A PRELIMINARY STUDY USING THE ENERGY DISPERSIVE X-RAYS (EDX) TECHNIQUE ON THE ELECTROLESS PLATED CERAMIC BODY

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ABSTRACT

The study of this research is focused on finding the optimum parameter of electroless plating. It is shown by the quality of the nickel coating. From this study, it is found that the best quality of coating is produced at the electroless plating parameter of 80°C and for the duration of soaking between 15-30 minutes However a few days after the electroless plating process has been done, it is found that the layer of electroless nickel plating is lost if the electroplating process is not done immediately after the electroless plating process. To investigate the loss of the nickel coating after the electroless plating process, the energy dispersive x-rays (EDX) technique is used. Results show that the nickel has penetrated into the ceramic through the capillary action, due to porosity. Therefore we suggest that the electroplating process should be done immediately after the electroless plating process. Basically, this research has proved that the clay ceramic body can be coated by nickel.

1.0 INTRODUCTION

Nickel coating on ceramic body is a new study compared to nickel coating on plastic. On the other hand nickel coating on metals is widely used world wide to prevent corrosion and to upgrade the appearance. In addition of upgrading the appearance of ceramic by nickel coating, the strength and the ductility of ceramic is believed to improve. This will increase the application of ceramic materials.In the preliminary study of nickel coating on clay ceramic body, prior to the electroplating process, the electroless nickel plating as discussed by Brenner and Riddell ([1,2,3]) has been utilized to deposit Ni-P coating onto the surface of clay based ceramic bodies. It is also required that the sensitization step (S_nCl_2) is followed by the nucleation step (P_dCl_2) before the electroless plating stage begins. The study of this research is focused on finding the optimum parameter of electroless plating. It is shown by the quality of the nickel coating. In this study the temperature parameter of electroless plating is changed at 50°C, 65°C, 80°C and 95°C. At every temperature the duration of electroless plating are 10 minutes, 15 minutes, 30 minutes and 60 minutes. There are sixteen samples for every test. The electroplating process should be done immediately after the electroless plating process. The parameters in the electroplating process were fixed at current of 1 Ampere and plating time of twenty minutes for each sample.

During this study, it is found that a few days after the electroless plating process has been done and the sample is kept in the incubator with silicon gel as the wet absorber, the nickel coat on the surface of the sample is loss. One may suggest that the loss is due to the evaporation of the nickel into air, which is quite impossible since nickel has a high density (28 grams). The other suggestion is that the nickel has penetrated through the capillary action since ceramic has high porosity. To investigate the loss of the coats, the energy dispersive x-rays (EDX) technique is used.

2.0 EXPERIMENTAL PROCEDURES

2.1 SAMPLE PREPARATION

The specimen used was a local clay body with 8% moisture formed at a pressure of 8 tons and sintered at 950°C for one hour. Figure 1 shows the flow chart for the experimental procedures. Table i shows the electroless nickel plating parameter. The range of the water bath is from 50 to 95°C and plating time between 10 to 60 minutes. The parameters in the electroplating process were fixed at current of 1 Ampere and plating time of twenty minutes for each sample. Table ii shows the Watts bath formulation used in this study.

2.2 TESTING

The energy dispersive x-ray technique is performed at the surface, below the surface known as spot 1 and at the middle of the samples known as spot 2. This is shown schematically in Figure 2.

3.0 RESULTS AND DISCUSSION

Results show that there is nickel in the sample's body and not on the surface of the samples. It is believed that the nickel from the electroless plating process has penetrated into the sample's body through the capillary action, which resulted from the high porosity in the ceramic. As a comparison, a control sample that is not coated using the electroless plating procedure, does not contain nickel, stannus and palladium that are used during the electroless plating process and the pre-electroless plating process.

Analysis shows that sample has some aluminium and silicon element, and a few of magnesium, ferum and kalium. The aluminium and silicon elements are resulted from the metakaolinite phase ($AL_2O_3.2SiO_2$) and the mullite ($3Al_2O_3.2SiO_2$) that are form during the firing process of the clay. The existence of ferum resulted in the red colour in the fireclay. From the EDX study, it shows that for all the electroless plating samples, the concentration of nickel decreases as the depth of the sample increase. The evidence of the changes of concentrations is shown in figure 3 to 14. The rate of nickel penetration into the ceramic body is very low because the atomic mass unit of nickel is high (28 grams). Therefore the concentration of nickel is lower at the center of the sample compared to the surface and the bottom of the sample. The The penetration that occurs is due to the existence of the pores the penetration is also related to the number and size of the pores that are affected by the process parameters, namely the mixing, the forming and the firing.

The contents of palladium in the nucleation solution are very low that is 0.25 grams per liter. Therefore, its existence is not detected using EDX. It is believed that the stannus penetration into the sample occurred due to the atomic mass unit for stannus is high (50 grams) compared to the atomic mass unit for nickel (28 grams) therefore it is difficult to move through the pores in the body.

4.0 CONCLUSION

From the result of the energy dispersive x-rays (EDX) technique, the existence of nickel is identified and it is believed that the nickel has penetrated through the capillary action into the sample. We suggest that the electroplating process should be done immediately after the electroless plating process. This is to overcome the loss of the nickel coats on the surface of the ceramic body due to the penetration of nickel into the ceramic body.

REFERENCES

Baharuddin Wanik; 1984, *Kajian Penyaduran Nikel Ke Atas Plastik ABS,* Final Year Project, Pusat Pengajian Sains Kimia, Universiti Sains Malaysia.

Anon, V., Mar-Apr 1997, *Low cost Thermal Barrier Metal and Ceramic Matrix Co,, Composite Coatings Made Electro chemically*, Materials technology 12(2) pg 52-54.

Dennis, J.K. & such, T.E., 1986, *Nickel and Chromium Plating*, Butterworth & Co (Publishers) Ltd. UK

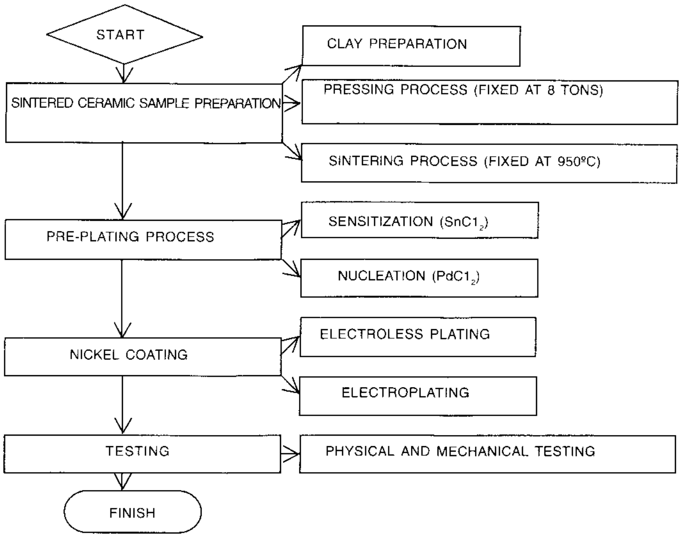

Figure 1. The flow chart for experimental procedures

Table ii. Concentration of ingredients of Watts bath

$NiSO_4.5H_2O:NiCl_2.6H_2O:H_3BO_3$	80 : 11 : 9
Distilled water	2 liters

$NiSO_4.6H_2O:Na_3C_6H_5O_7.2H_2O:NaH_2PO_2.H_2O$	11 : 10 : 4
Distilled water	986.00 ml
Bath pH (pH modifier – H_2SO_4)	4.30 (fixed)
Bath temperature	(50, 65, 80 and 95)°C
Plating time	(10, 15, 30 and 60) minutes

Table i. Electroless Nickel Plating Parameters

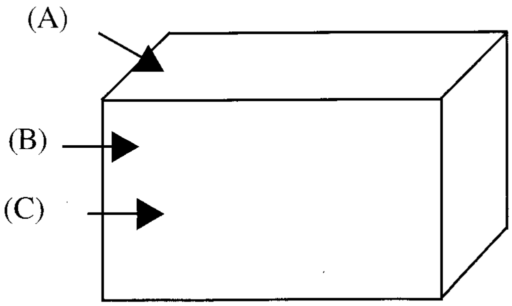

Figure 2. A Schematic showing the spots where EDX are performed. (A) is a spot on the surface of the sample, (B) is a spot below the surface of the sample (spot 1) and (C) is a spot in the middle of the sample (spot2).

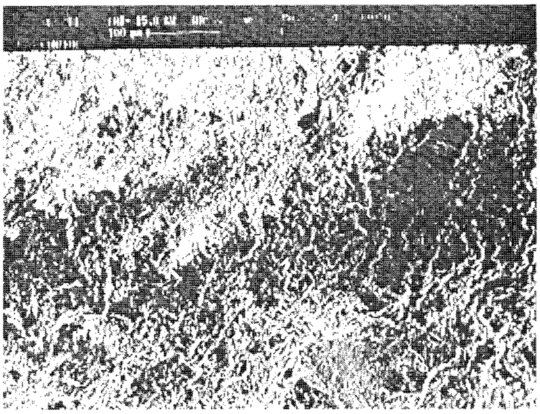

Figure 3. EDX on the surface of the uncoated ceramic sample

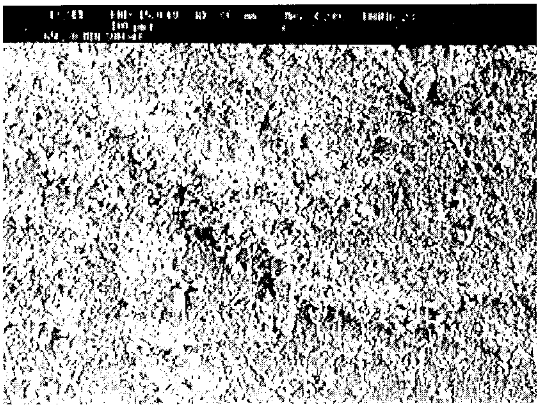

Figure 5. EDX on the surface of ceramic sample coated by electroless plating process at (65°C, 30 minutes)

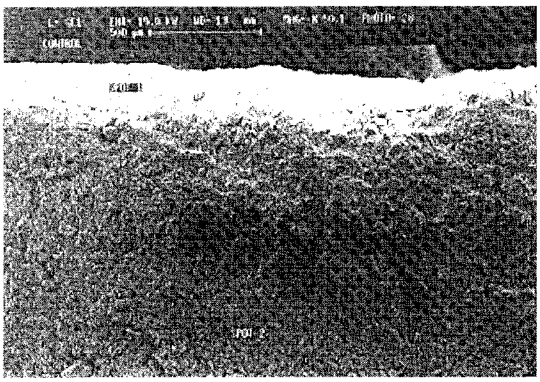

Figure 3. EDX on spot 1 and spot 2 of the uncoated ceramic sample

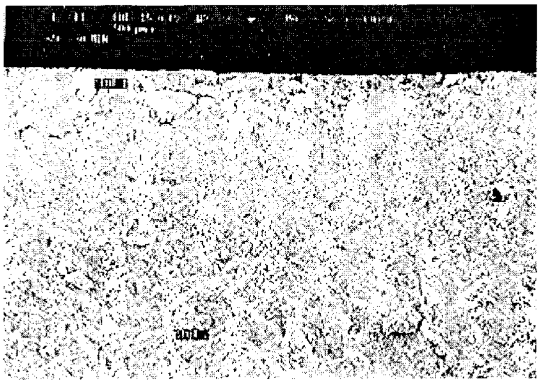

Figure 5. EDX on spot 1 and spot 2 of ceramic sample coated by electroless plating process at (65°C, 30 minutes)

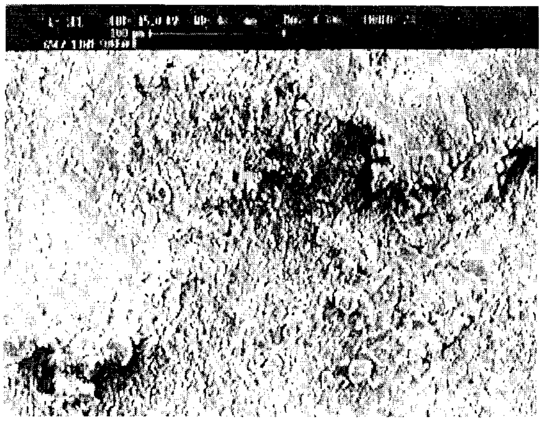

Figure 7. EDX on the surface of ceramic sample coated by electroless plating process at (65°C, 60 minutes)

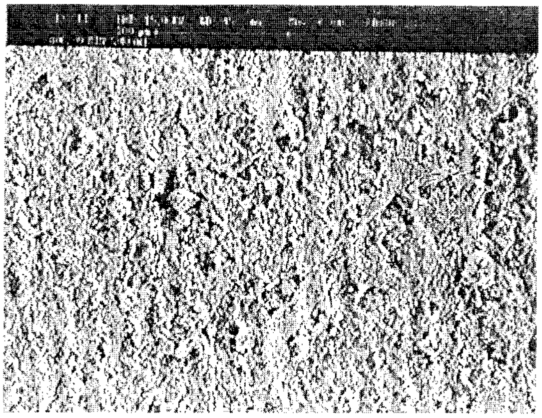

Figure 9. EDX on the surface of ceramic sample coated by electroless plating process at (80°C, 30 minutes)

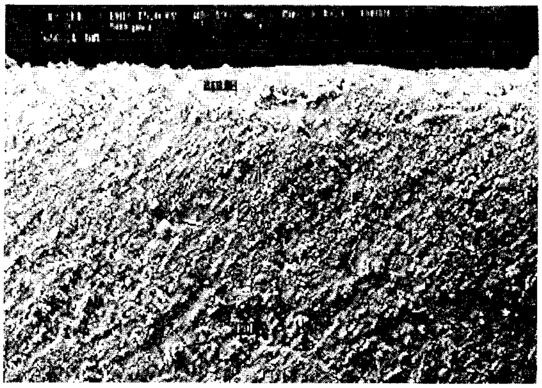

Figure 8. EDX on spot 1 and spot 2 of ceramic sample coated by electroless plating process at (65°C, 30 minutes)

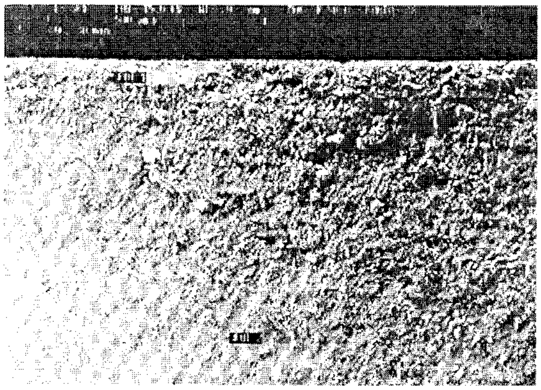

Figure 8. EDX on spot 1 and spot 2 of ceramic sample coated by electroless plating process at (80°C, 30 minutes)

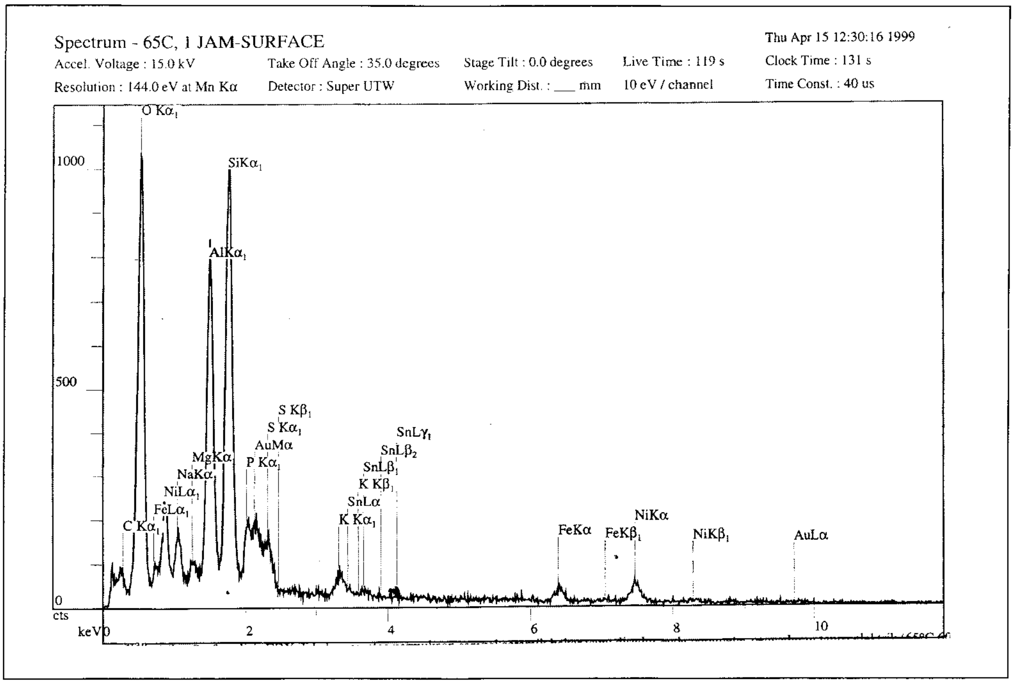

Figure 11a. EDX analysis at the surface of a control sample

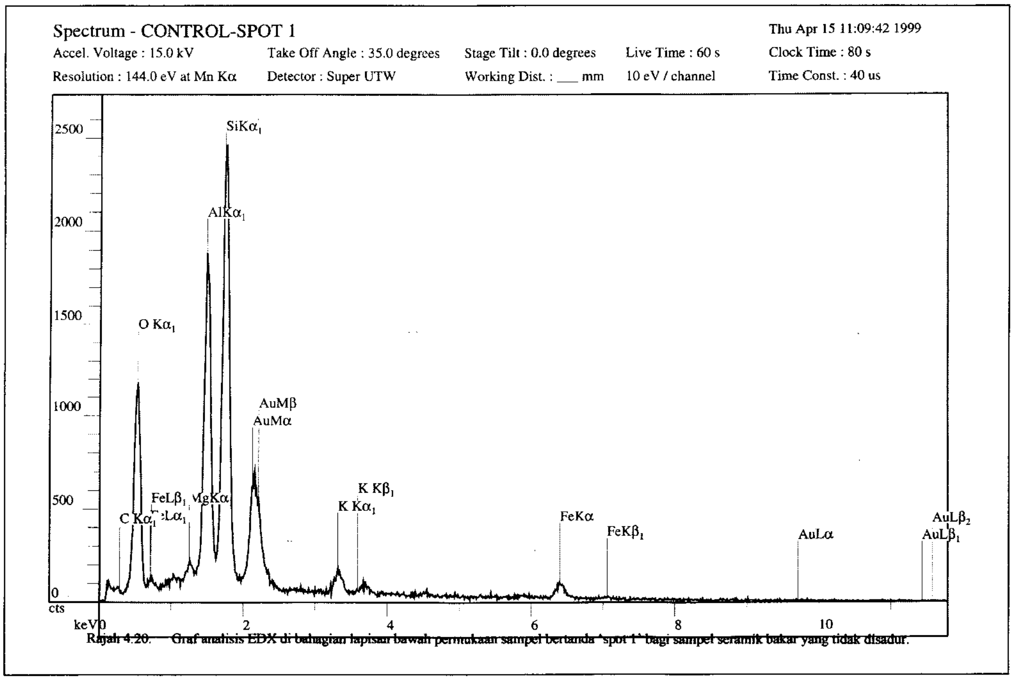

Figure 11b. EDX at spot 1 of a control sample

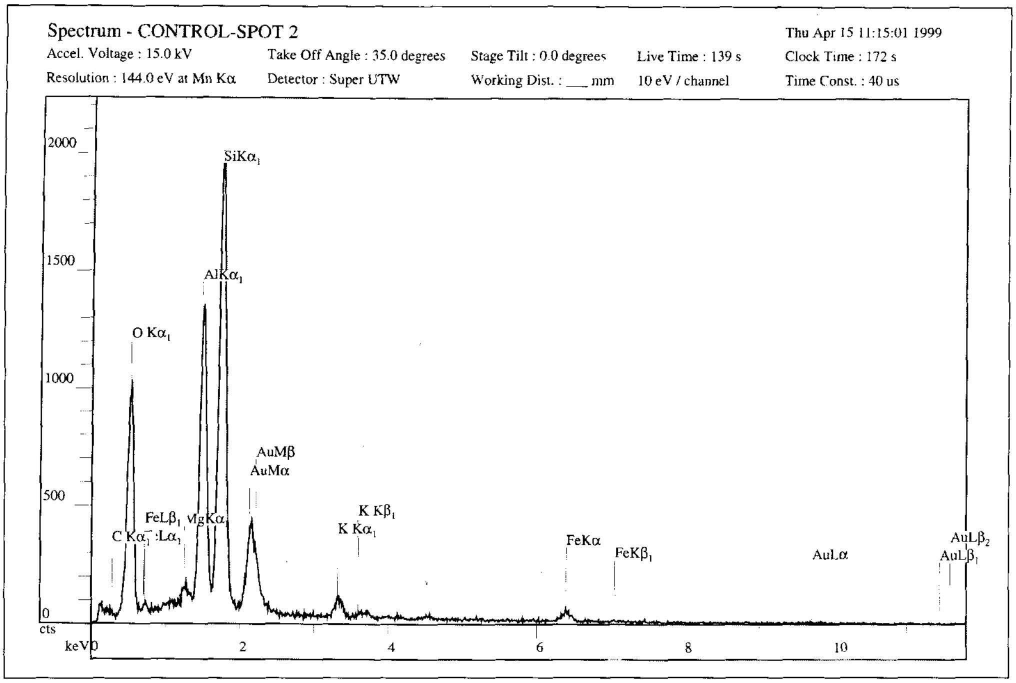

Figure 11c. EDX at spot 2 of a control sample

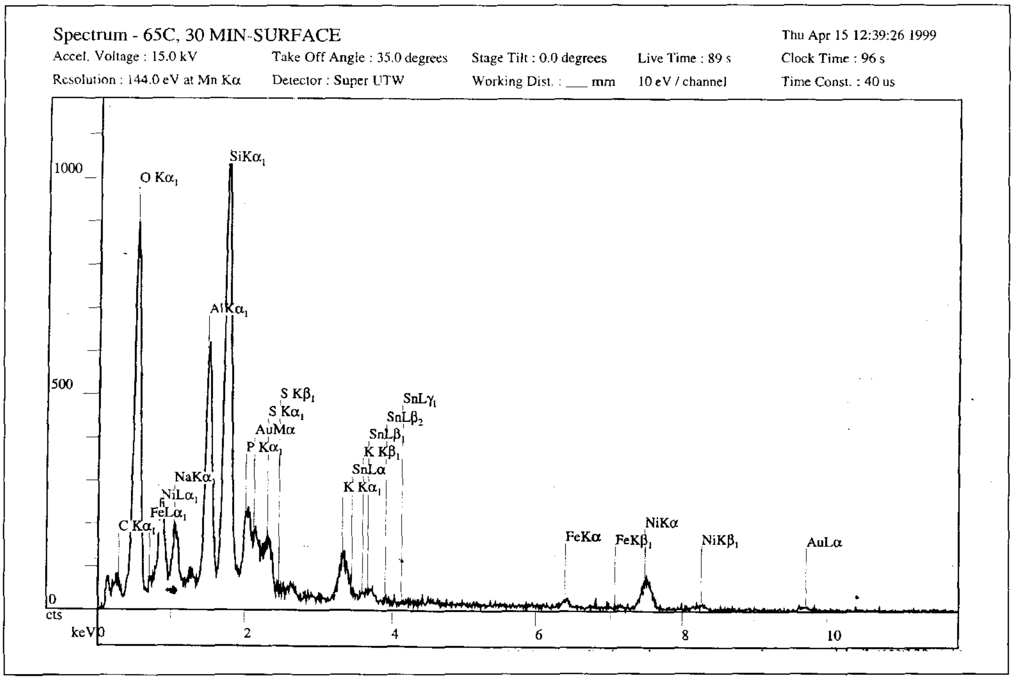

Figure 12a. EDX at surface of sample (65°C, 30 minutes)

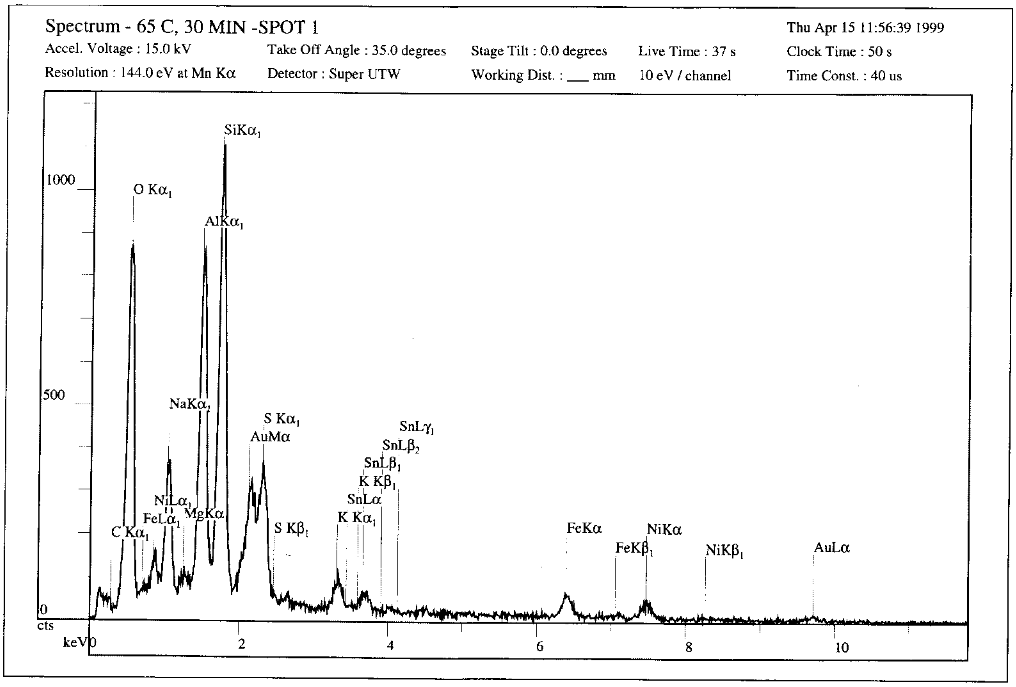

Figure 12b. EDX at spot 1 of a sample (65°C, 30 minutes)

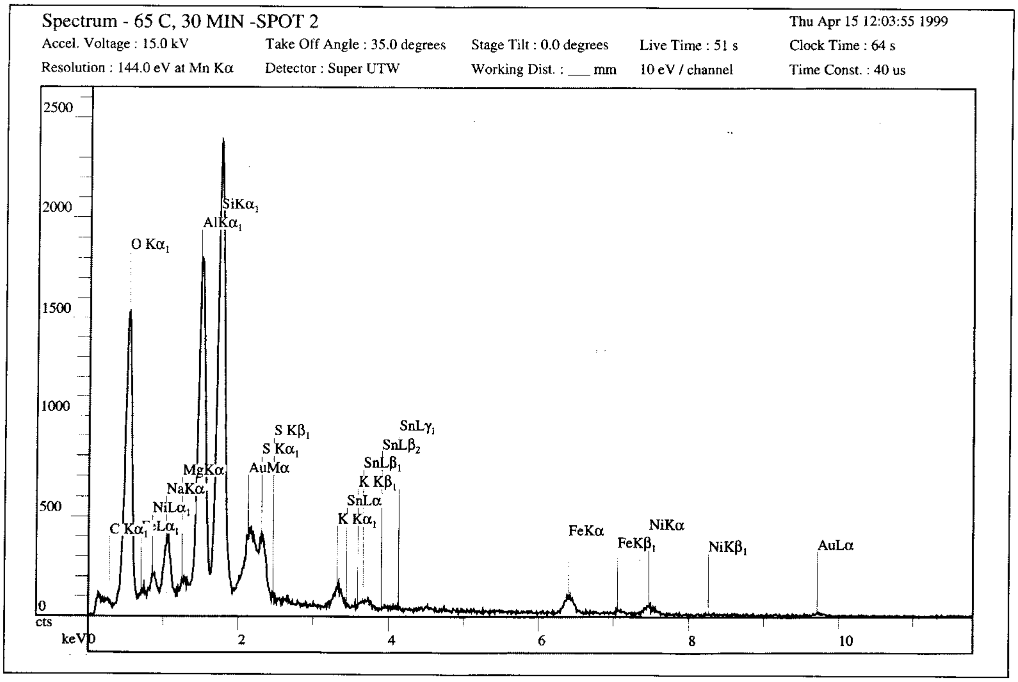

Figure 12c. EDX at spot 2 of sample (65°C, 30 minutes)

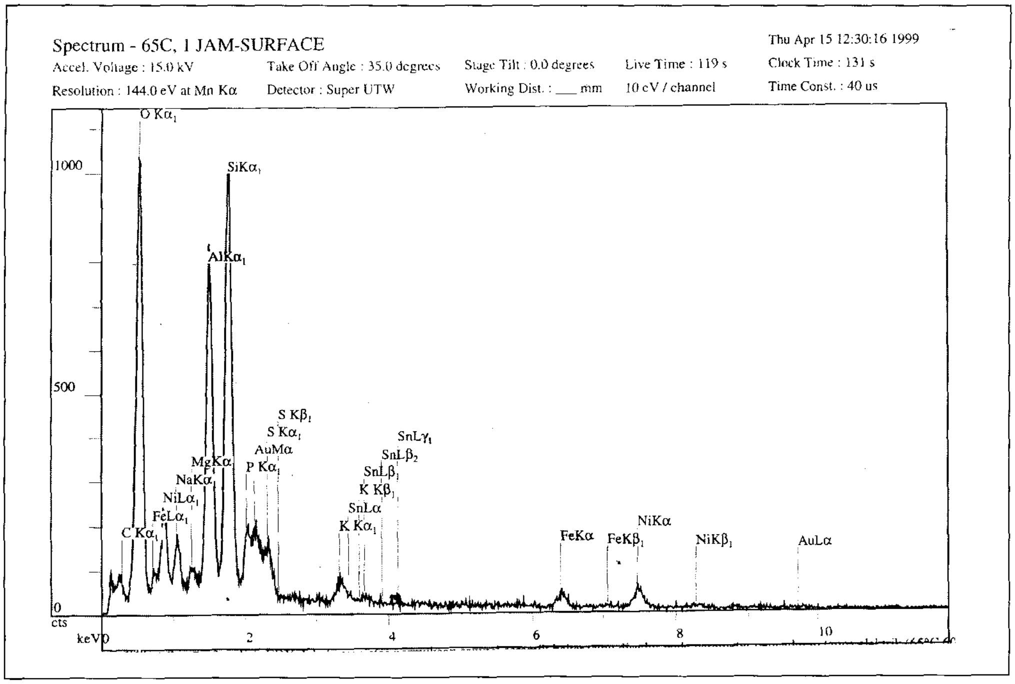

Figure 13a. EDX at the surface of sample (65°C, 60 minutes)

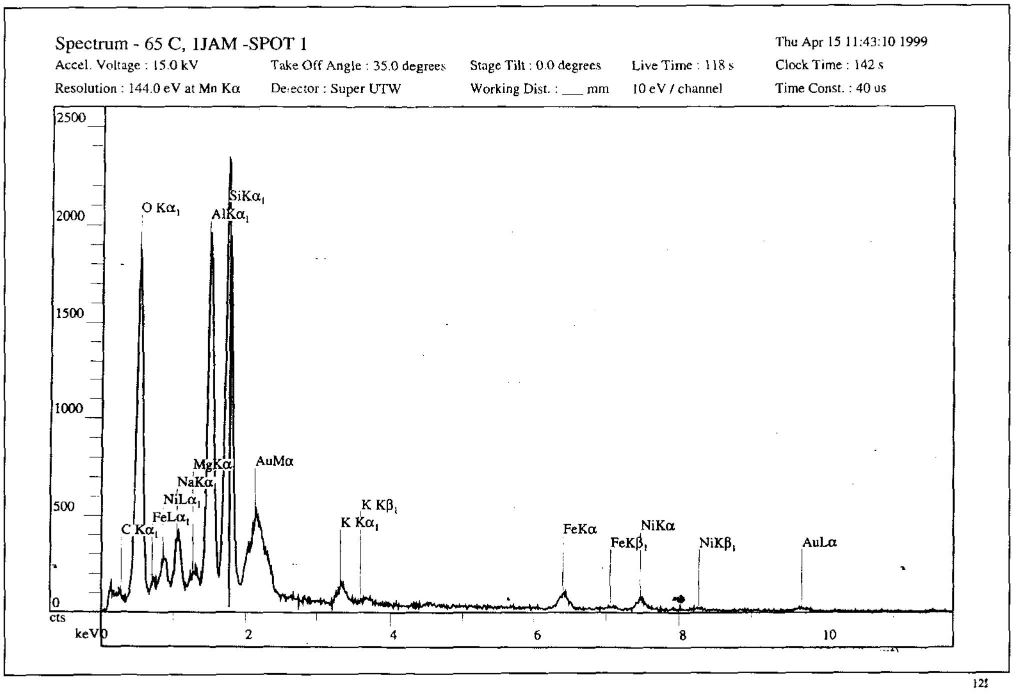

Figure 13b. EDX at spot 1 of sample (65°C, 60 minutes)

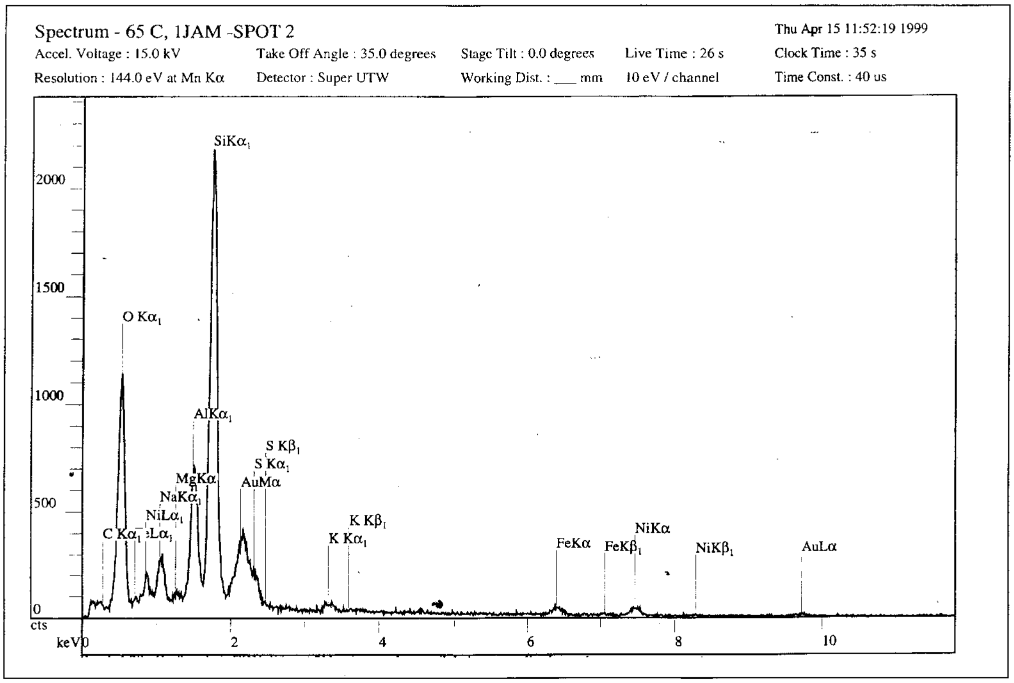

Figure 13c. EDX at spot 2 of sample (65°C, 60 minutes)

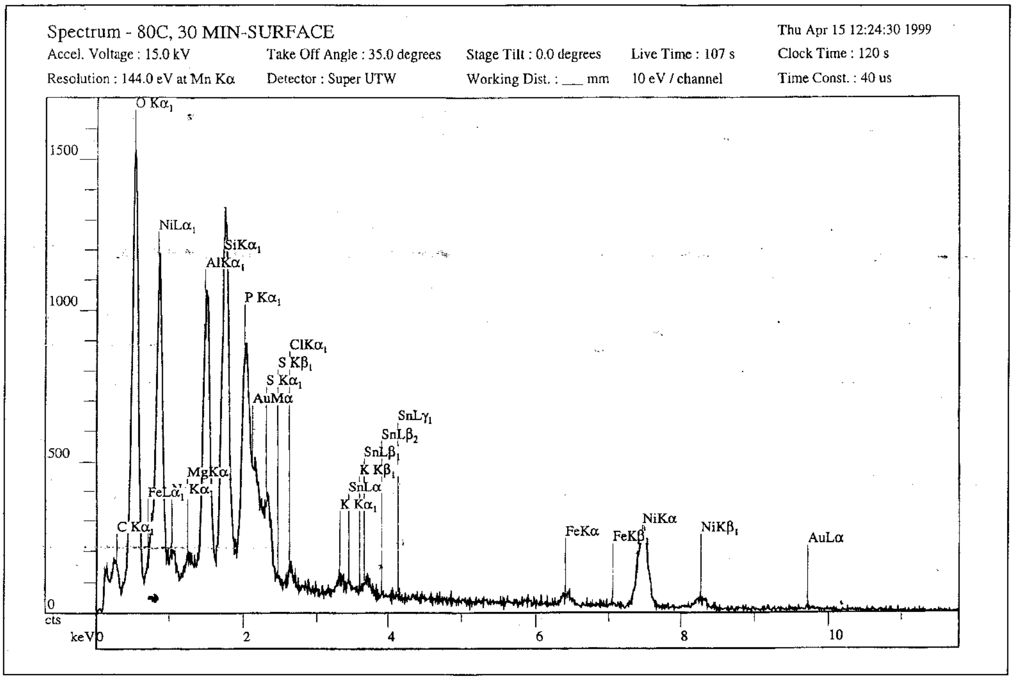

Figure 14a. EDX at the surface of sample (80°C, 30 minutes)

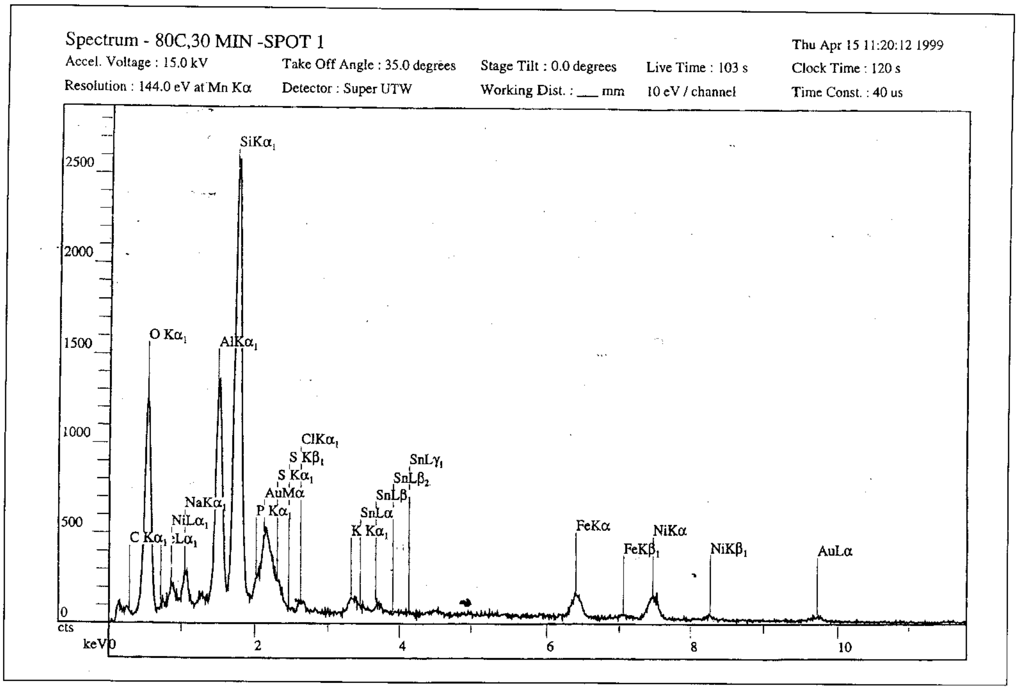

Figure 13c. EDX at spot 1 of sample (80°C, 30 minutes)

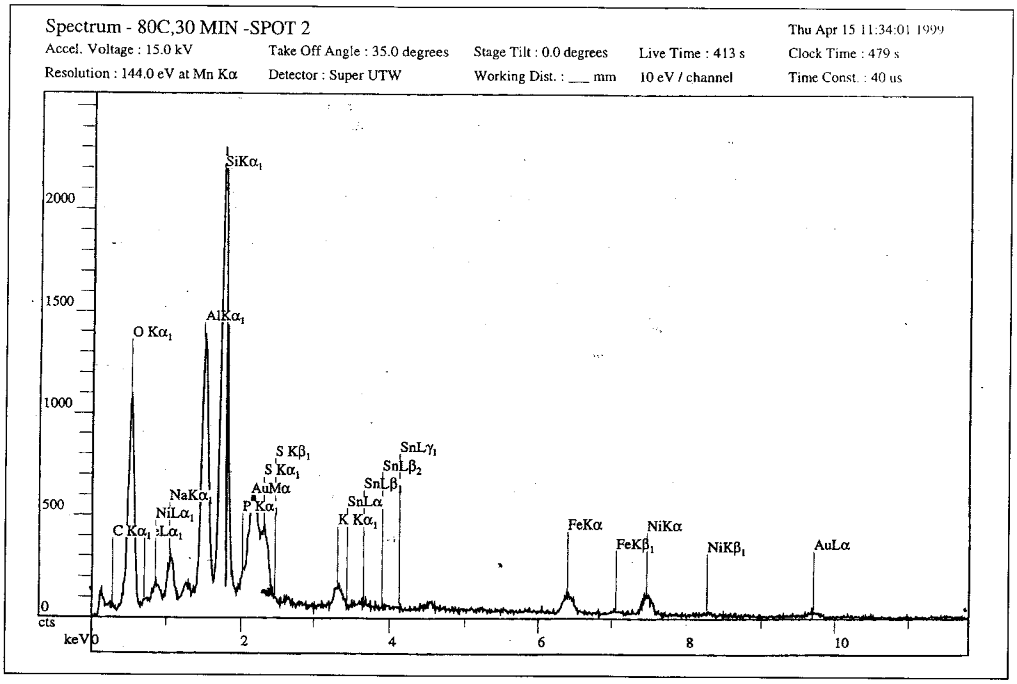

Figure 14c. EDX at spot 2 of sample (80°C, 30 minutes)