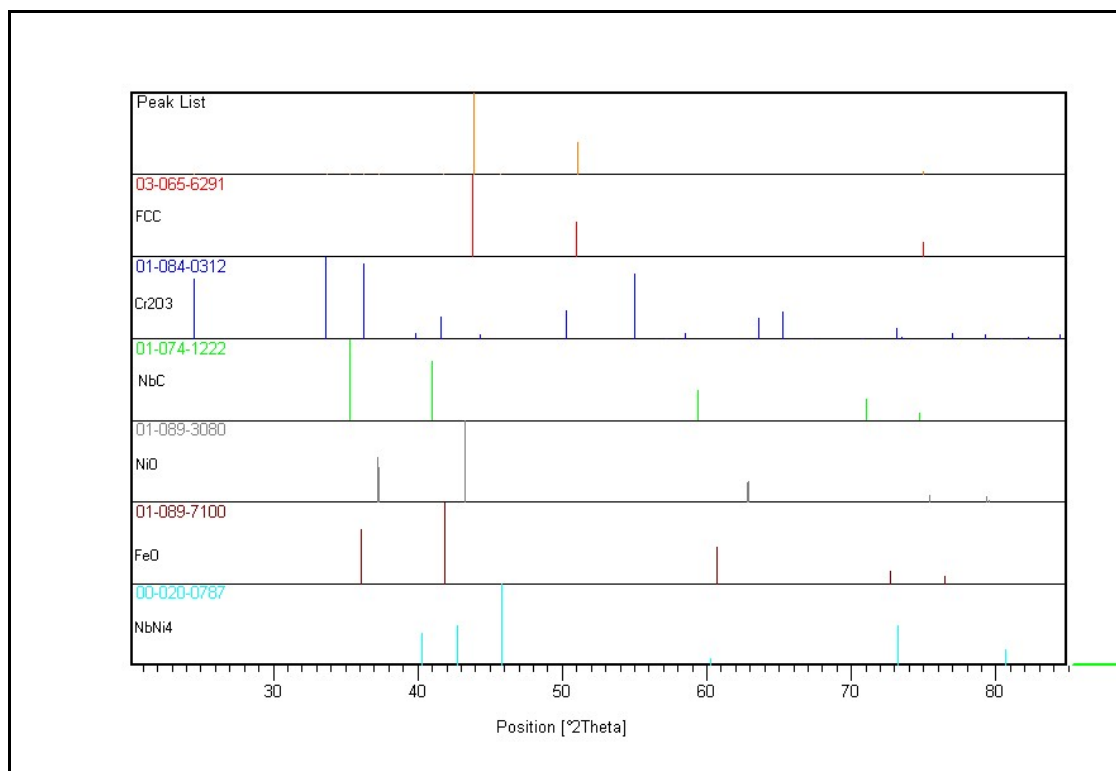
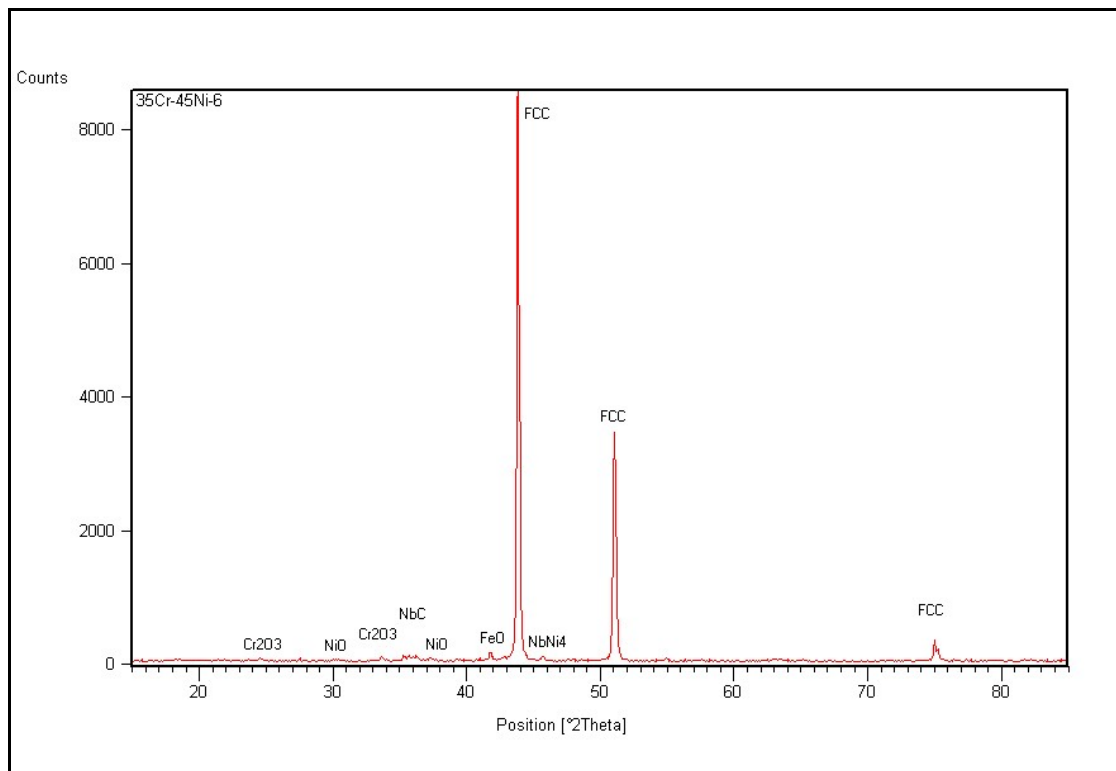
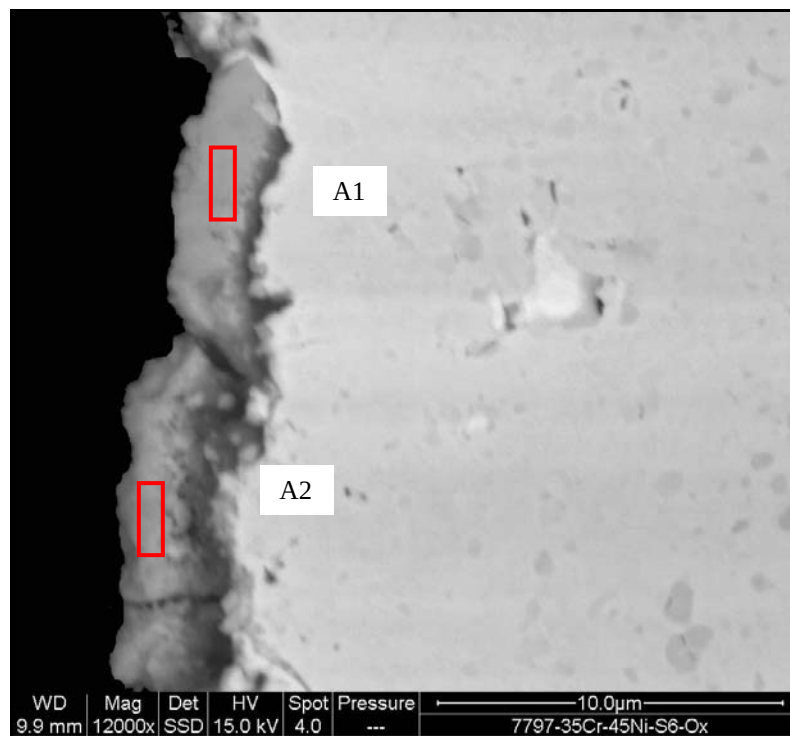
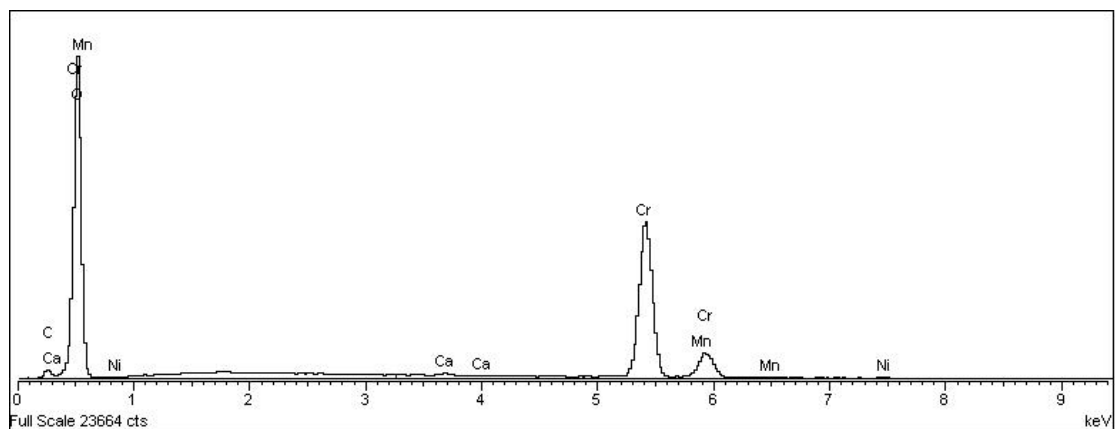


Figure 2.23 XRD pattern recorded from the alloy 35Cr-45Ni after exposure at 850°C.



A1



A2

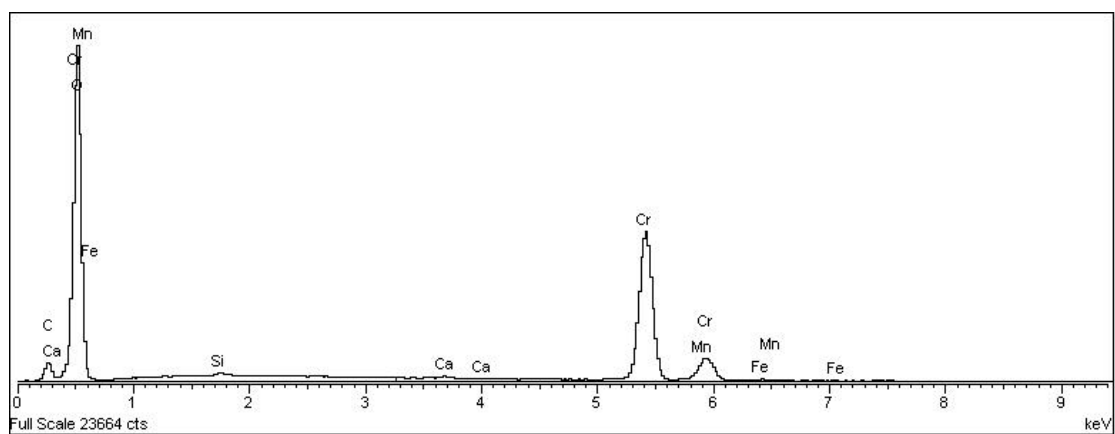


Figure 2.24 EDX analyses of the oxides formed on alloy 35Cr-45Ni after the 850°C experiment.

G Oxidation of Alloy UCX at 650°C for 100h

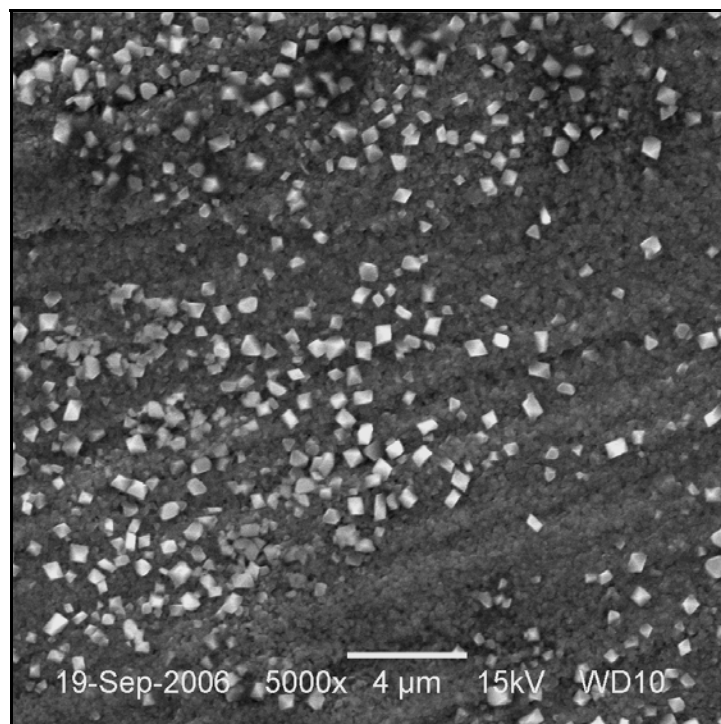
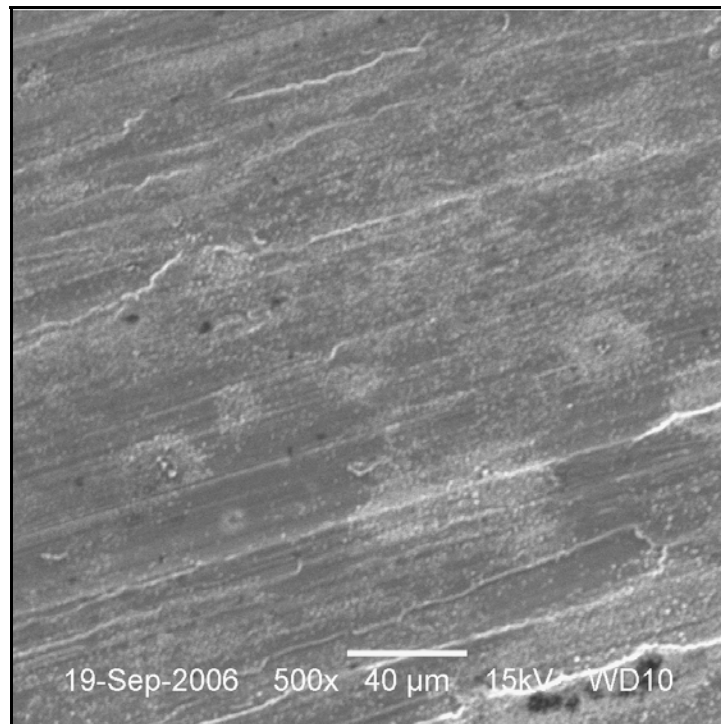


Figure 2.25 SEM/SE images of alloy UCX surfaces exposed at 650°C.

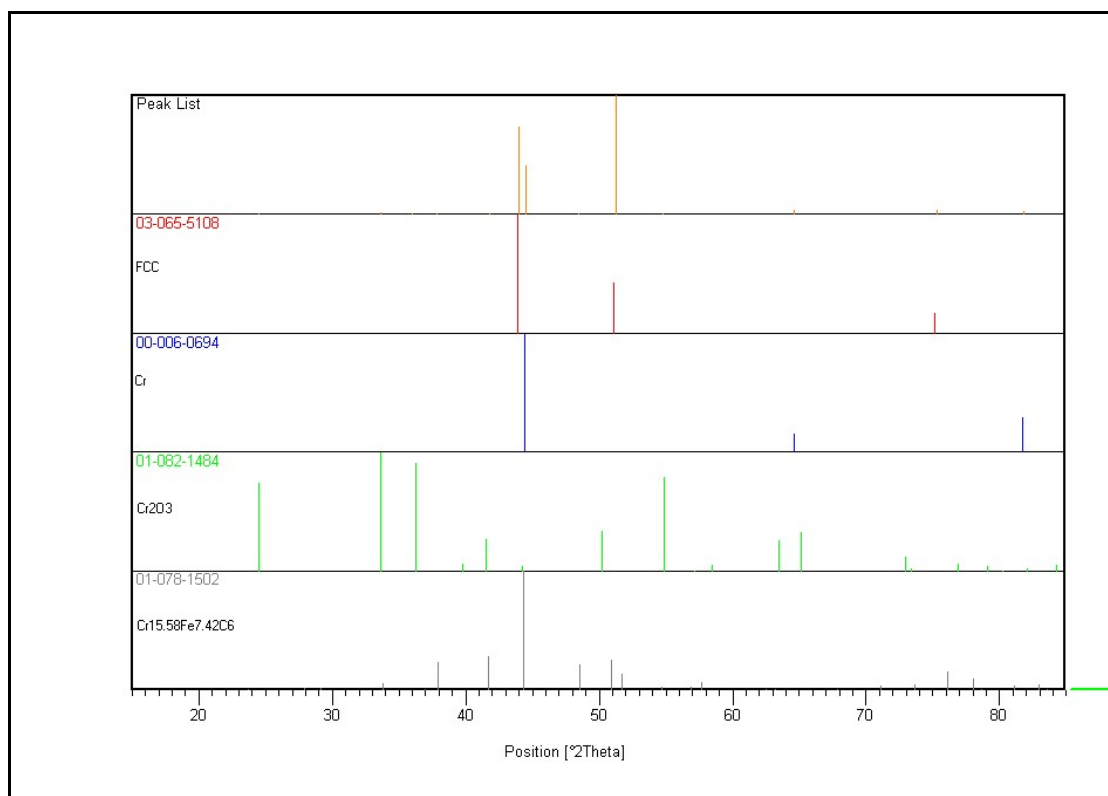
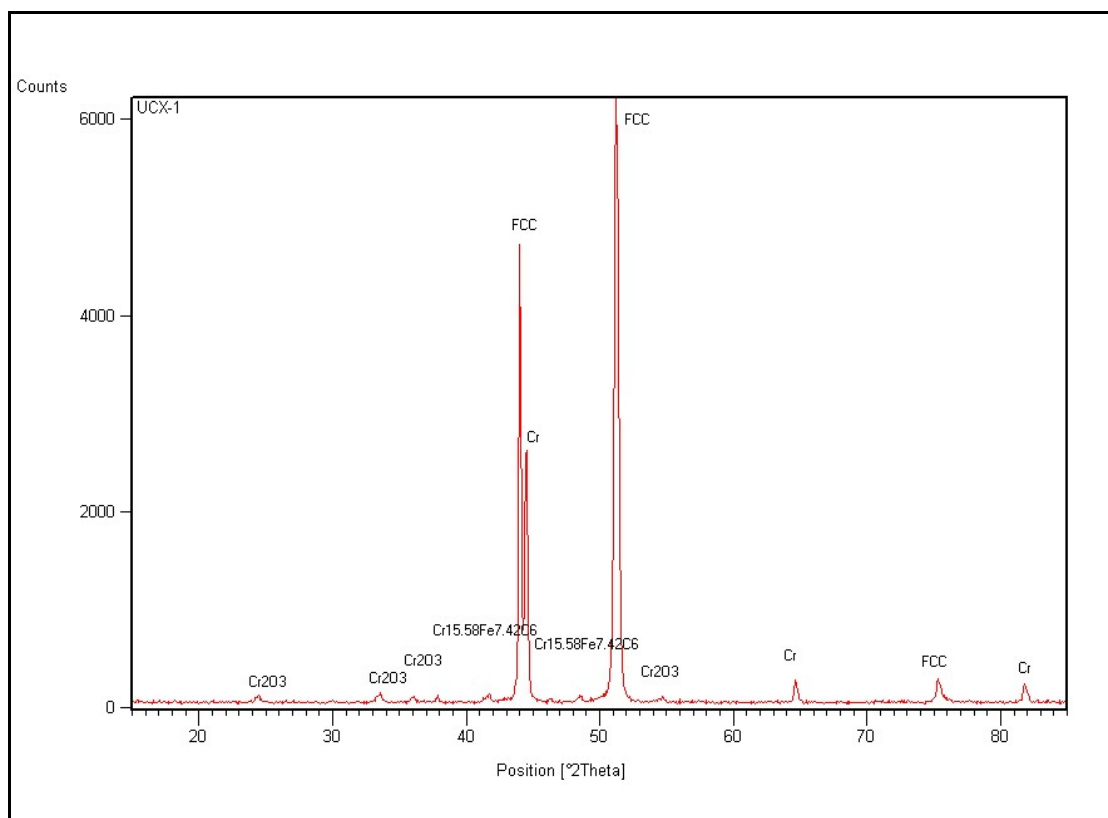
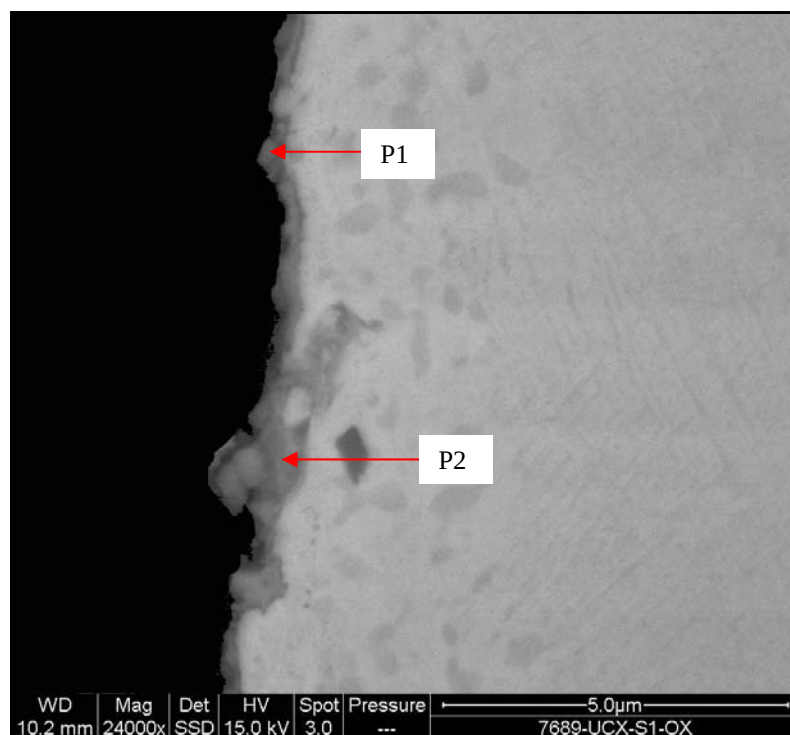
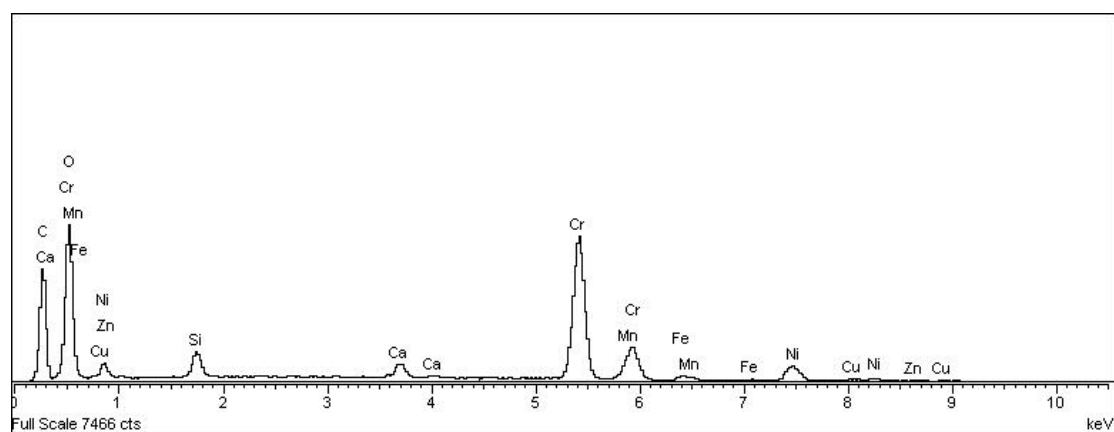


Figure 2.26 XRD pattern recorded from the alloy UCX after exposure at 650°C.



P1



P2

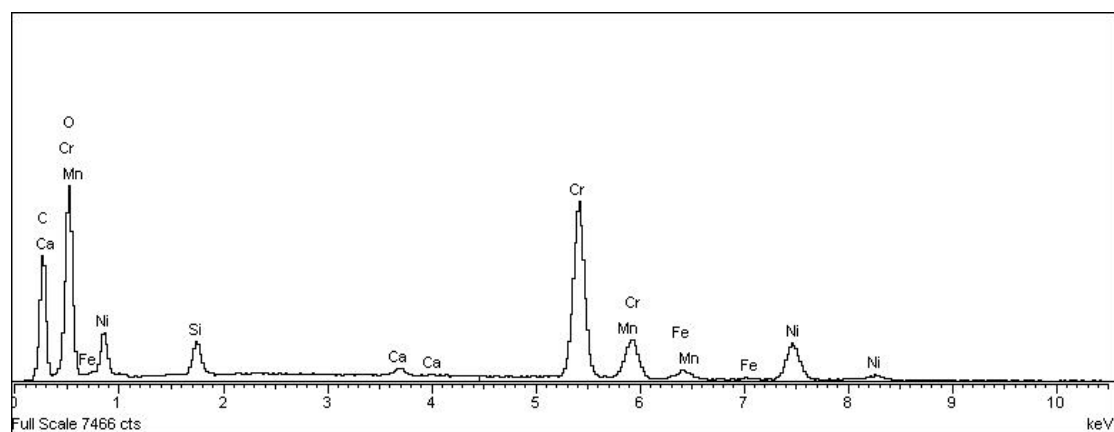


Figure 2.27 EDX analyses of the oxides formed on alloy UCX after the 650°C experiment.

H Oxidation of Alloy UCX at 750°C for 100h

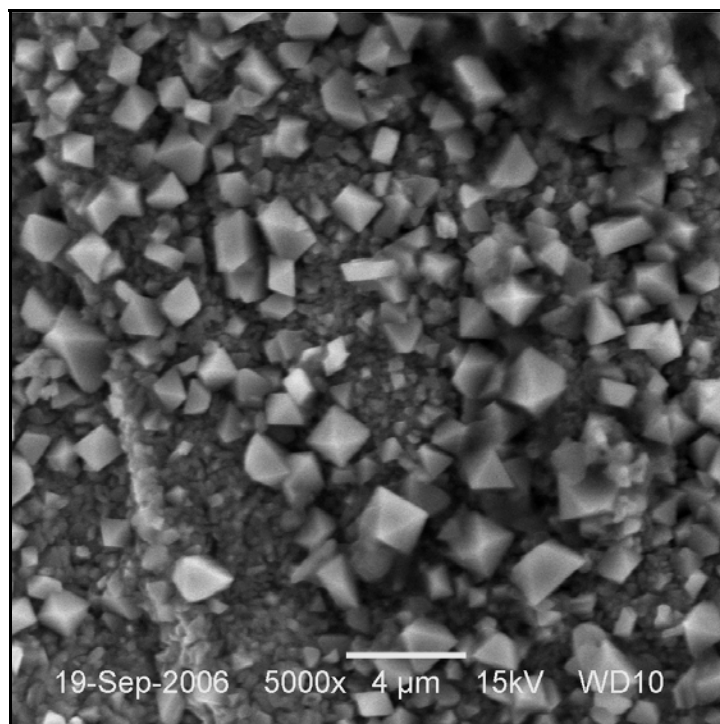
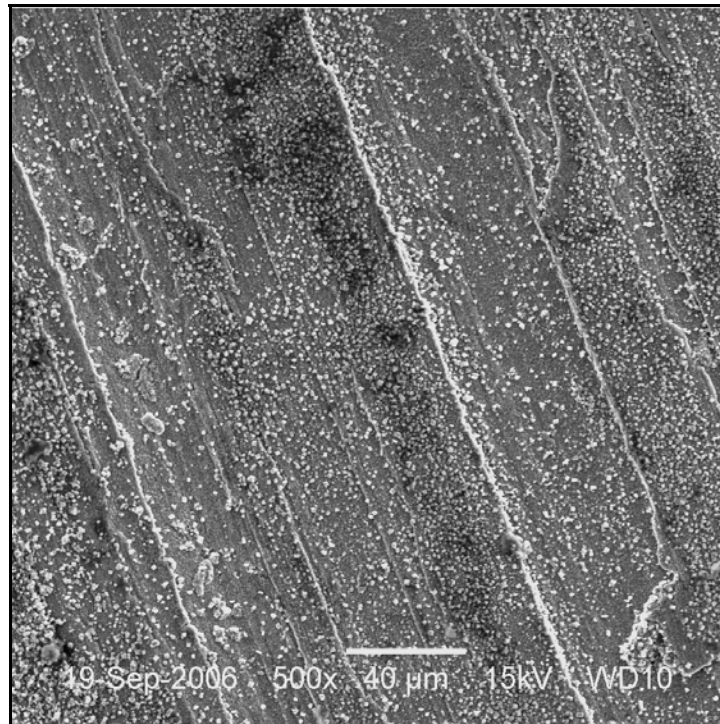


Figure 2.28 SEM/SE images of alloy UCX surfaces exposed at 750°C.

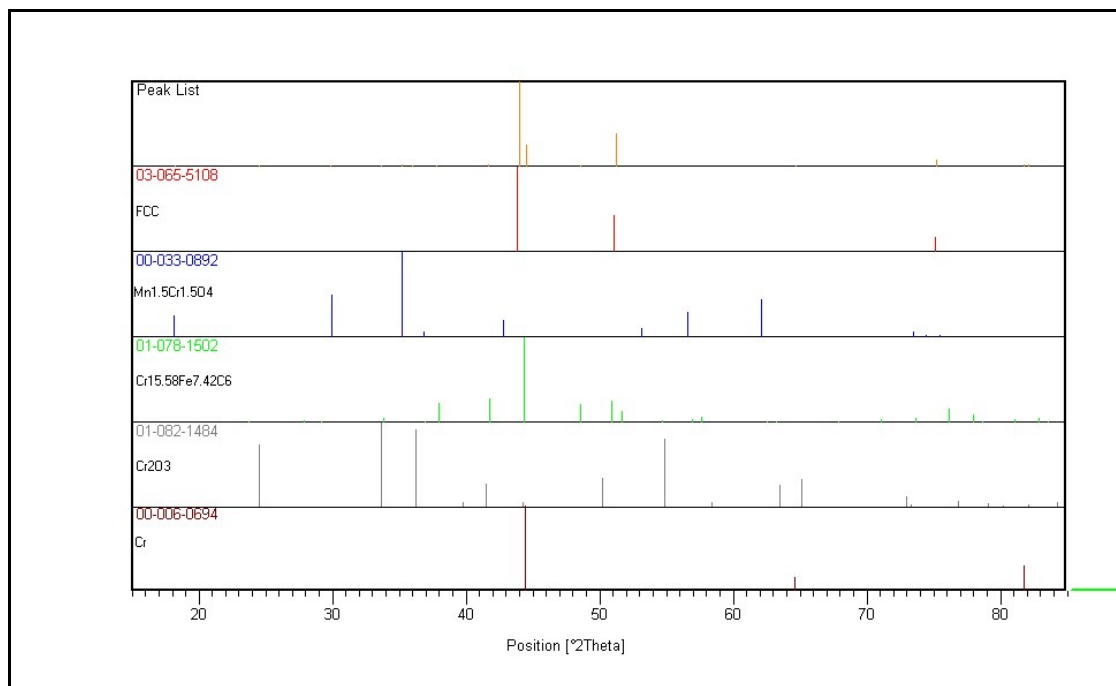
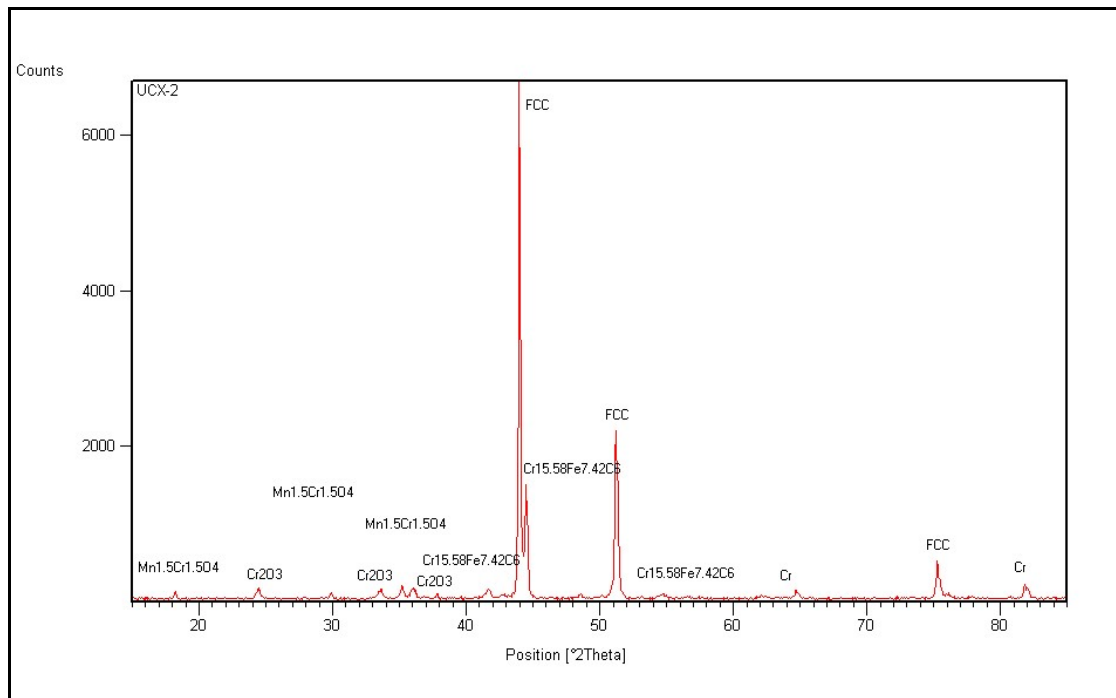
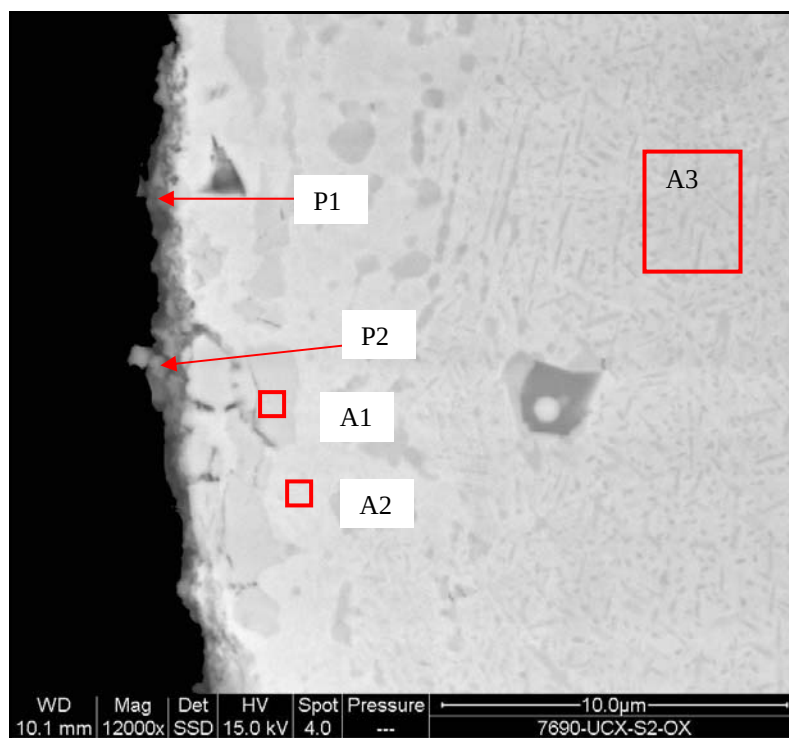
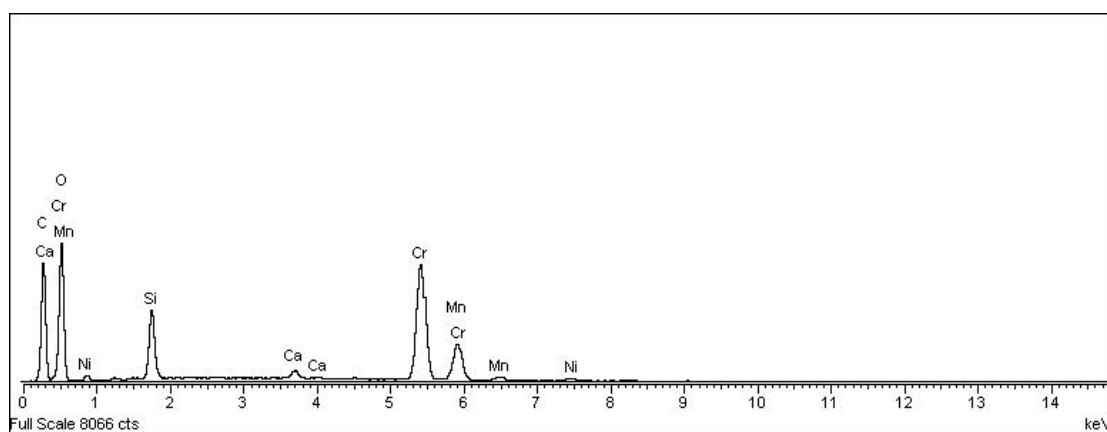


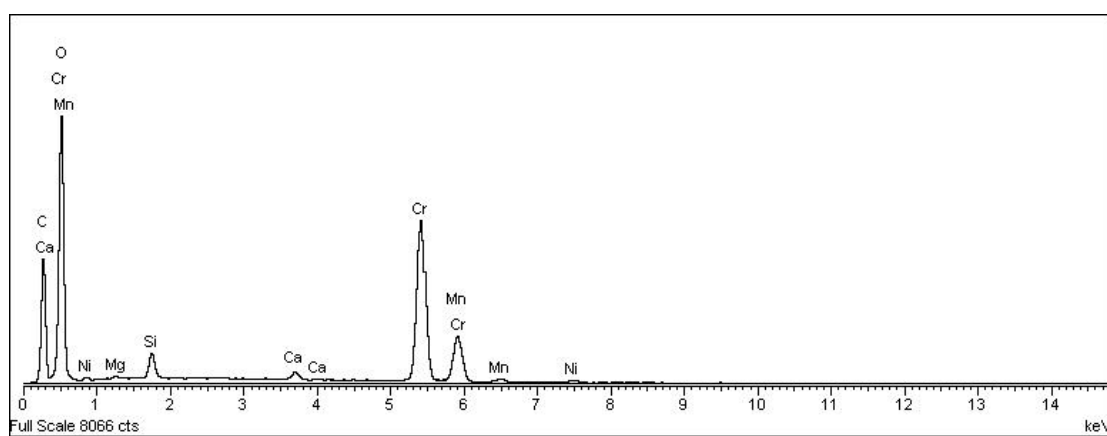
Figure 2.29 XRD pattern recorded from the alloy UCX after exposure at 750°C.



P1

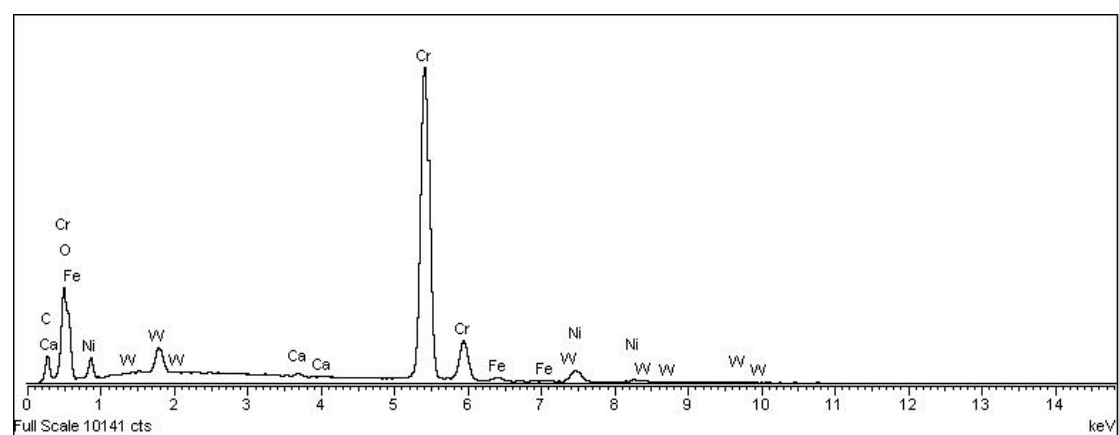


P2

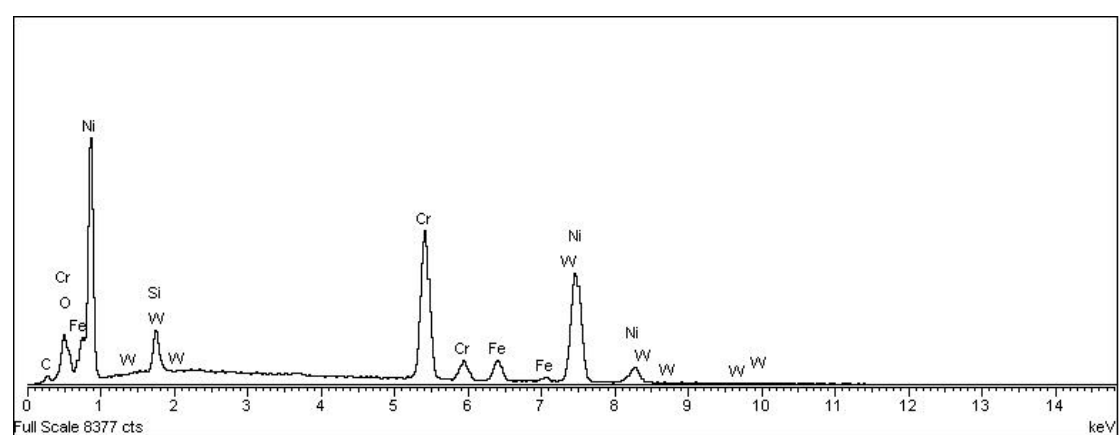


continue...

A1



A2



A3

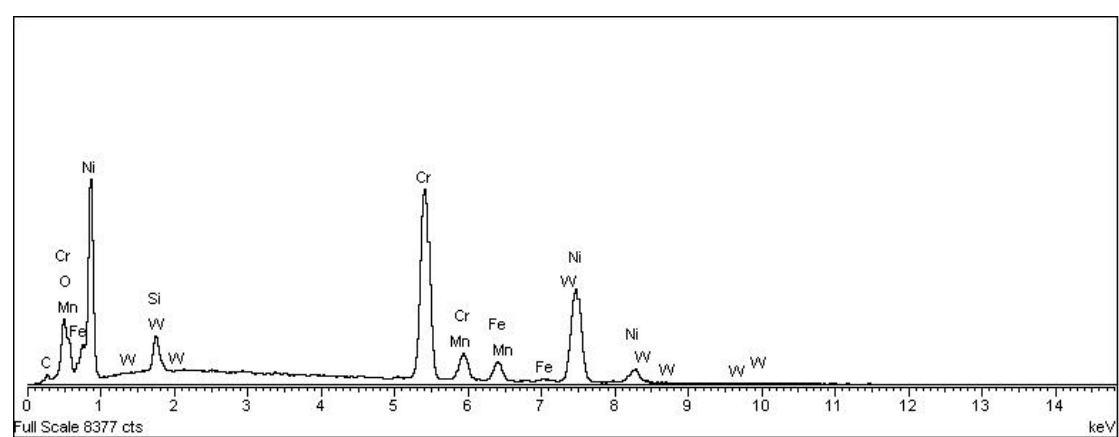


Figure 2.30 EDX analyses of the oxides formed on alloy UCX after the 750°C experiment.

I Oxidation of Alloy UCX at 850°C for 100h

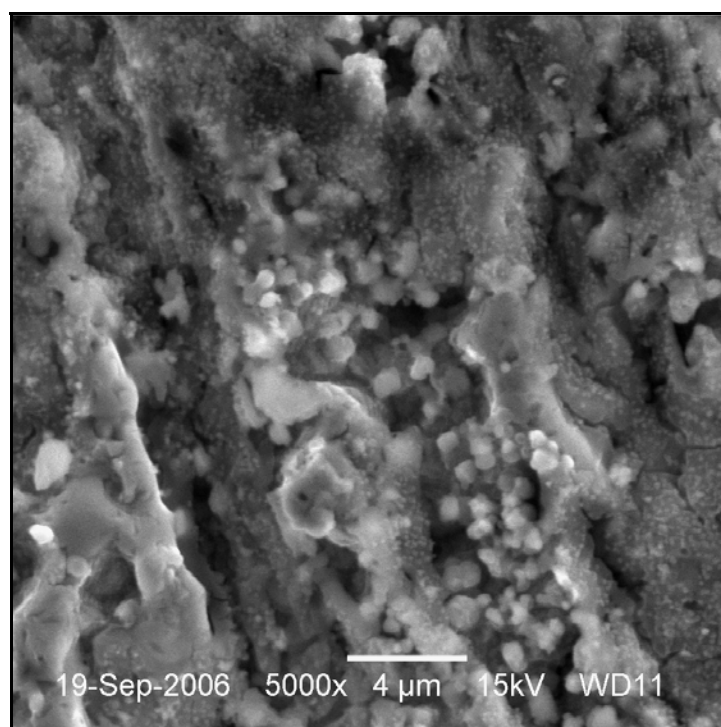
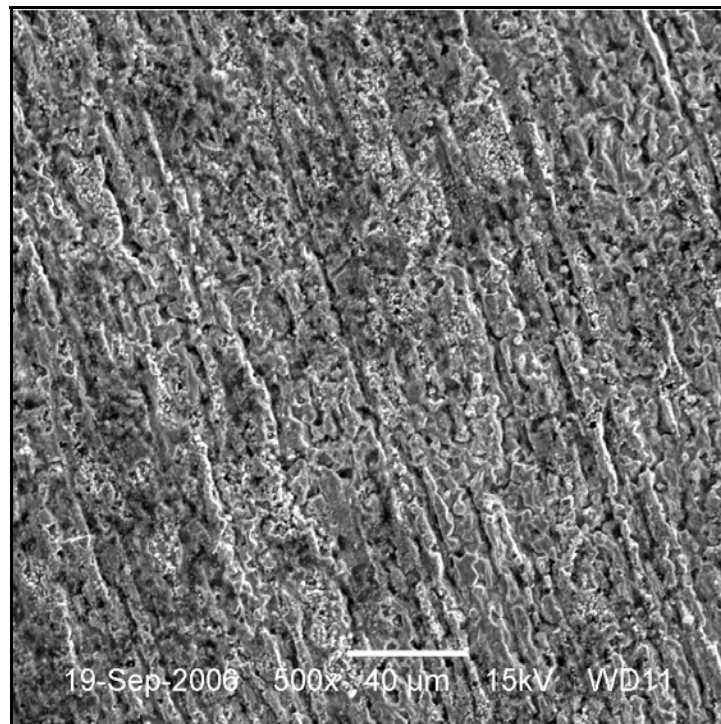


Figure 2.31 SEM/SE images of alloy UCX surfaces exposed at 850°C.

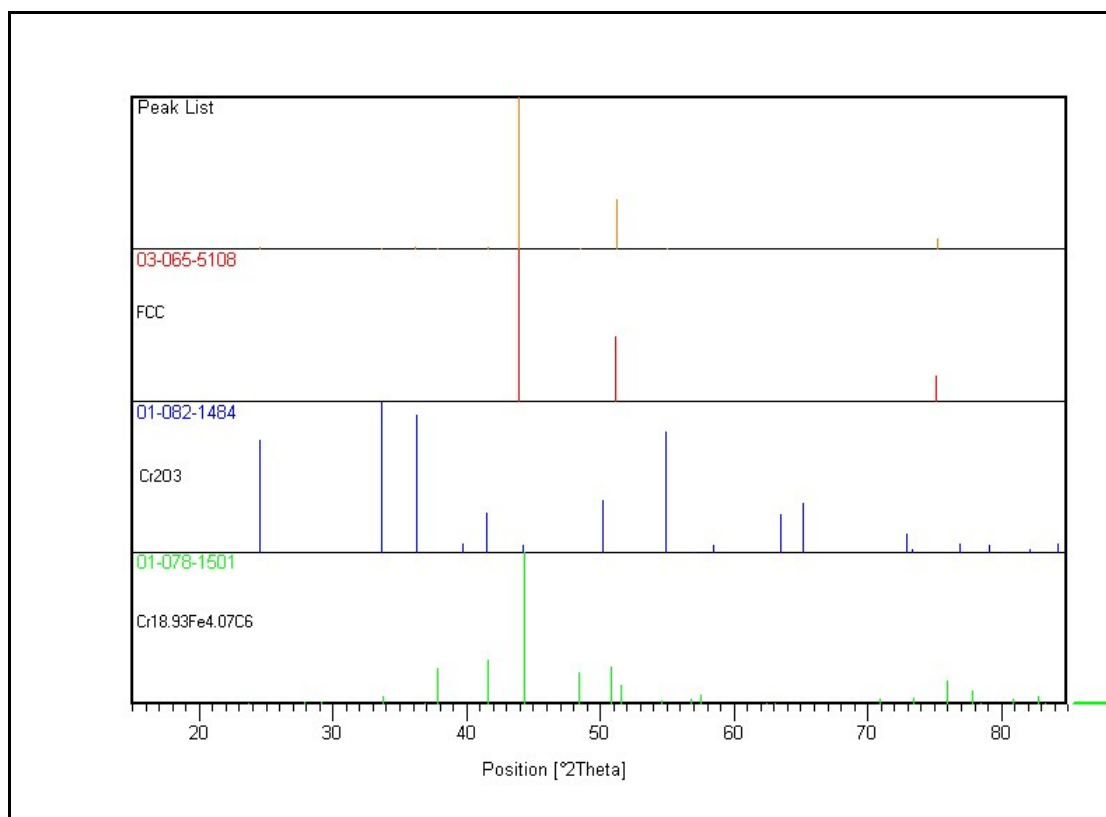
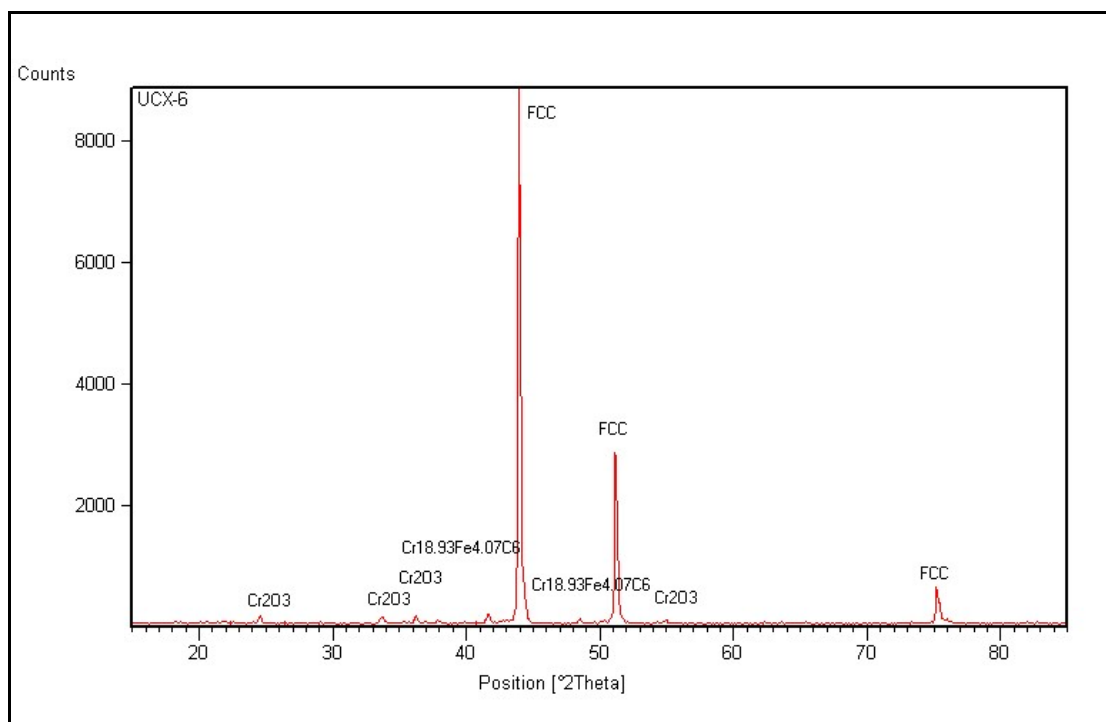
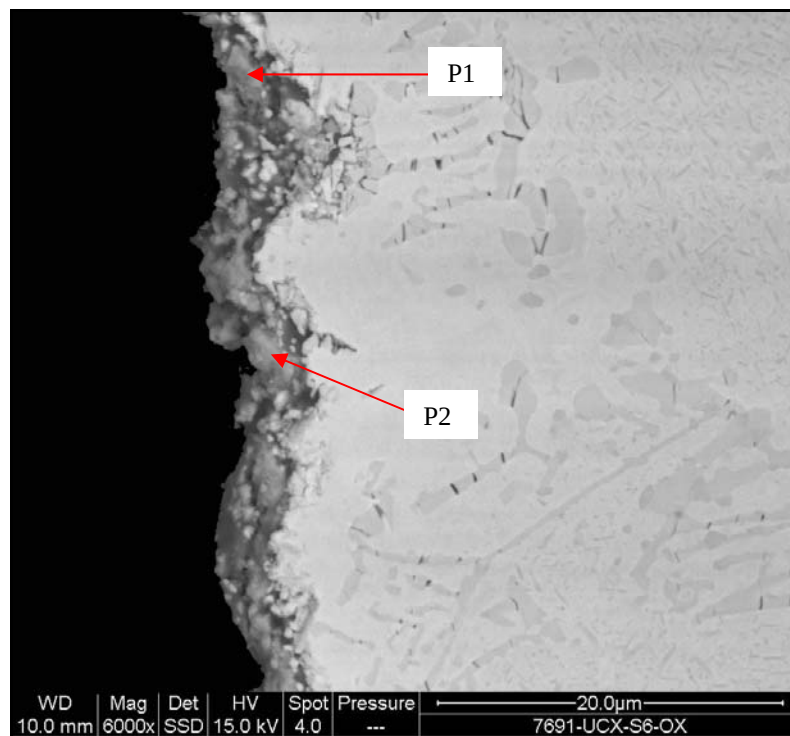
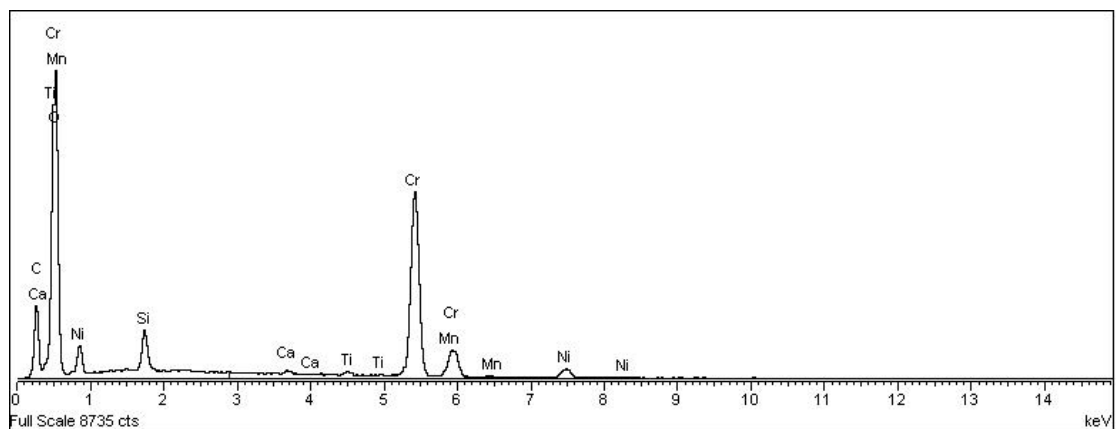


Figure 2.32 XRD pattern recorded from the alloy UCX after exposure at 850°C.



P1



P2

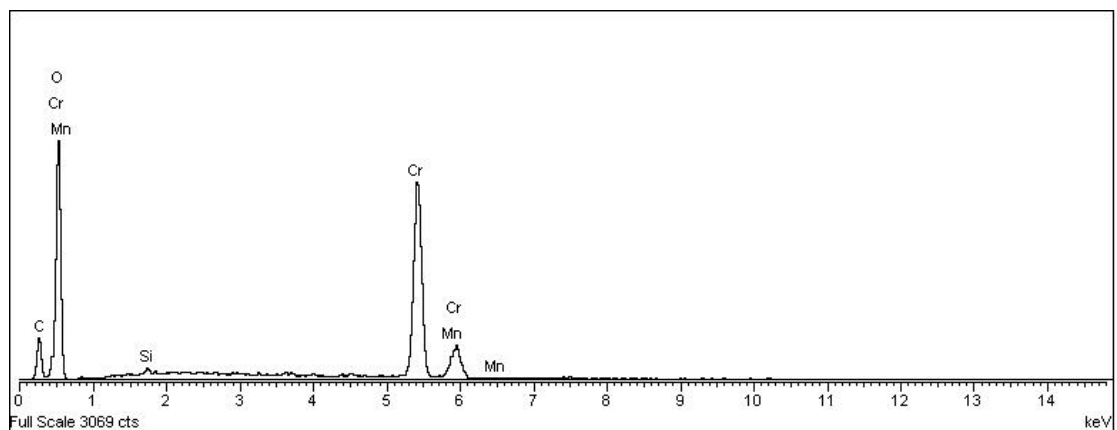


Figure 2.33 EDX analyses of the oxides formed on alloy UCX after the 850°C experiment.

2.2 Long-Term Experiments

2.2.1 Furnaces Heating and Cooling

Two, chamber furnaces were utilised to conduct the long-term studies. The 650°C test was carried out in Carbolite furnace (type CSF 11/7) whilst the others were accomplished in Carbolite furnace (type ELF 11/6). The heating and cooling rates of both furnaces are plotted in Figures 2.34-2.38.

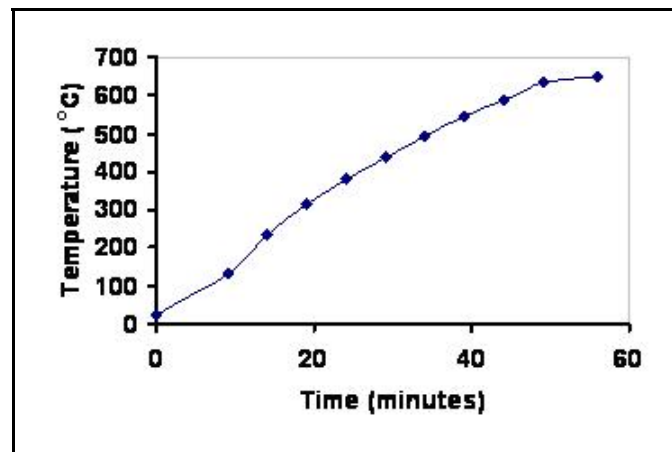


Figure 2.34 Heating up to 650°C.

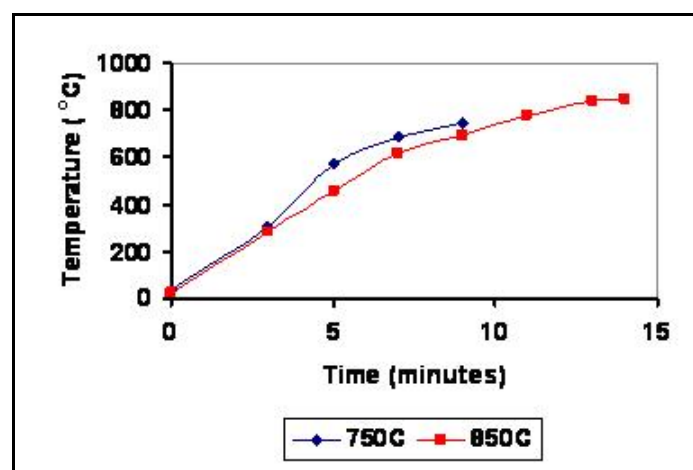


Figure 2.35 Heating up to 750 and 850°C.

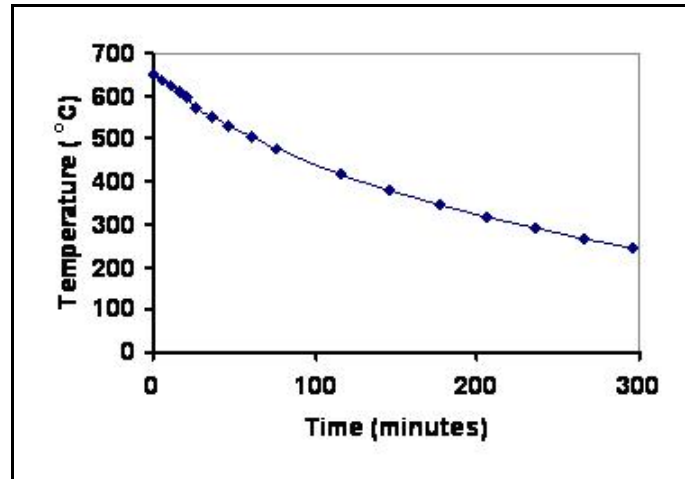


Figure 2.36 Cooling down from 650°C.

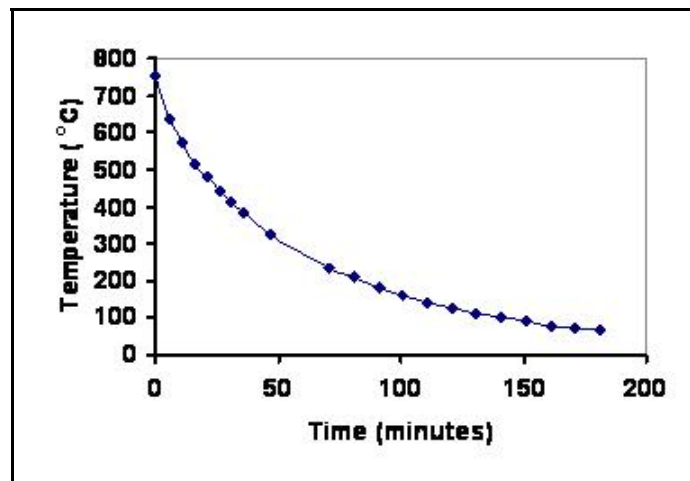


Figure 2.37 Cooling down from 750°C.

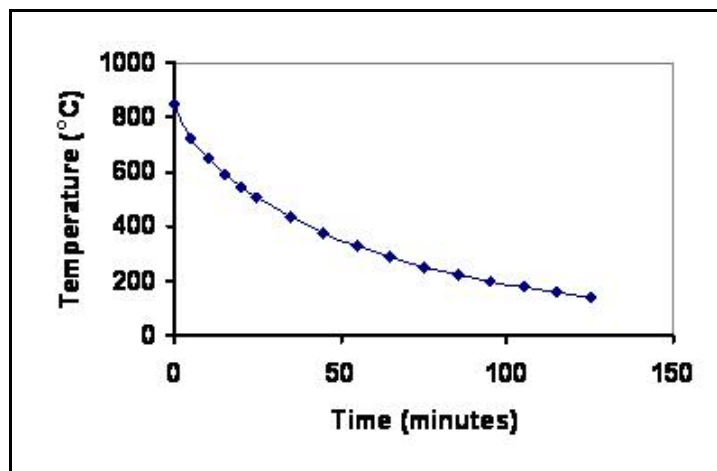


Figure 2.38 Cooling down from 850°C.

2.2.2 Weight Change Measurements

A four decimal, digital balance (Mettler AT261 DeltaRange) was used for weight measurements, Table 2.2. Each sample dimension was measured using digital callipers and a micrometer. Figure 2.39 revealed that the oxides formed on the alloys exposed at 850°C were subject to spallation.

Table 2.2 Weight change measurements after the exposure to air at 650, 750, and 850°C for 1000h. The actual surface areas of the samples were measured as well.

Alloy	T, °C	W _{before} ,g	W _{after} ,g	A, cm ²	ΔW, mg/cm ²
HP	650	15.3703	15.3725	11.8861	0.1851
35Cr-45Ni	650	14.7666	14.7684	11.8076	0.1524
UCX	650	15.3703	15.3724	11.9149	0.1762
HP	750	15.4383	15.4411	11.8972	0.2353
35Cr-45Ni	750	14.5529	14.5573	11.7143	0.3756
UCX	750	15.3981	15.4009	11.9193	0.2349
HP	850	15.4570	15.4576	11.9191	0.0503
35Cr-45Ni	850	15.3450	15.3426	11.8986	-0.2017
UCX	850	14.6661	14.6705	11.7593	0.3657



Figure 2.39 The specimens suffered spallation after the exposure at 850°C for 1000h.

2.2.3 SEM/EDX and XRD Analyses

The same SEM/EDX and XRD machines used to analyse the short-term samples were utilised in examining the long-term specimens.

A Oxidation of Alloy HP at 650°C for 1000h

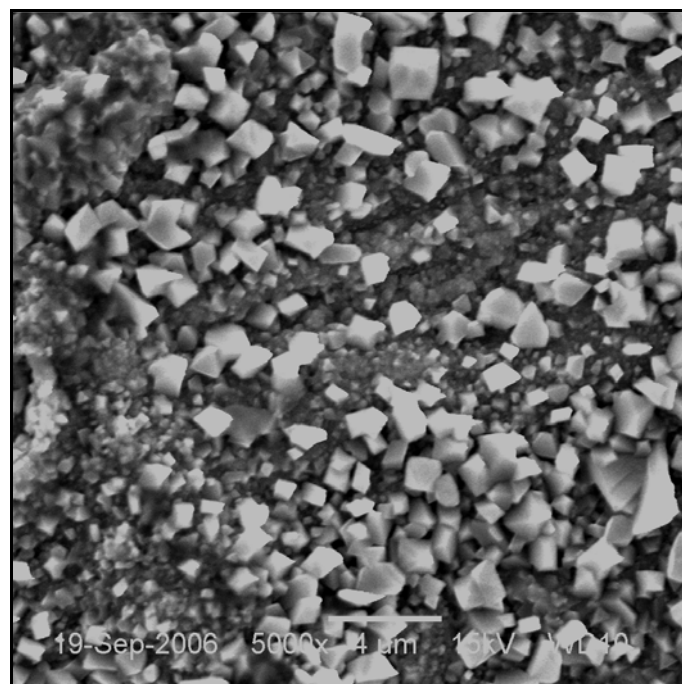
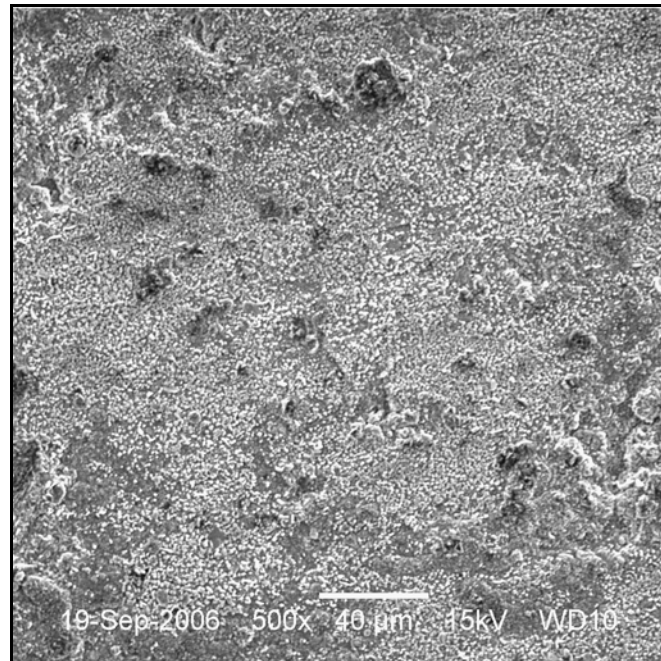


Figure 2.40 SEM/SE images of alloy HP surfaces exposed at 650°C.

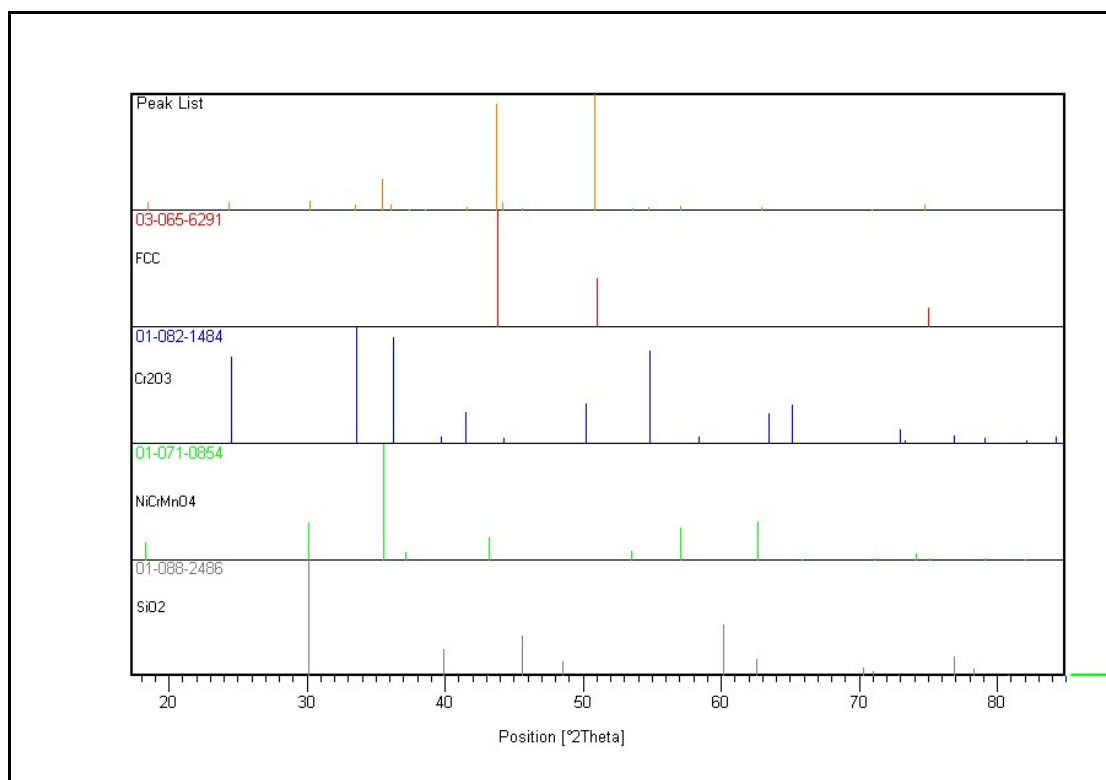
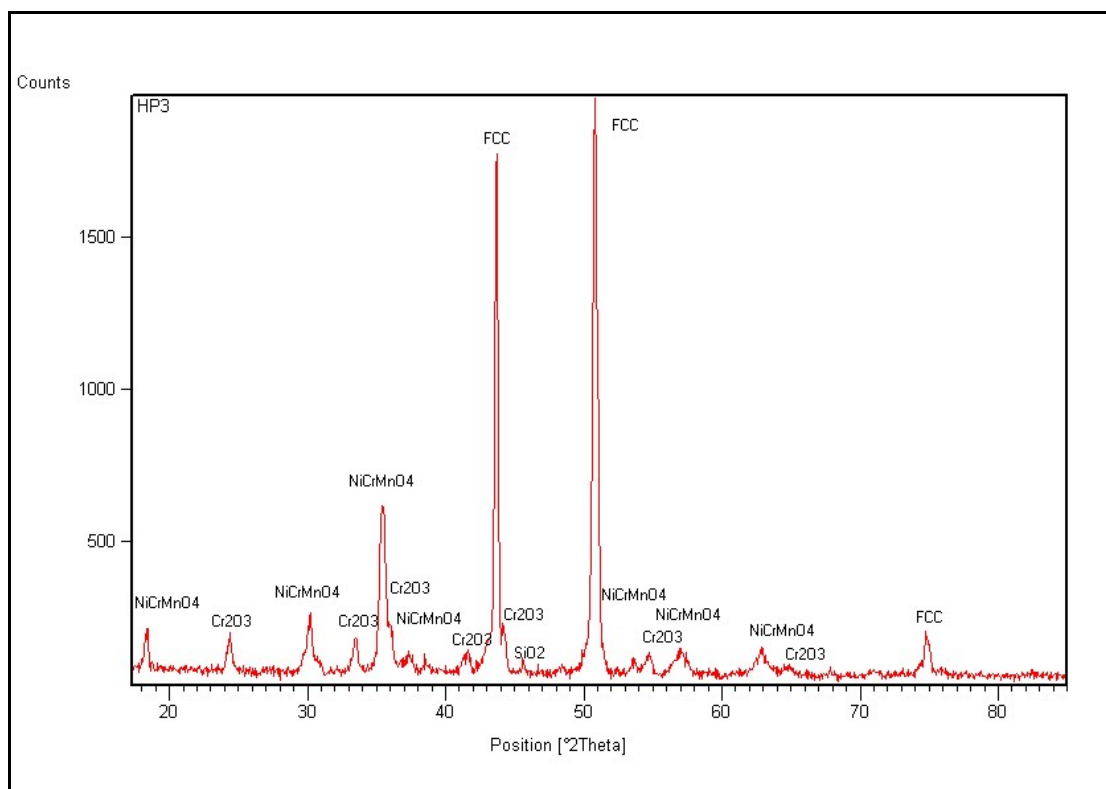
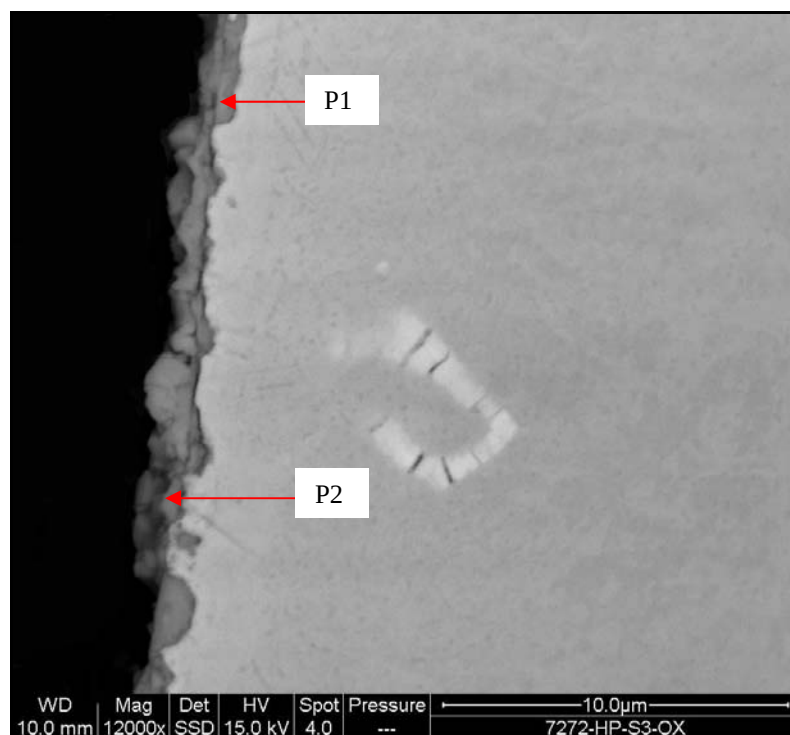
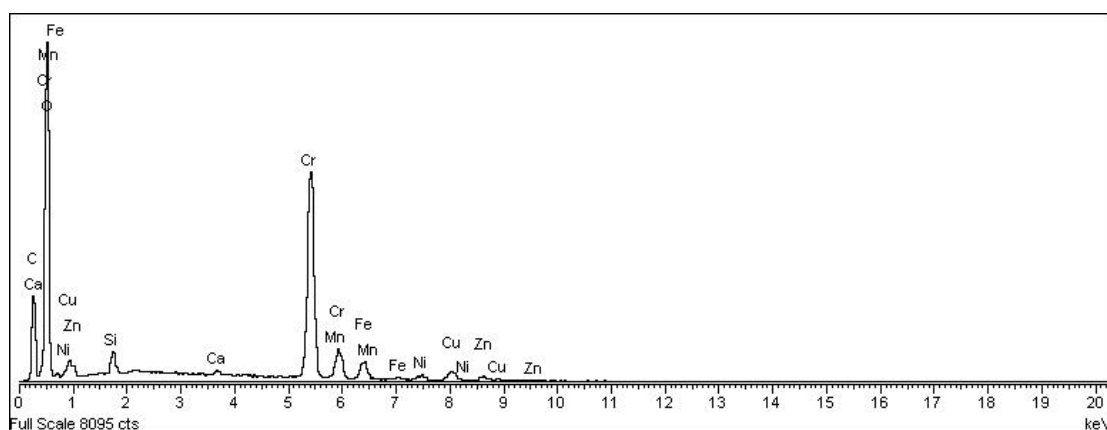


Figure 2.41 XRD pattern recorded from the alloy HP after exposure at 650°C.



P1



P2

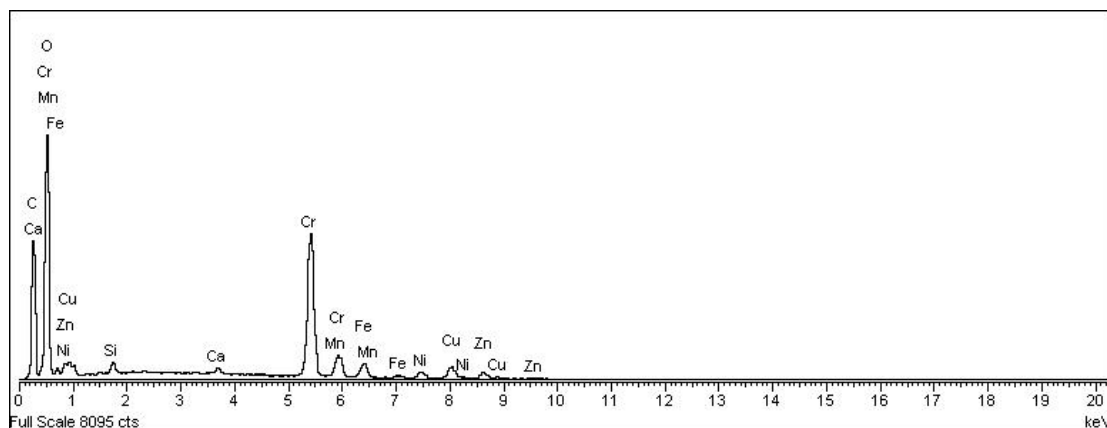


Figure 2.42 EDX analyses of the oxides formed on alloy HP after the 650°C experiment.

B Oxidation of Alloy HP at 750°C for 1000h

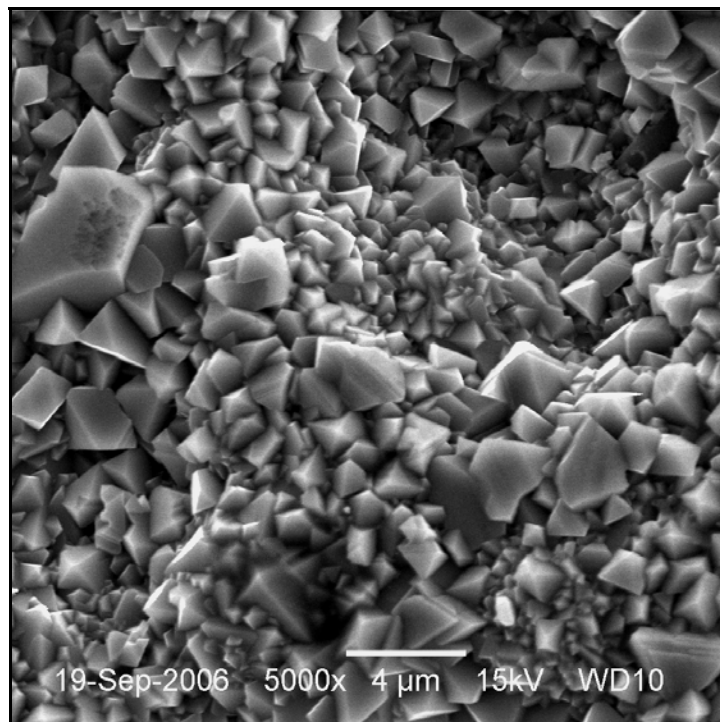
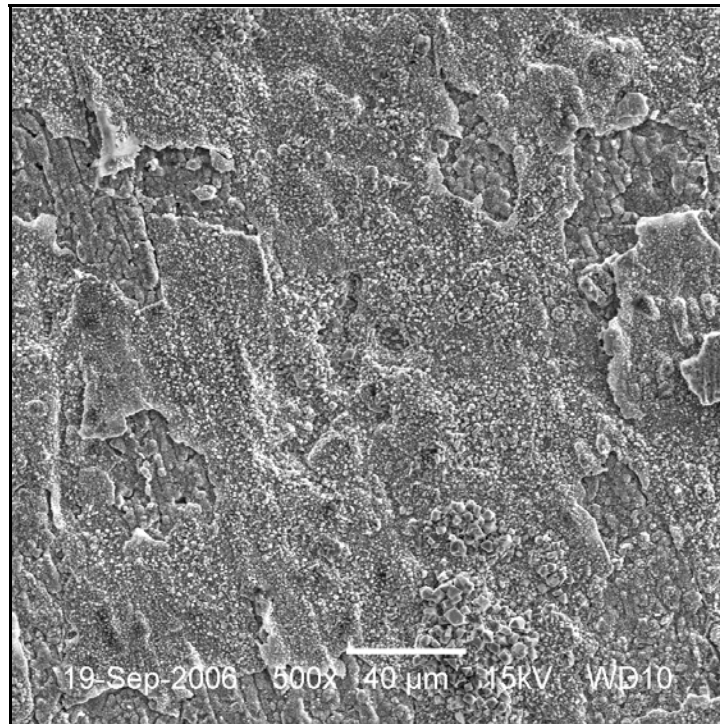
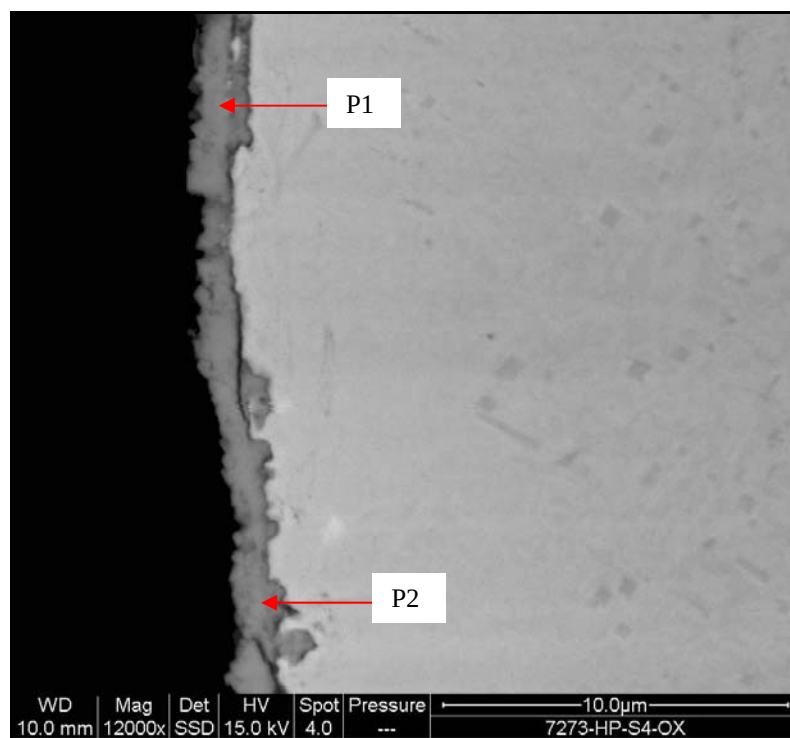
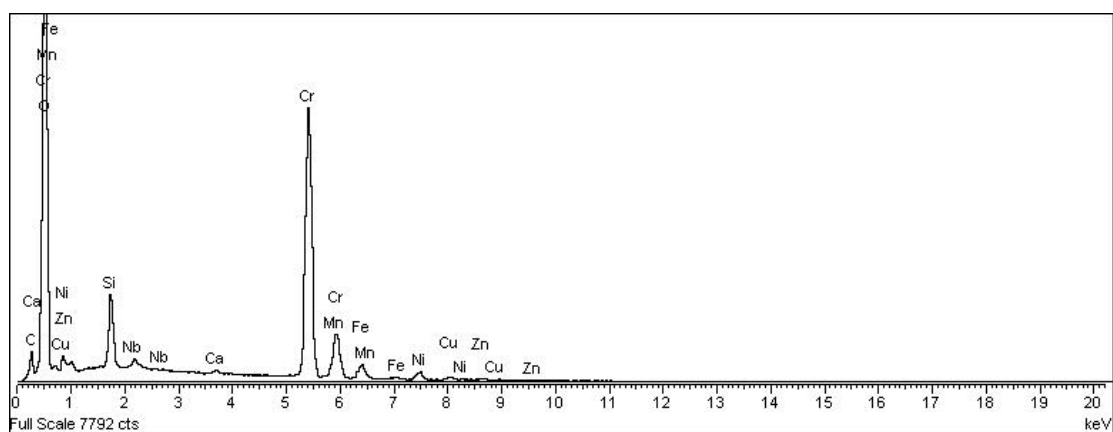


Figure 2.43 SEM/SE images of alloy HP surfaces exposed at 750°C.



P1



P2

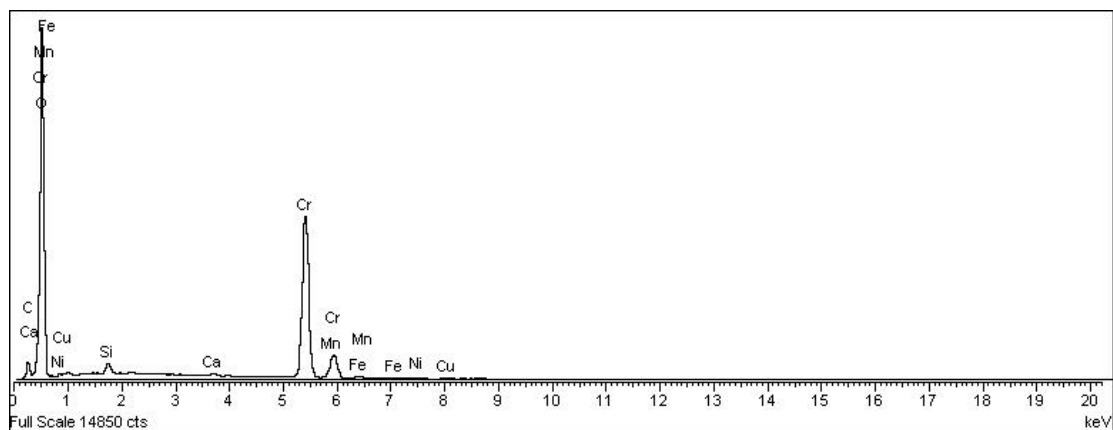


Figure 2.45 EDX analyses of the oxides formed on alloy HP after the 750°C experiment.

C *Oxidation of Alloy HP at 850°C for 1000h*

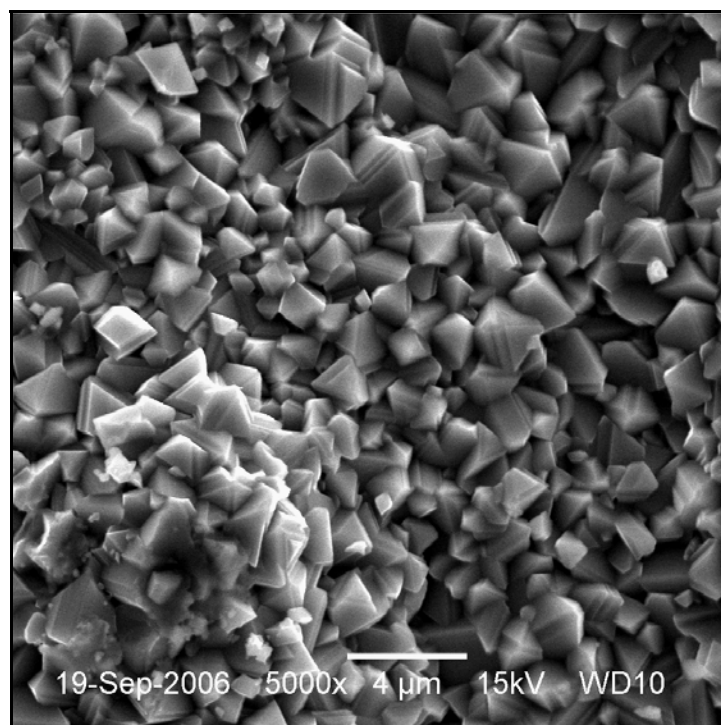
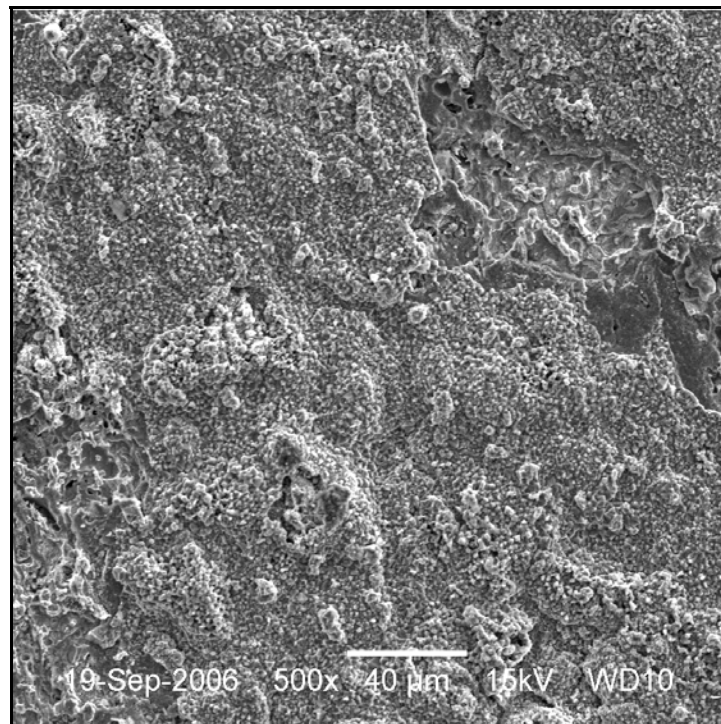
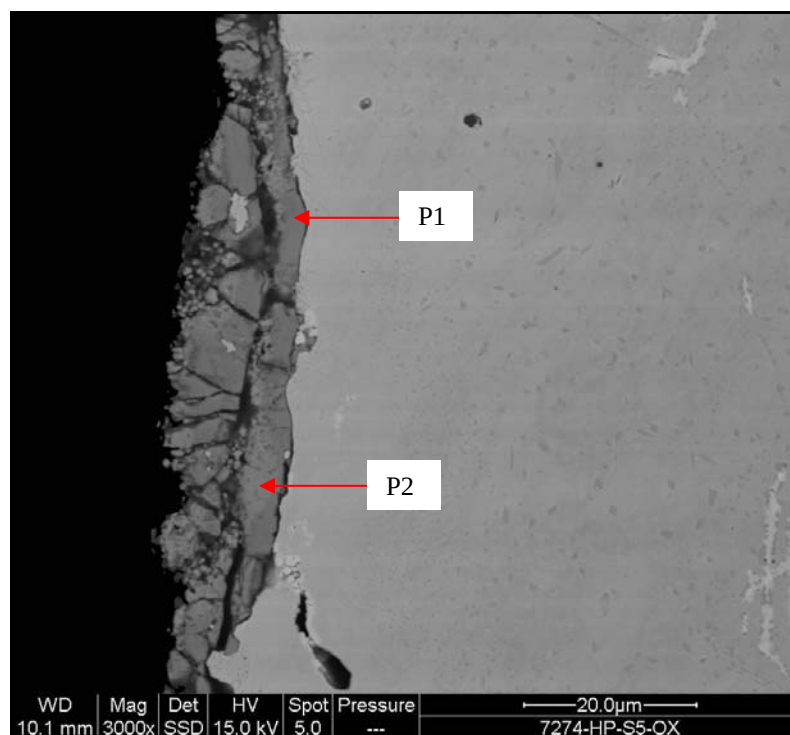
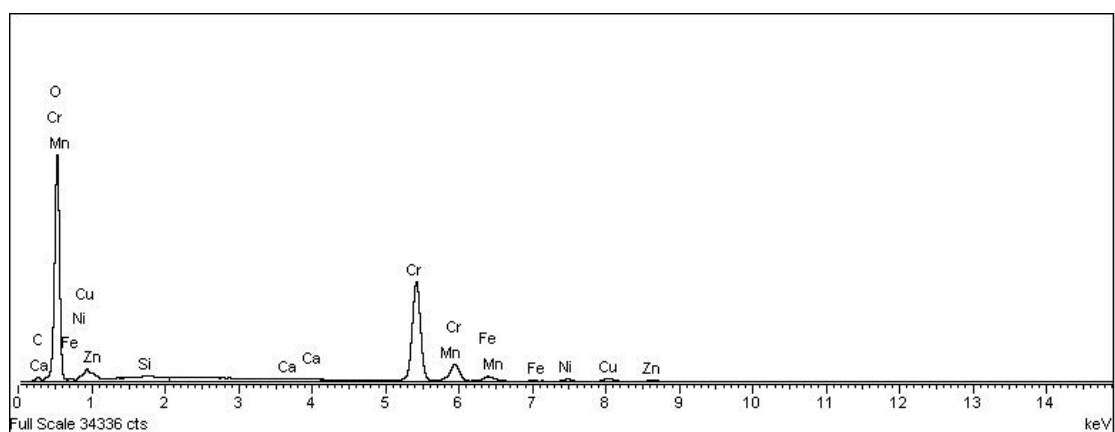


Figure 2.46 SEM/SE images of alloy HP surfaces exposed at 850°C.



P1



P2

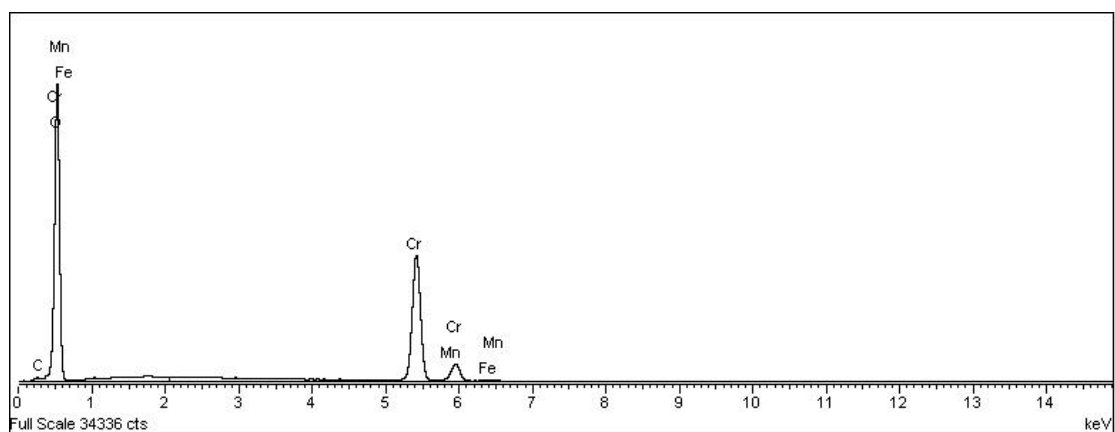


Figure 2.48 EDX analyses of the oxides formed on alloy HP after the 850°C experiment.

D Oxidation of Alloy 35Cr-45Ni at 650°C for 1000h

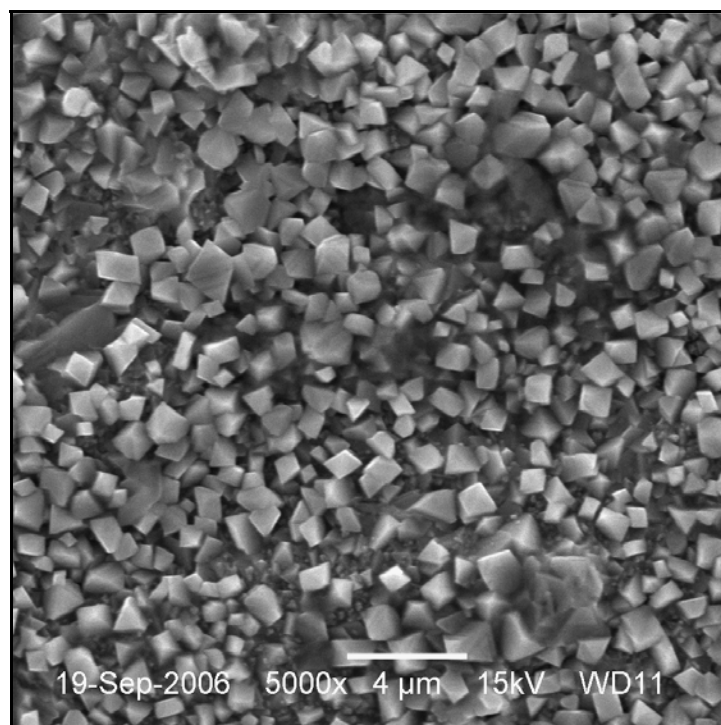
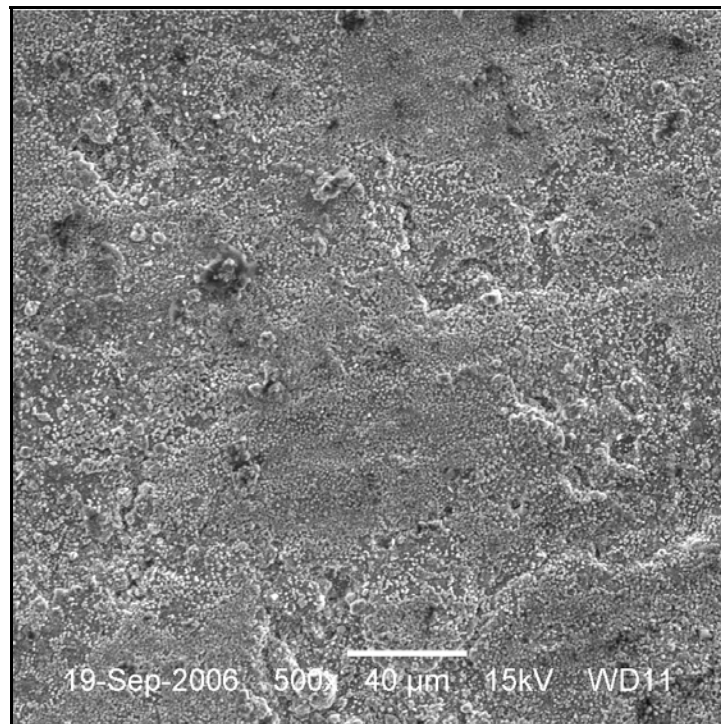


Figure 2.49 SEM/SE images of alloy 35Cr-45Ni surfaces exposed at 650°C.

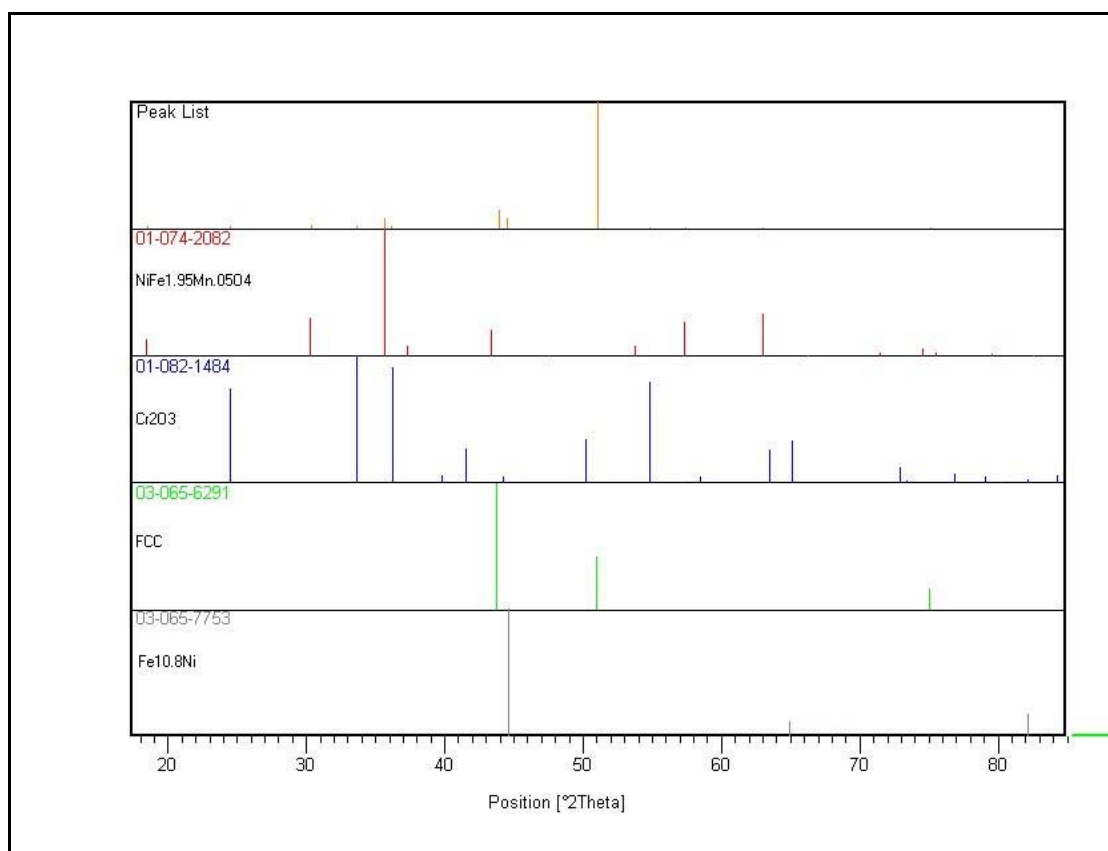
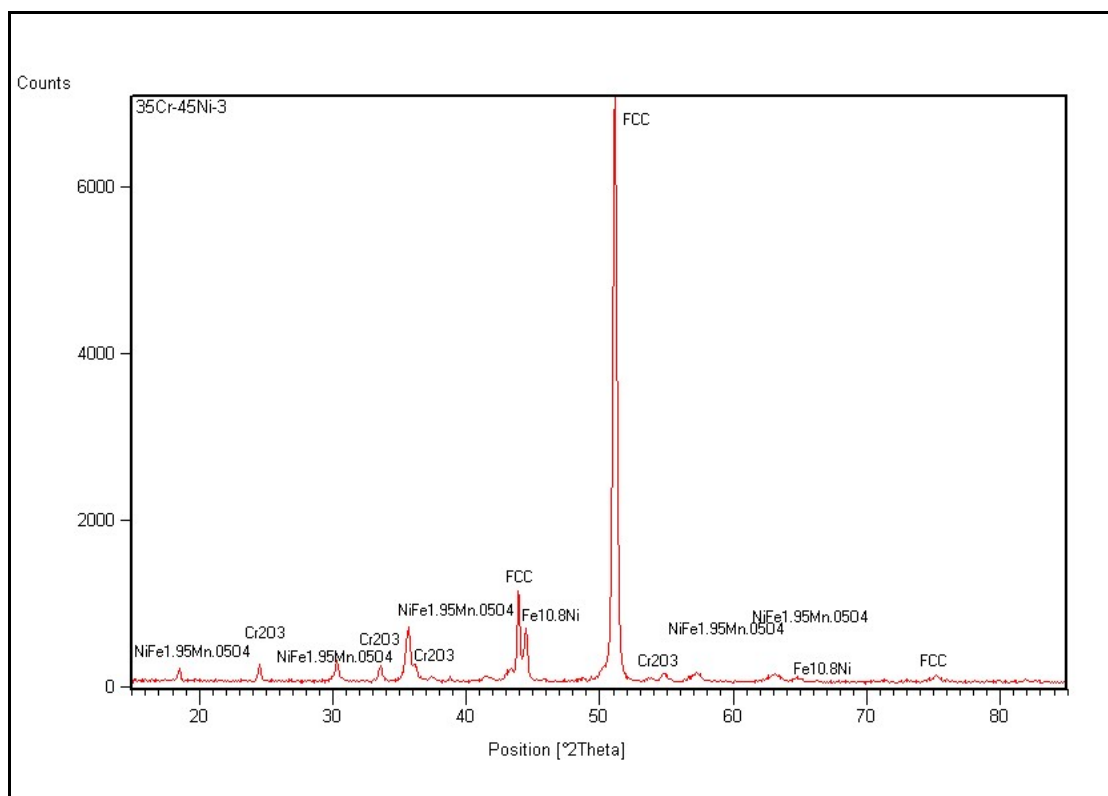
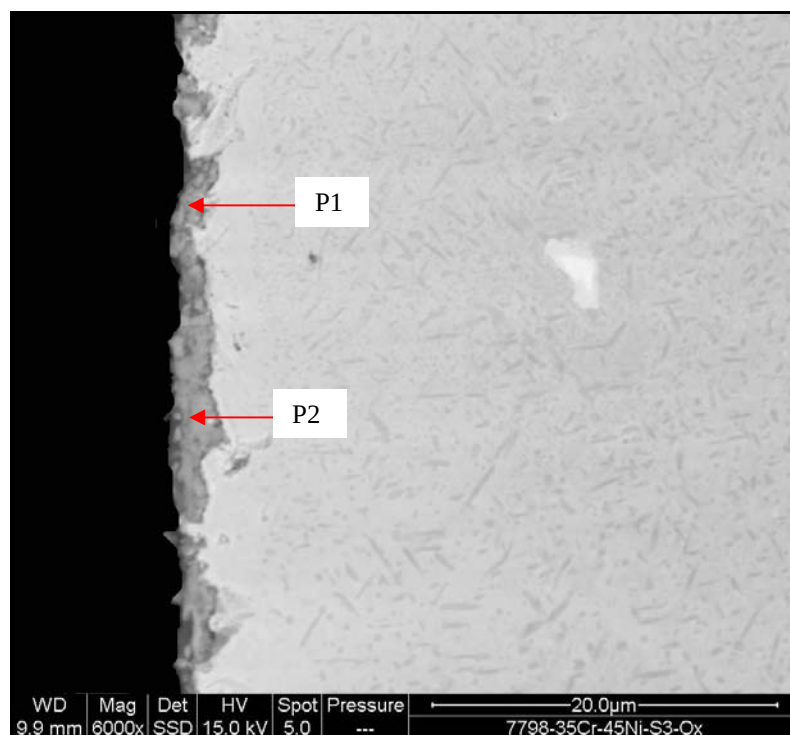
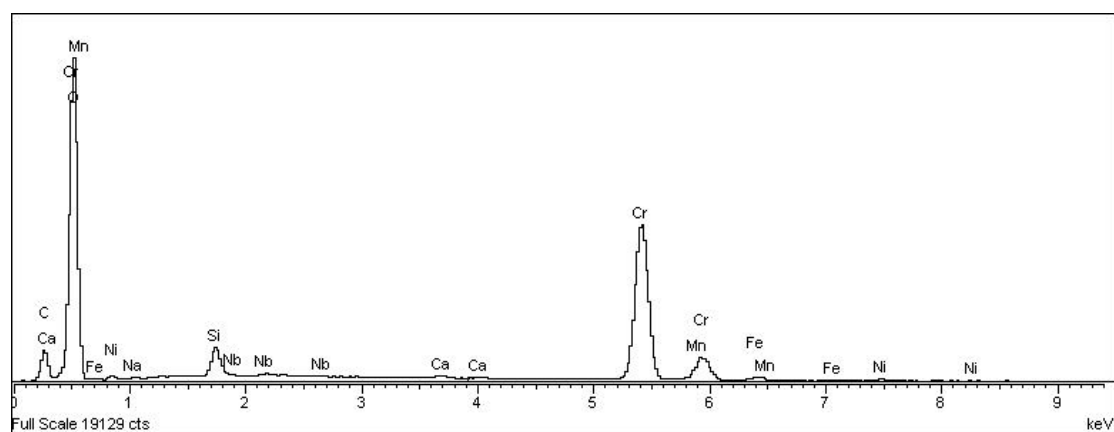


Figure 2.50 XRD pattern recorded from the alloy 35Cr-45Ni after exposure at 650°C.



P1



P2

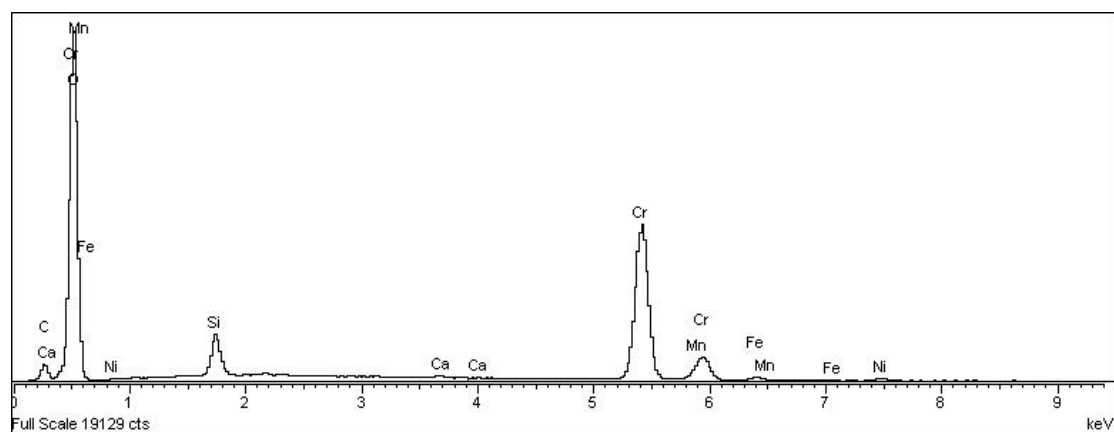


Figure 2.51 EDX analyses of the oxides formed on alloy 35Cr-45Ni after the 650°C experiment.

E Oxidation of Alloy 35Cr-45Ni at 750°C for 1000h

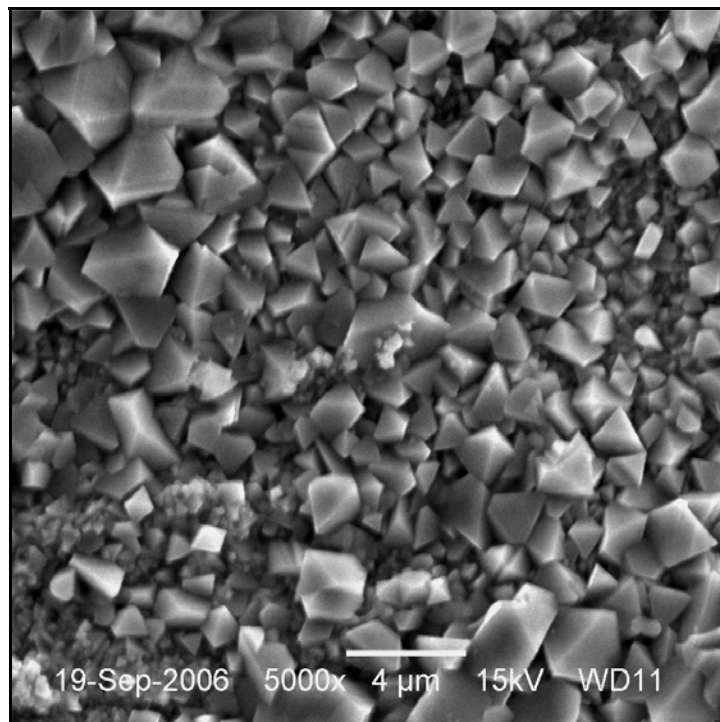
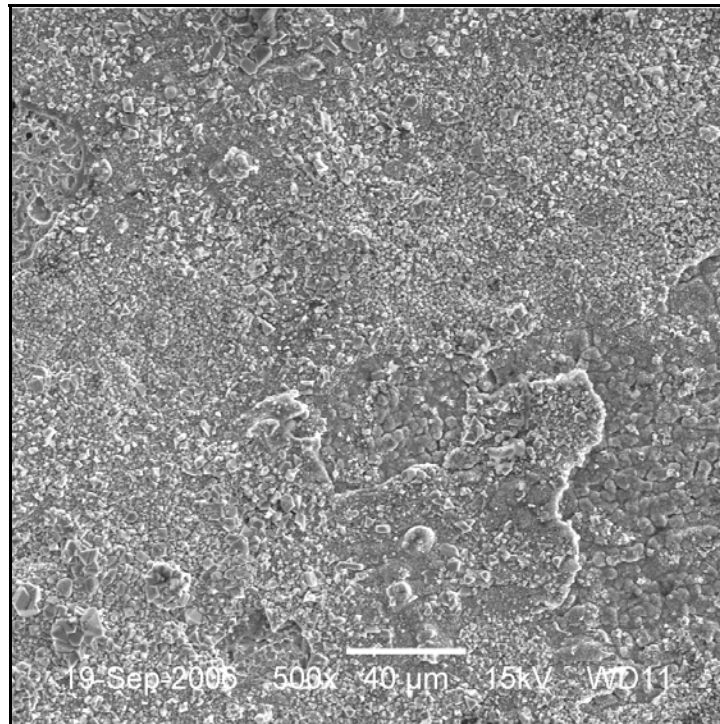


Figure 2.52 SEM/SE images of alloy 35Cr-45Ni surfaces exposed at 750°C.

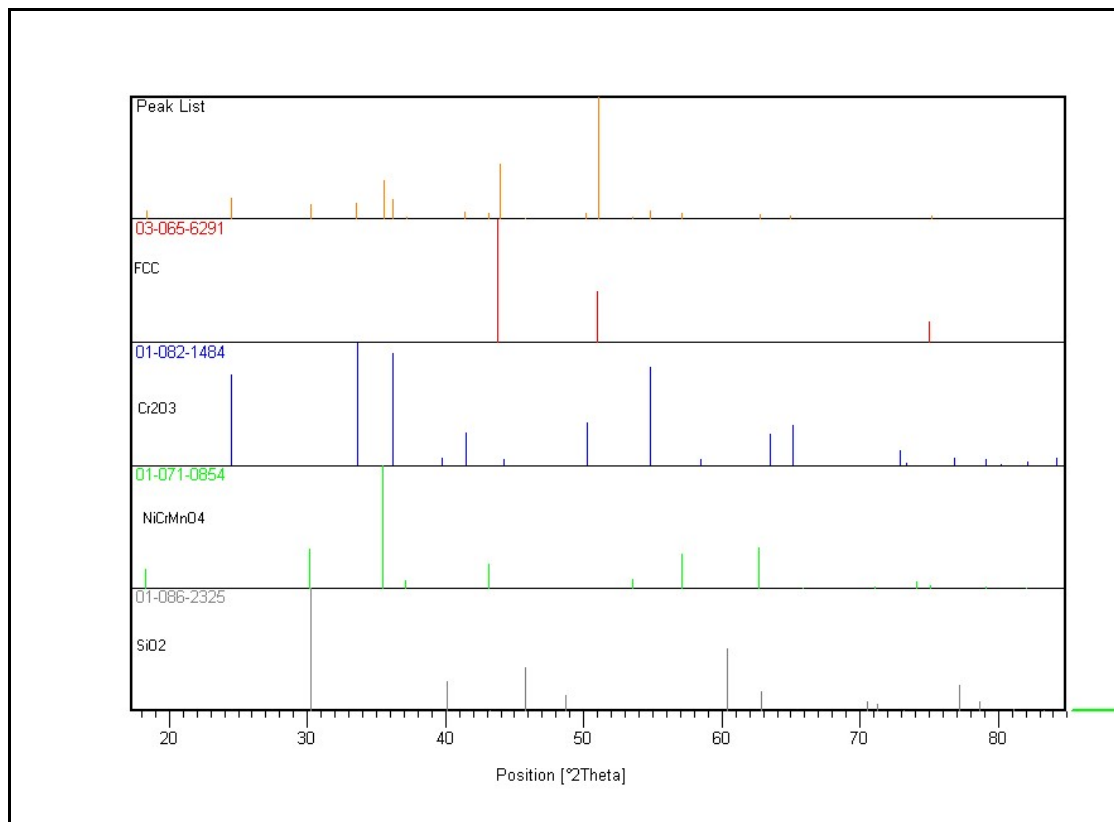
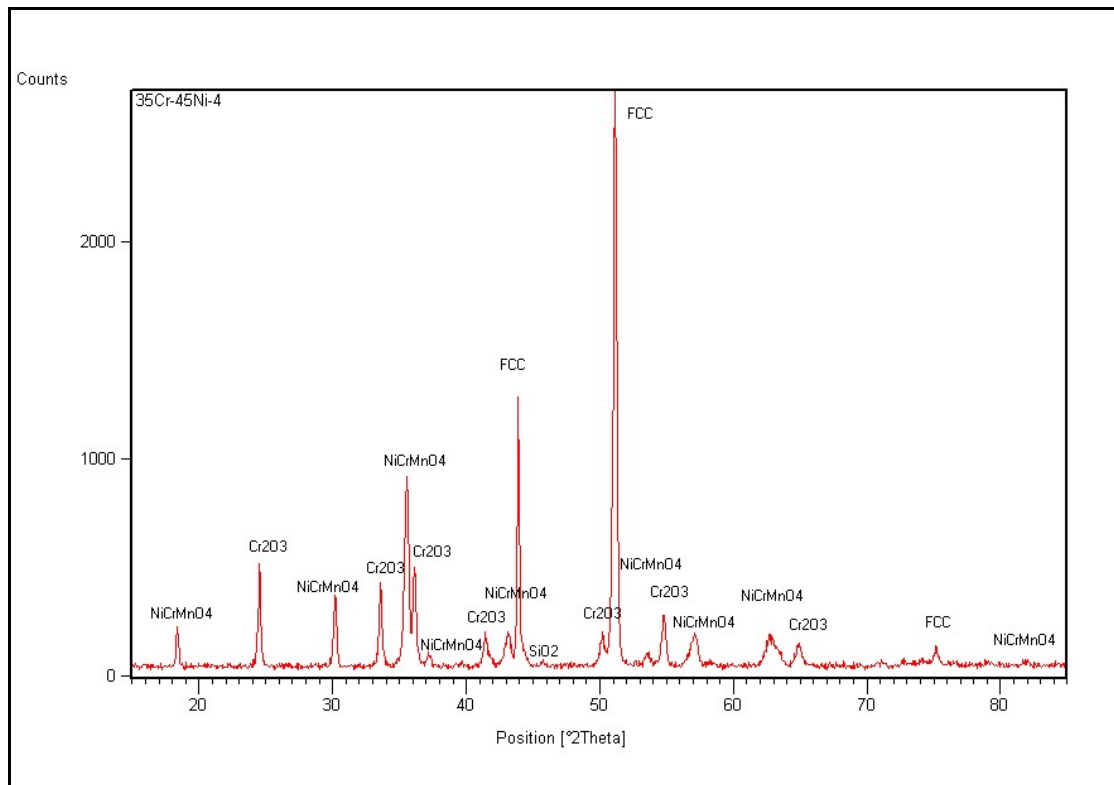
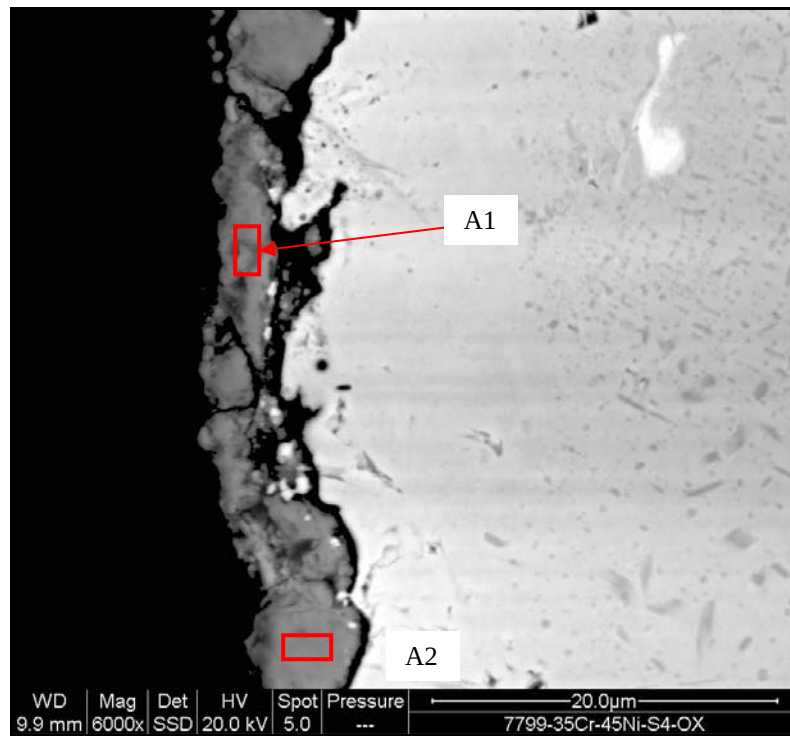
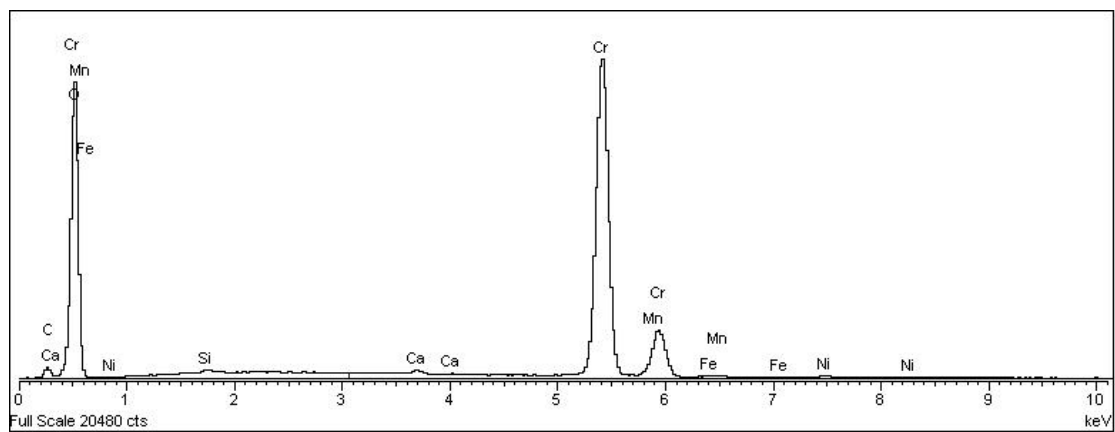


Figure 2.53 XRD pattern recorded from the alloy 35Cr-45Ni after exposure at 750°C.



A1



A2

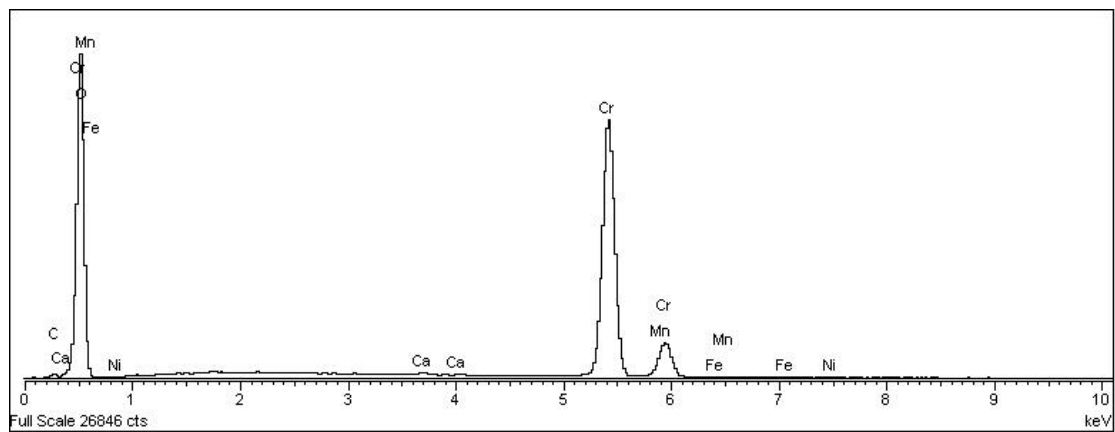


Figure 2.54 EDX analyses of the oxides formed on alloy 35Cr-45Ni after the 750°C experiment.

F Oxidation of Alloy 35Cr-45Ni at 850°C for 1000h

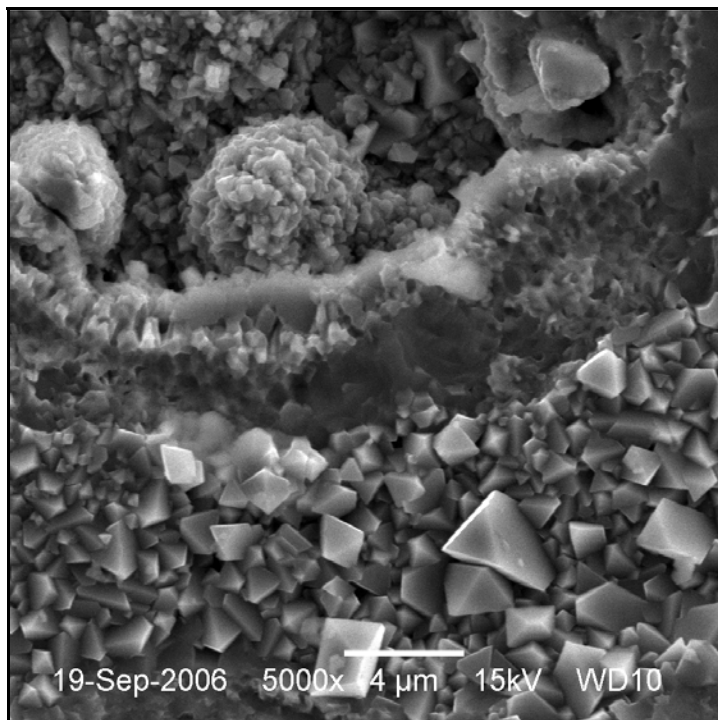
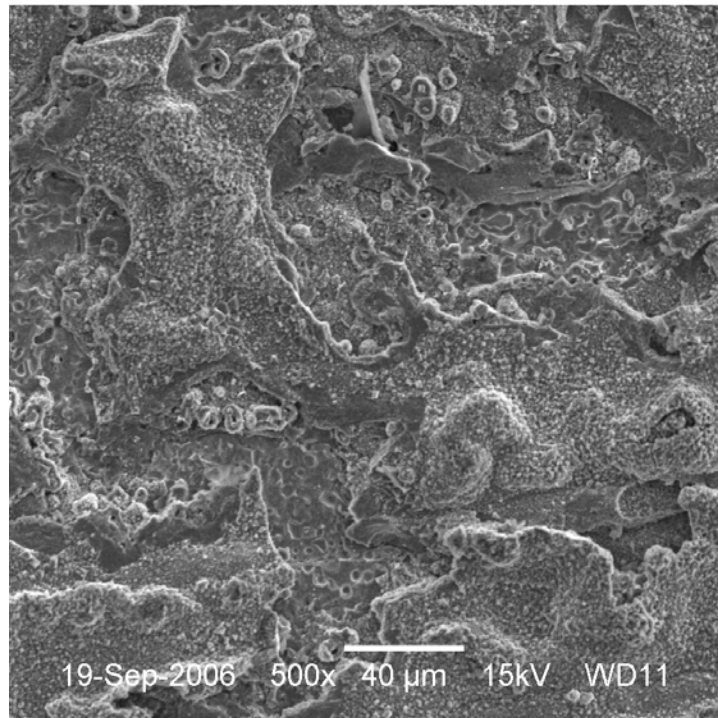


Figure 2.55 SEM/SE images of alloy 35Cr-45Ni surfaces exposed at 850°C.

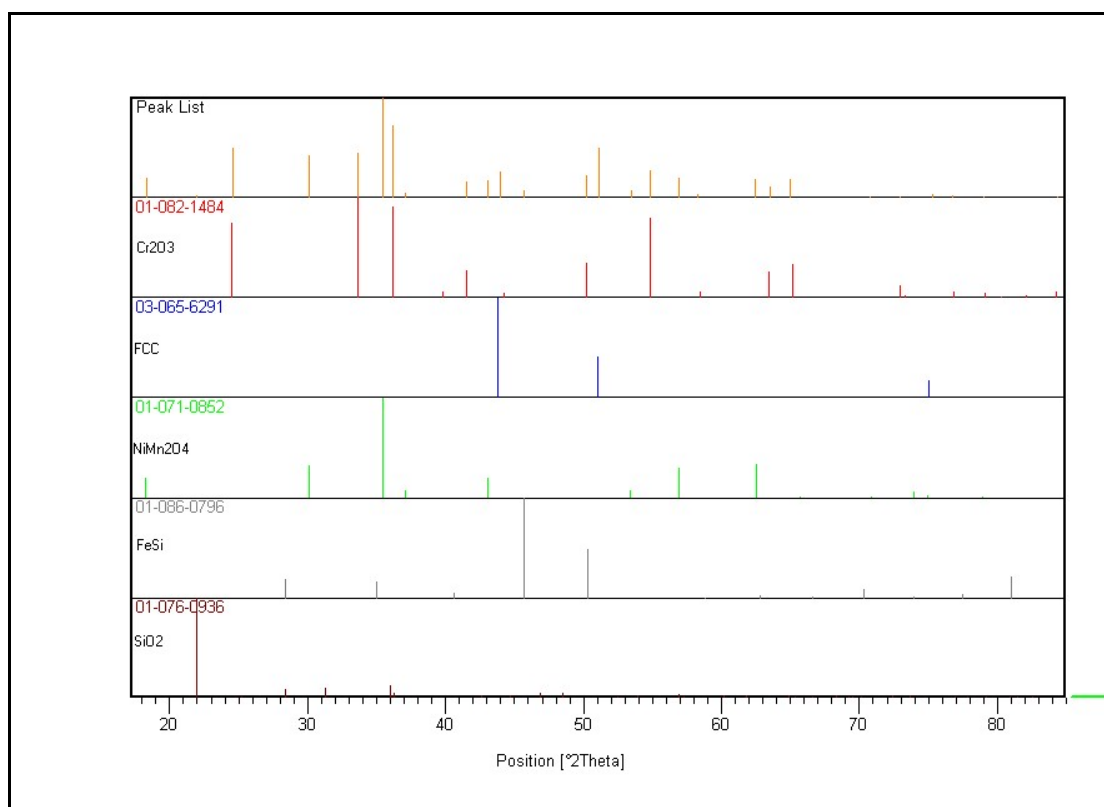
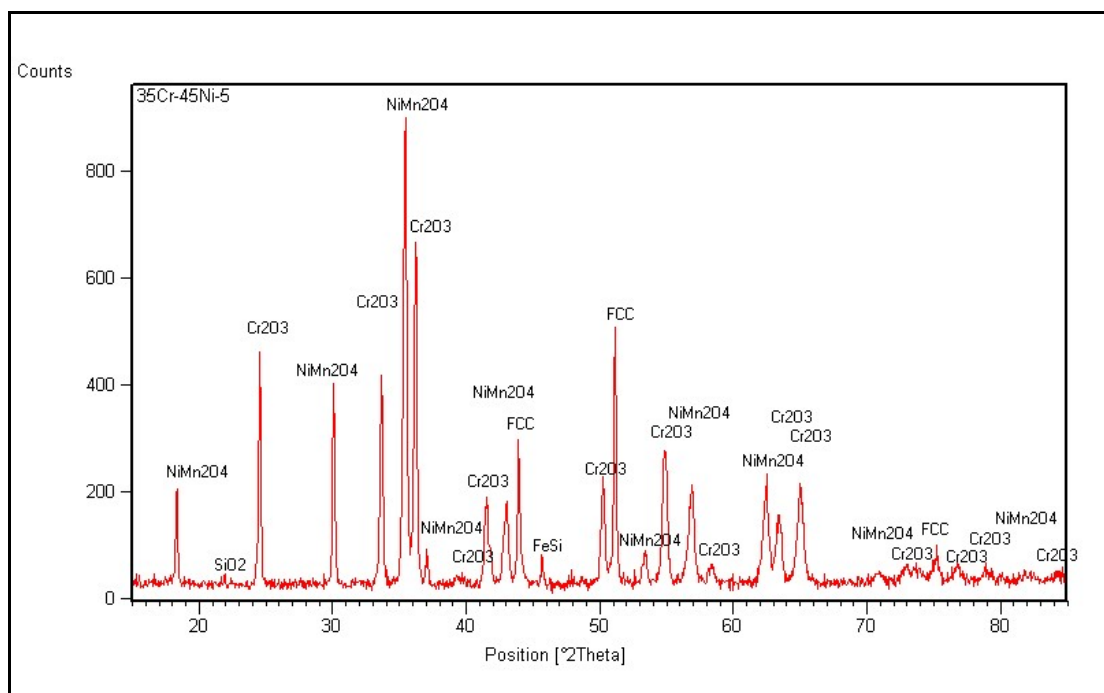
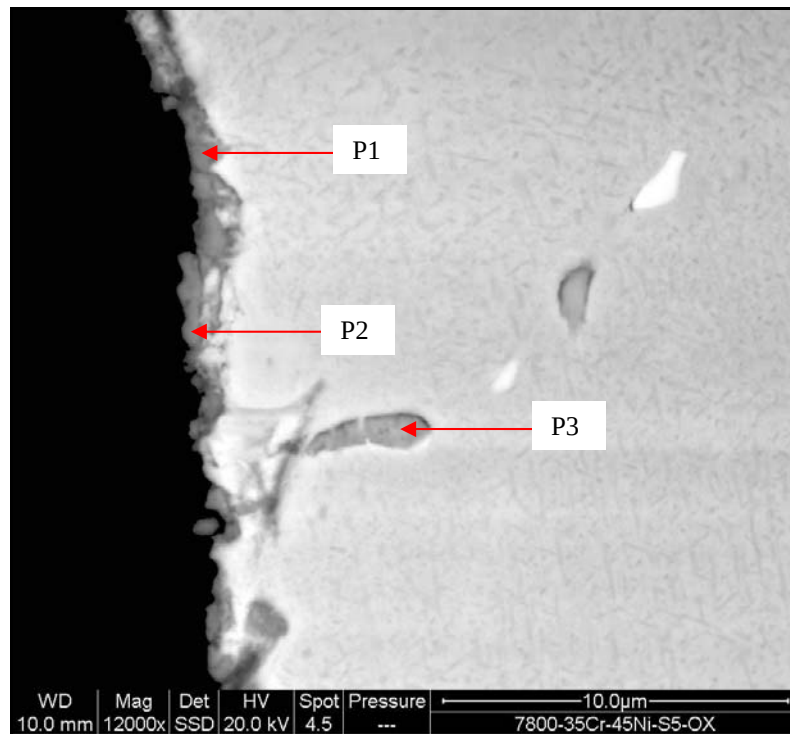
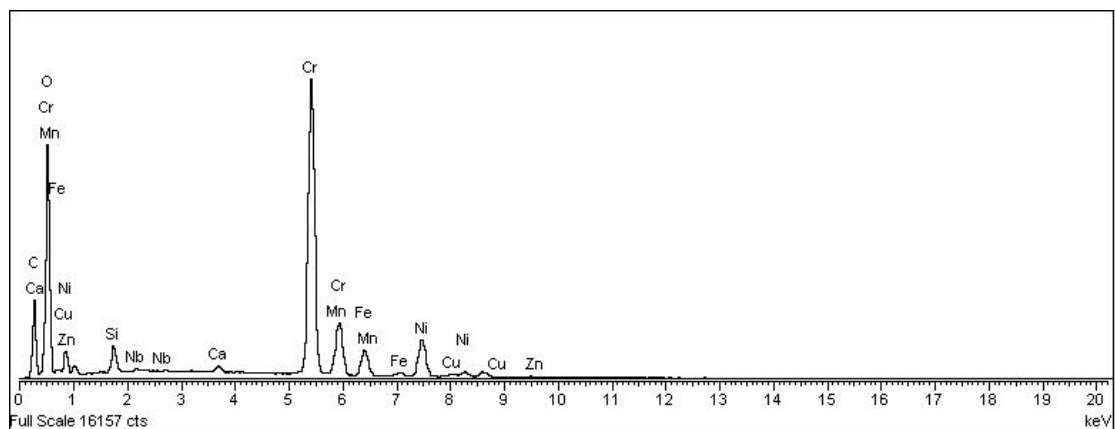


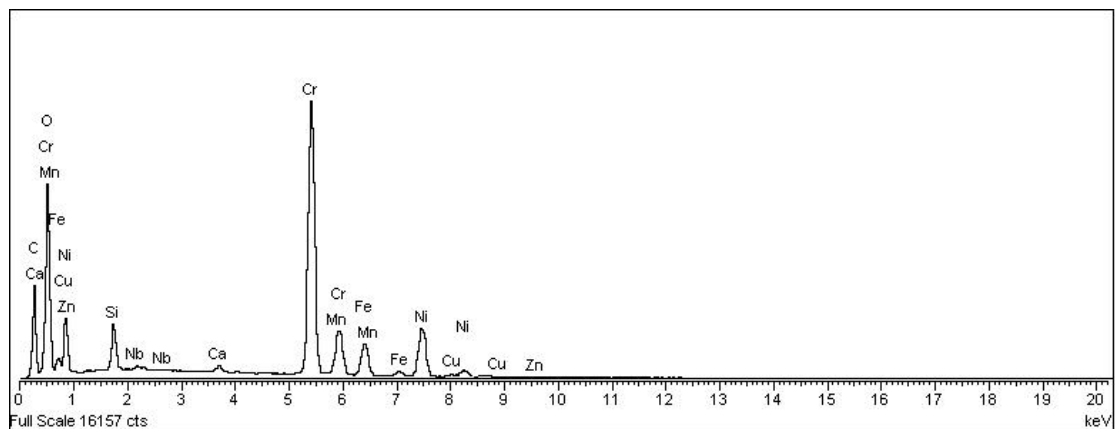
Figure 2.56 XRD pattern recorded from the alloy 35Cr-45Ni after exposure at 850°C.



P1



P2



continue...

P3

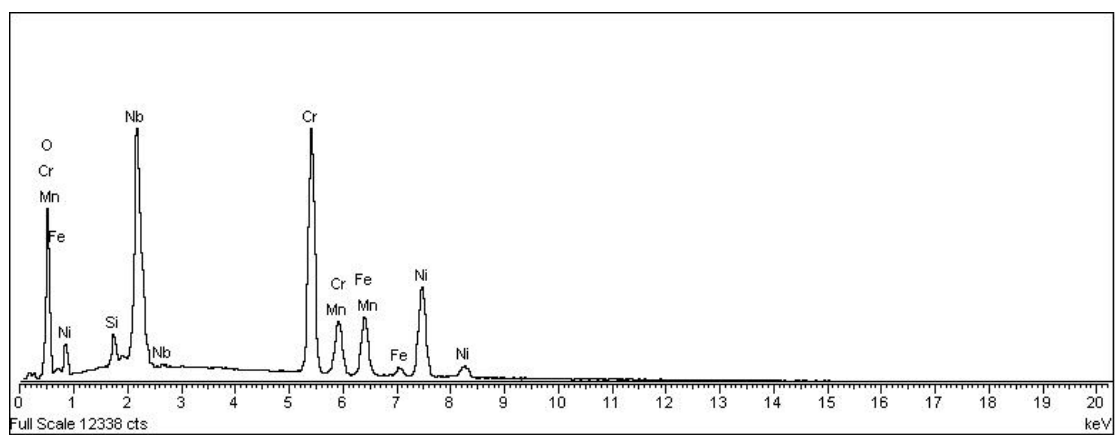


Figure 2.57 EDX analyses of the oxides formed on alloy 35Cr-45Ni after the 850°C experiment.

G Oxidation of Alloy UCX at 650°C for 1000h

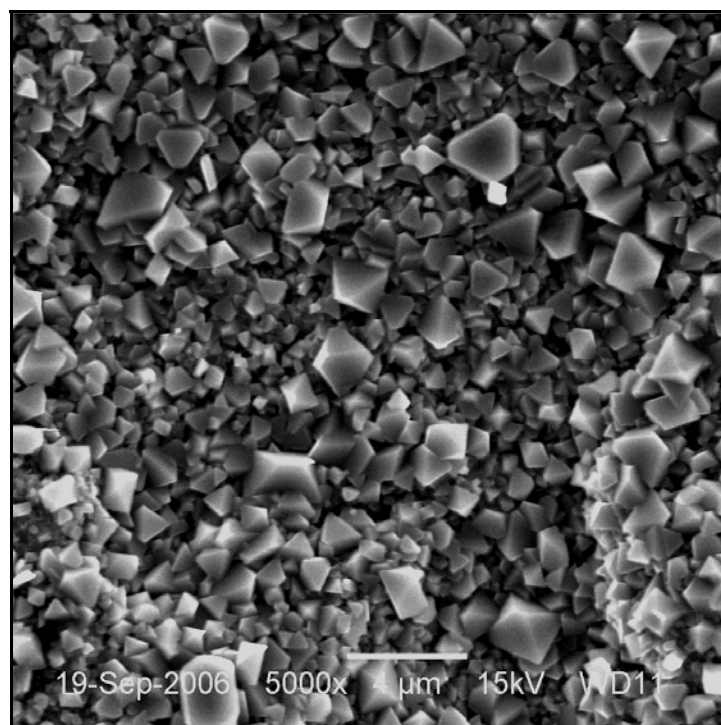
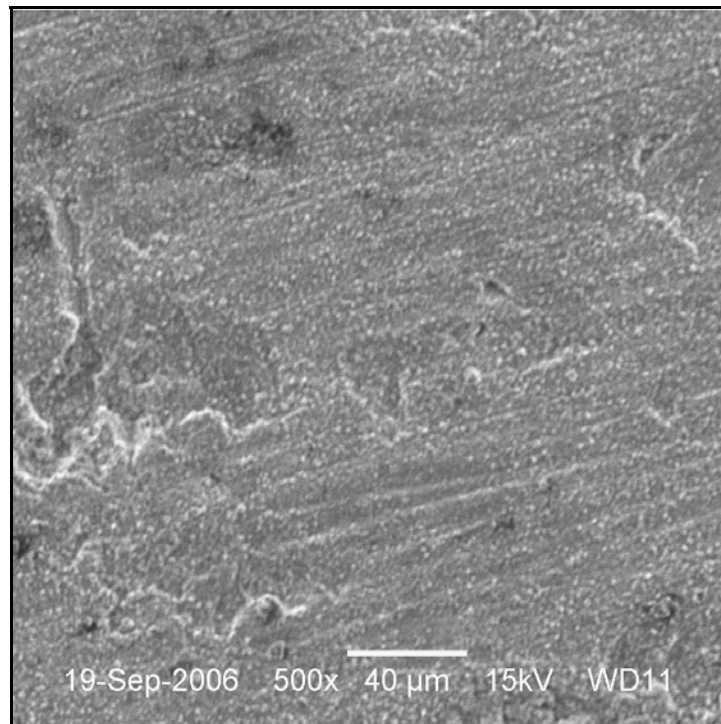


Figure 2.58 SEM/SE images of alloy UCX surfaces exposed at 650°C.

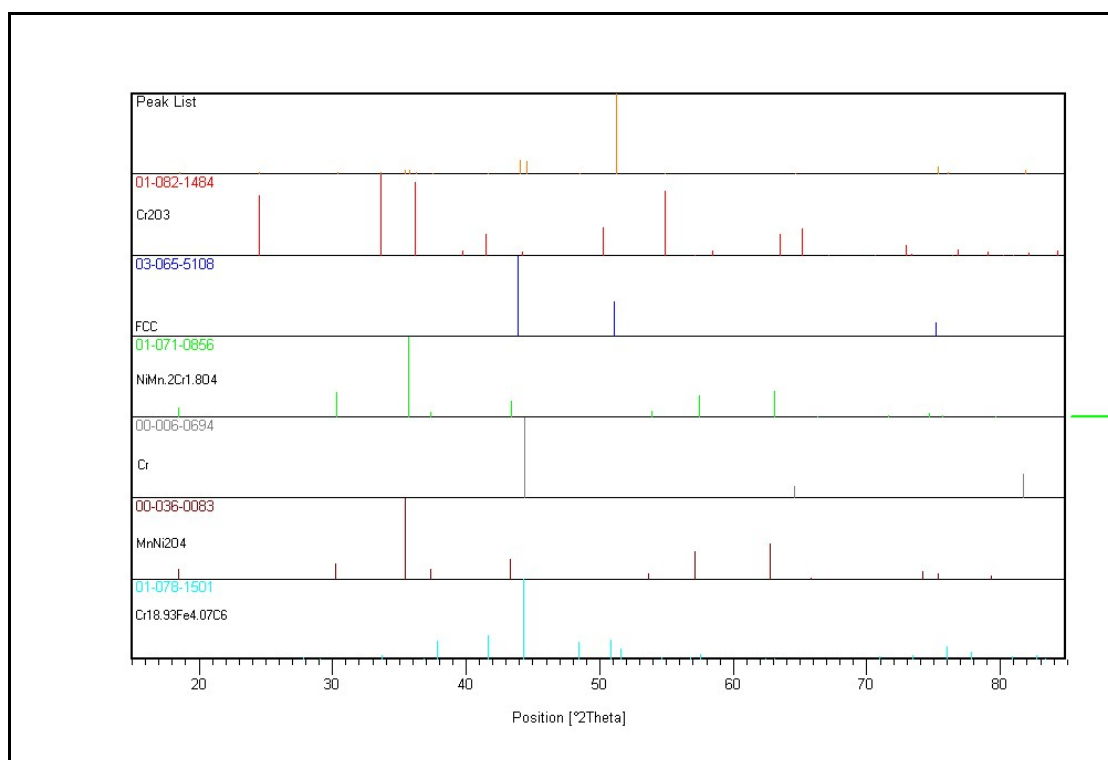
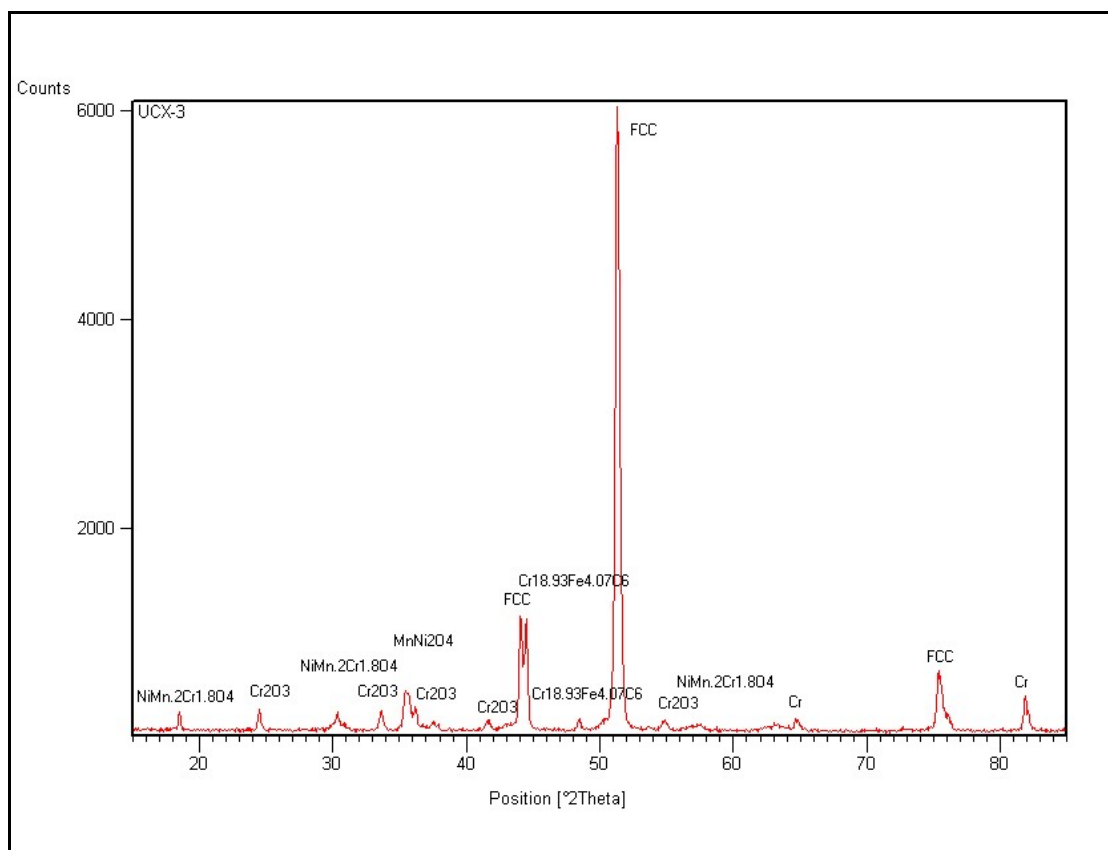
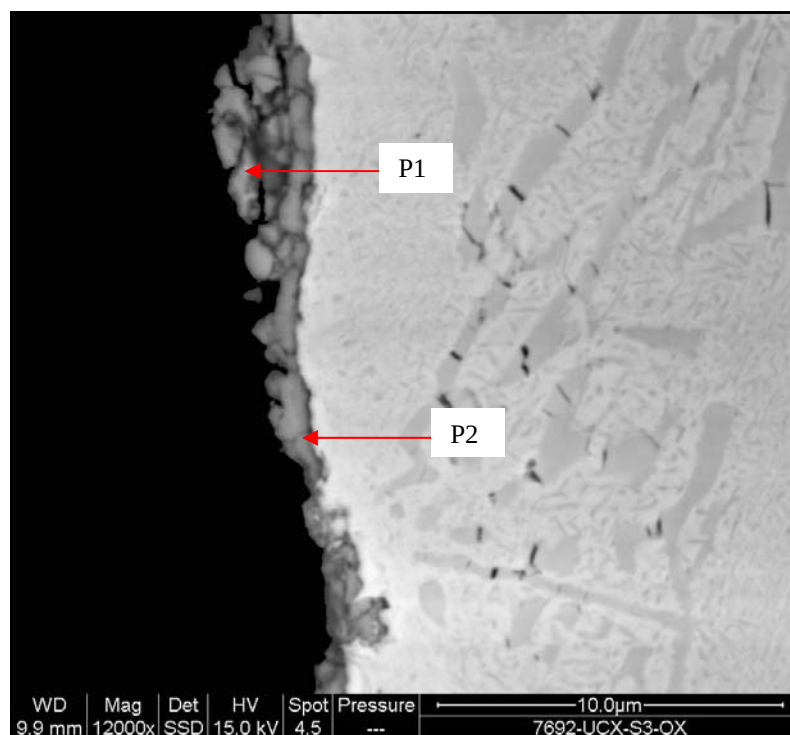
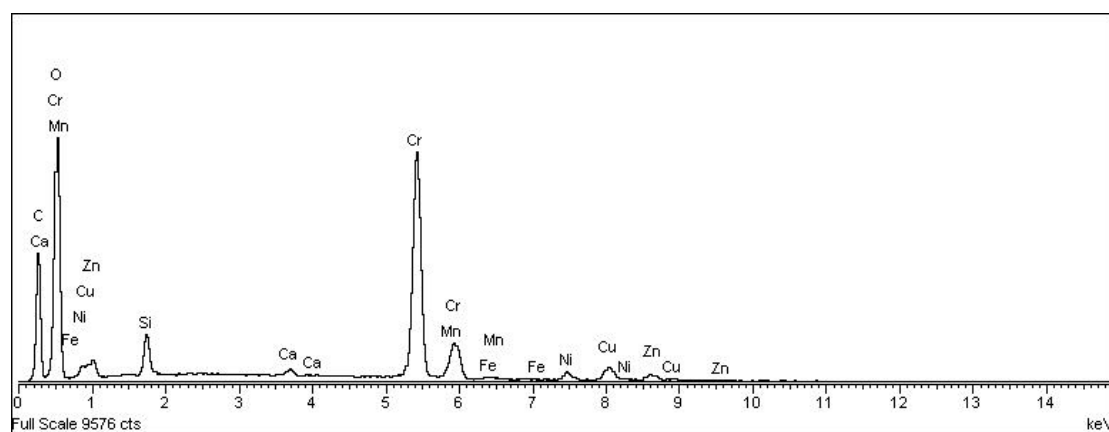


Figure 2.59 XRD pattern recorded from the alloy UCX after exposure at 650°C.



P1



P2

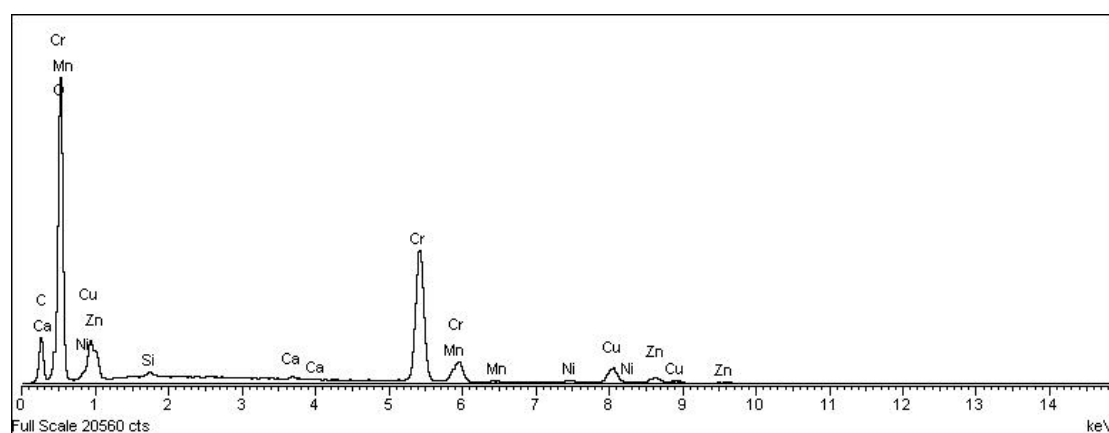


Figure 2.60 EDX analyses of the oxides formed on alloy UCX after the 650°C experiment.

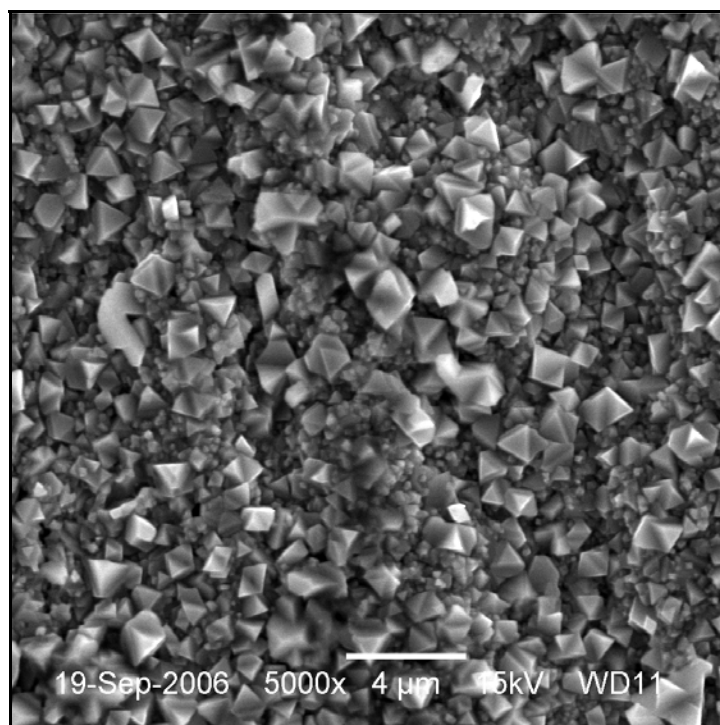
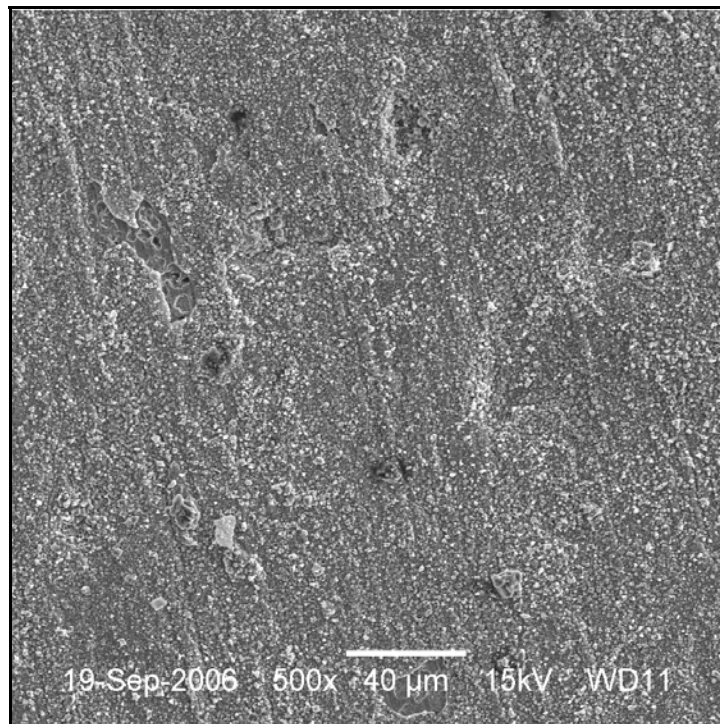


Figure 2.61 SEM/SE images of alloy UCX surfaces exposed at 850°C.

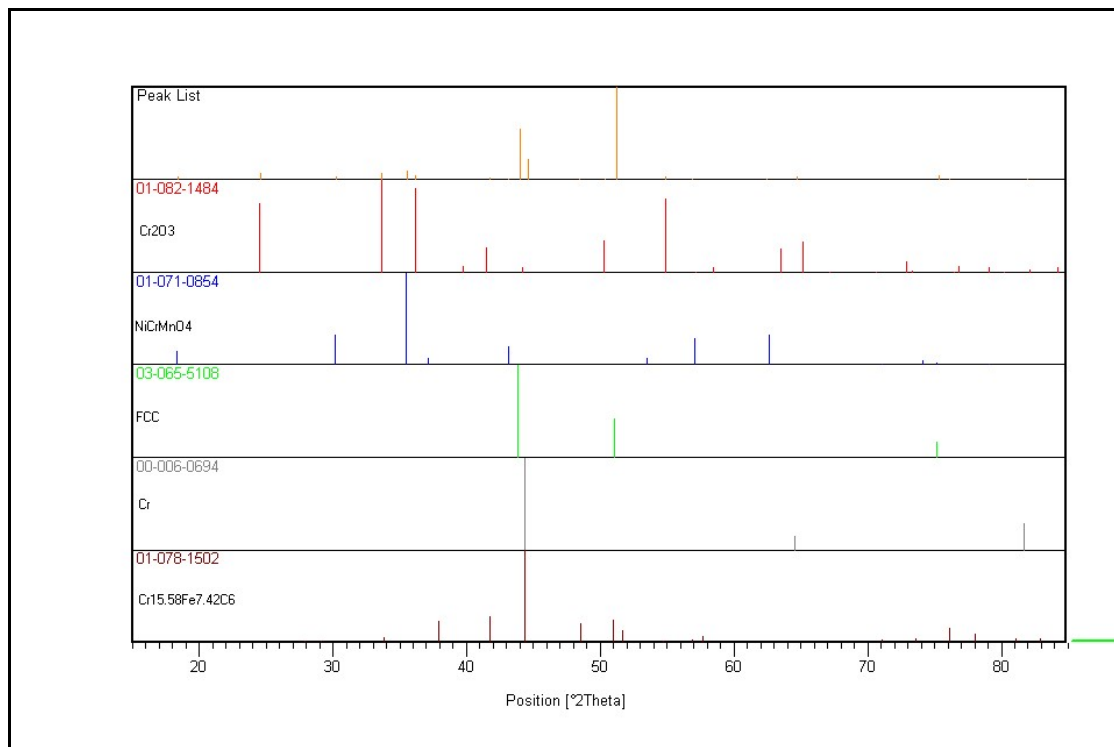
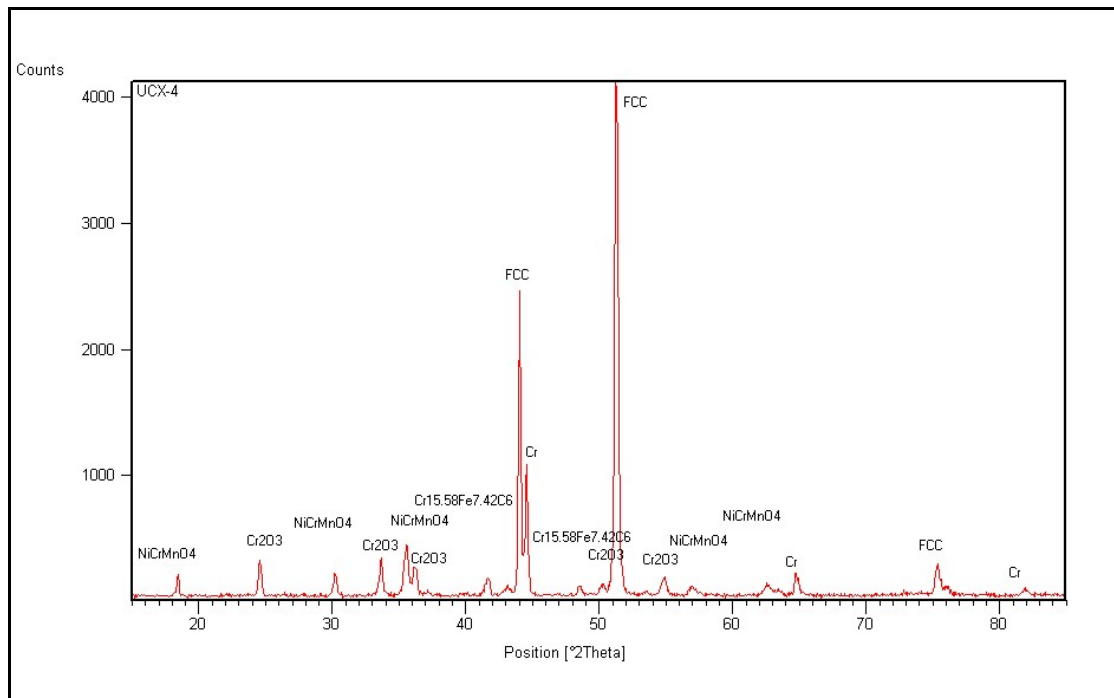
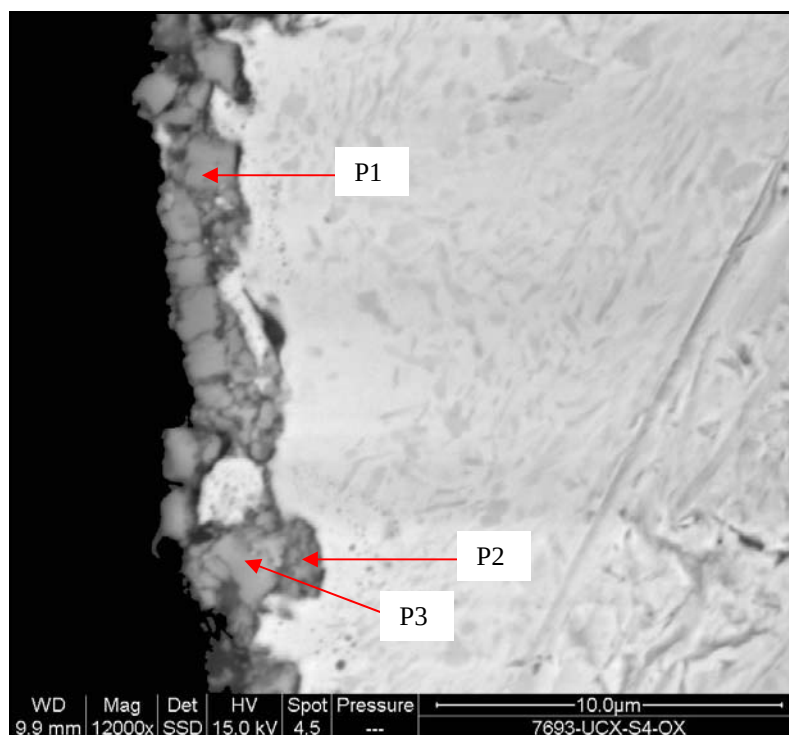
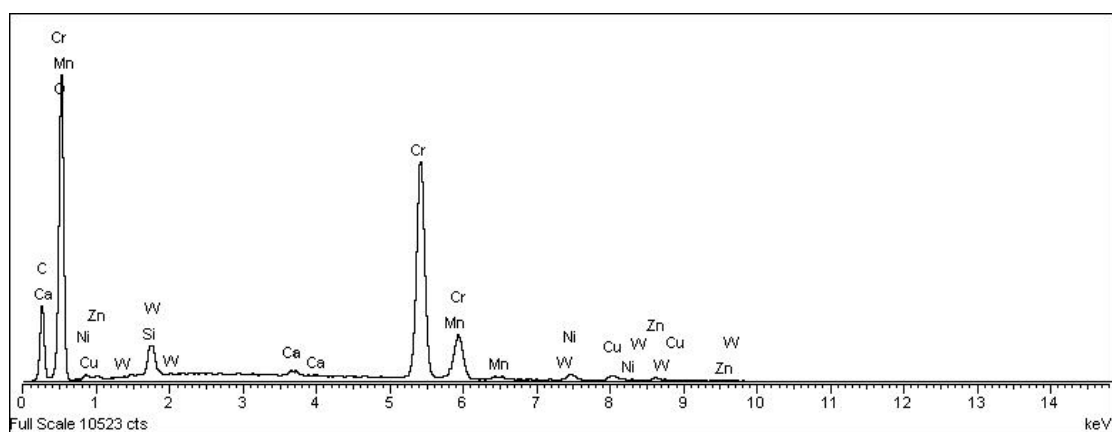


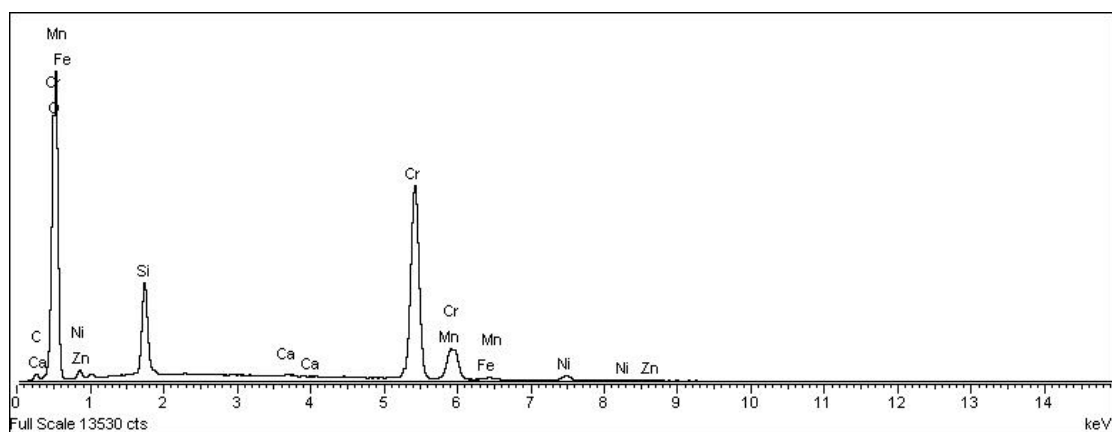
Figure 2.62 XRD pattern recorded from the alloy UCX after exposure at 750°C.



P1



P2



continue...

P3

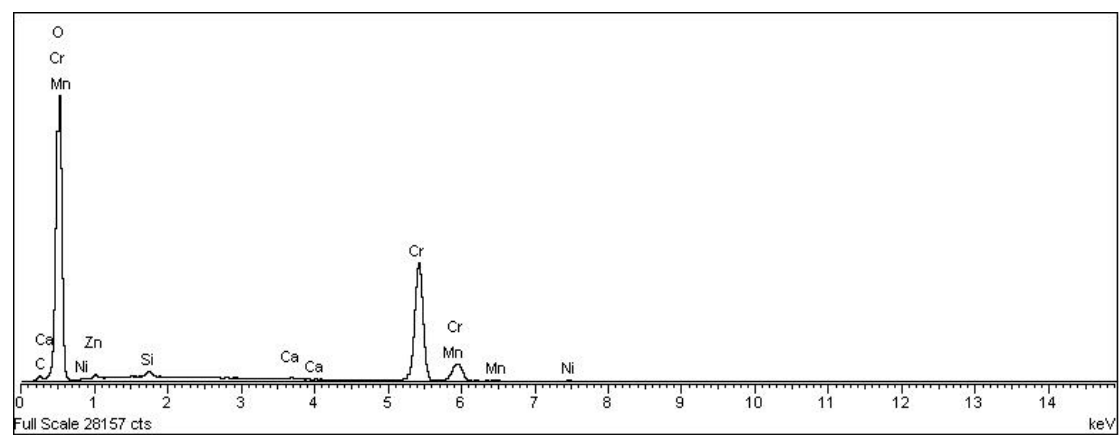


Figure 2.63 EDX analyses of the oxides formed on alloy UCX after the 750°C experiment.

I Oxidation of Alloy UCX at 850°C for 1000h

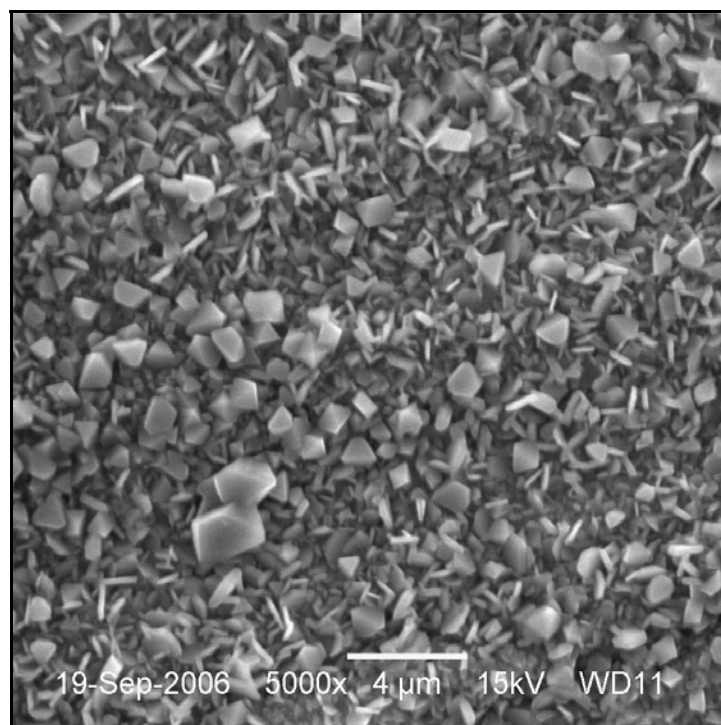
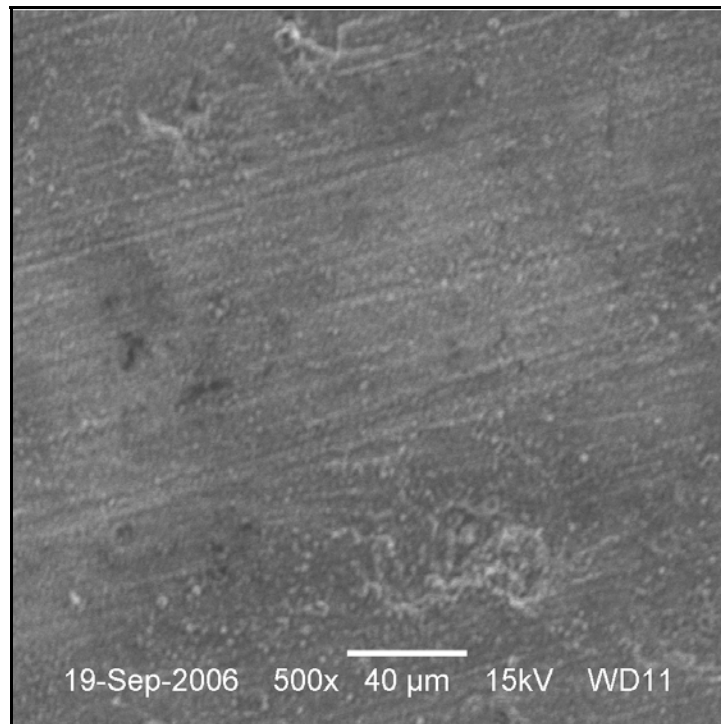


Figure 2.64 SEM/SE images of alloy UCX surfaces exposed at 850°C.

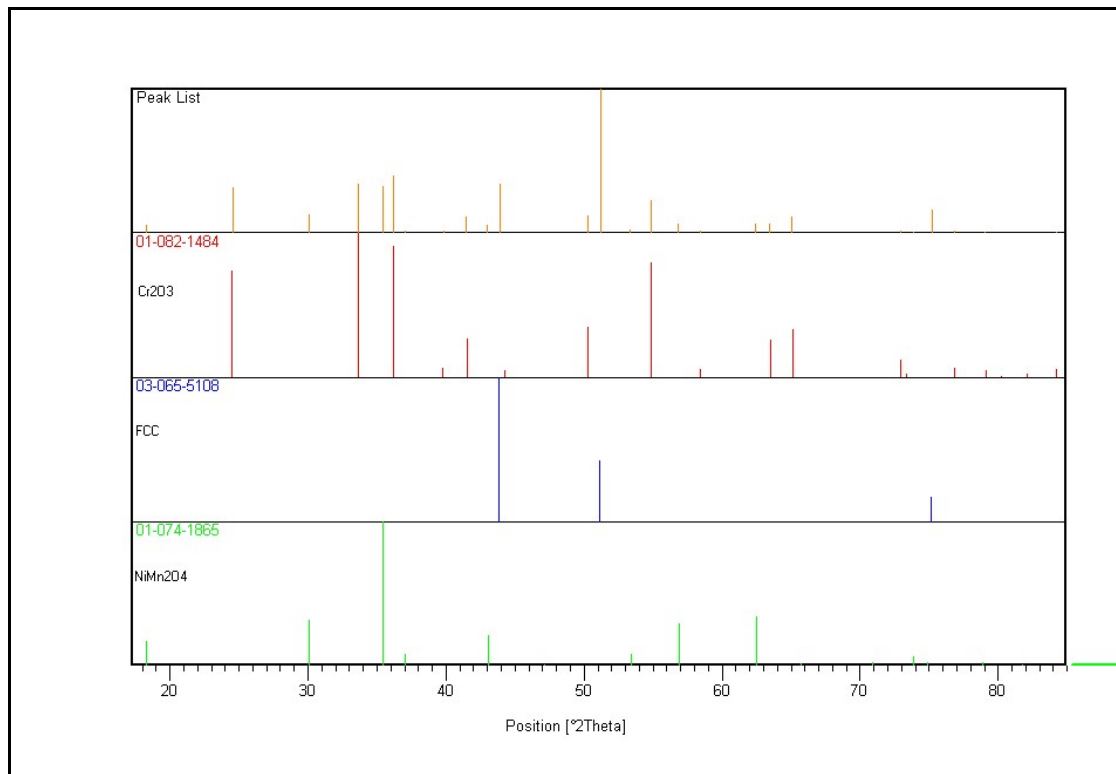
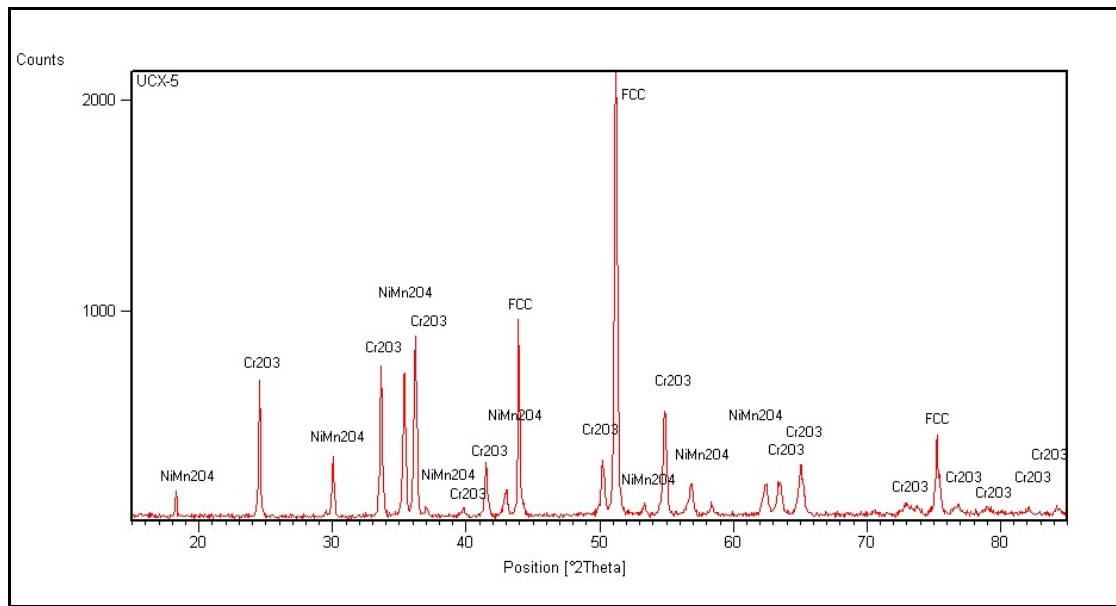
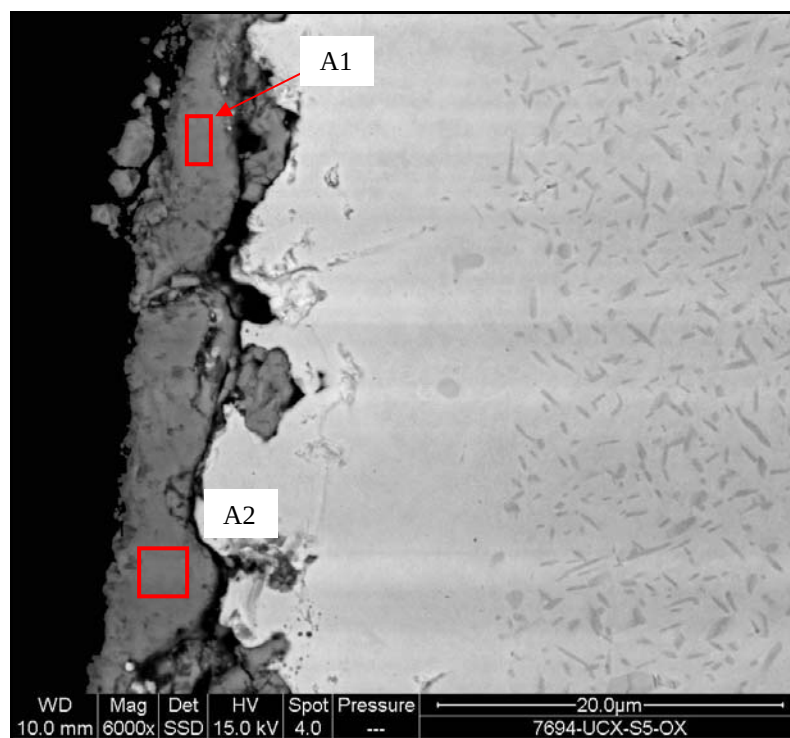
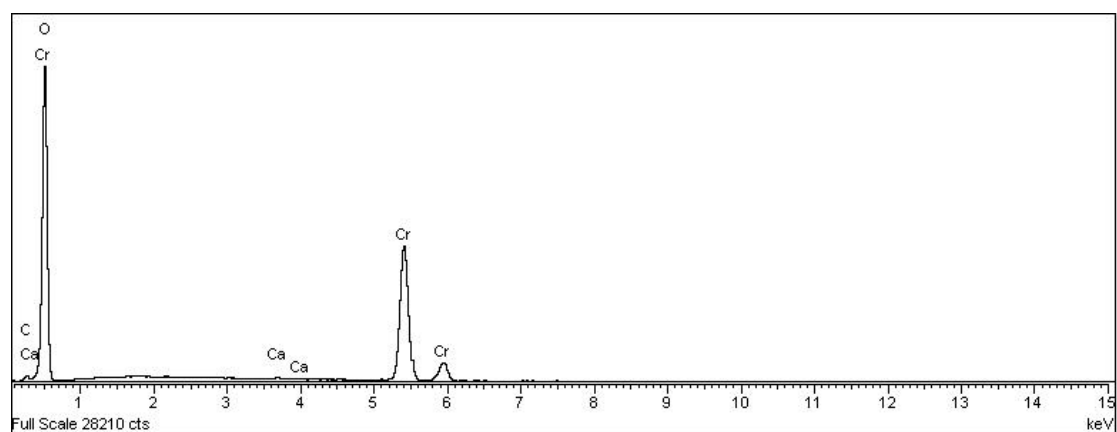


Figure 2.65 XRD pattern recorded from the alloy UCX after exposure at 850°C.



A1



A2

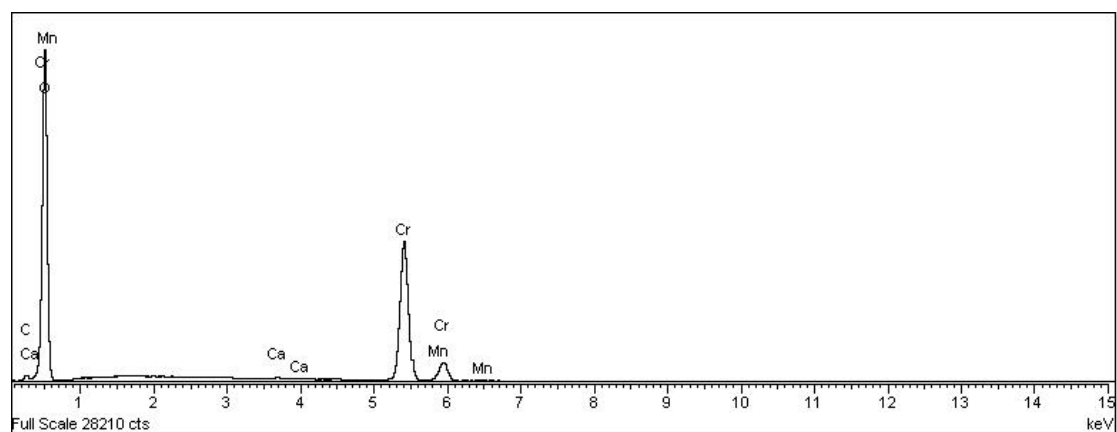


Figure 2.66 EDX analyses of the oxides formed on alloy UCX after the 850°C experiment.