SILVERFILDENTAL PRODUCTS Sdn. Bhd.

TECHNICAL REPORT

REPORT NO: S01-SPSD

Determination of Presence of Excess Hg in Dental Amalgams

Submitted By

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406
FAX: +60 3 7967-4146

TECHNICAL REPORT

REPORT NO: S01-SPSD
Title of Project: **Determination of Presence of Excess Hg in Dental Amalgams**
Client: **Silverfildental Products Sdn. Bhd.** Date: **05 June 2015**

Date:	**Prepared by:**
05 June 2015	**Dr. Vengadaesvaran a/l Balakrishnan** **(Senior Research Officer)**

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

1. INTRODUCTION

Significantly Malaysia produced the first mercury free dental amalgam namely Silverfil Argentum. It is manufactured purely from silver particle and mercury. According to the manufacture, Silverfil Argentum has been tested and conforms the following standards; ISO 1559: 1995 (Dental Material-Alloys for Dental Amalgam, EN 1641:2004 (Dentistry- Medical Devices for Dentistry) and ISO 24234:2004 (Dental Mercury). According to the manufacturer, the director of the Oral Health Divison of the Ministry of Health Malaysia issued directive that all the government dental hospitals and clinic shall use Silverfil™ as filling material due to the absence of excess mercury. Therefore, Silverfil™ currently is used by dental clinicians in Malaysia as an alternative material for traditional dental amalgam. In unpublished work, according to the manufacturer, Silverfil™ has been tested confirming absence of free mercury in the setting amalgam by using X-ray mapping and metallographic examinations at City University, London in 1990. Hence, similar results obtained by using optical absorption and EDX at the Institute of Advance Studies, University Malaya in 1995. Further work has been carried out for in-vivo cytotoxicity and genotoxicity tests of Silverfil™. Both studies have been tested following international guidelines, ISO 10993-5 and ISO 10993 respectively. The studies confirmed that Silverfil™ do not have any cytotoxycity or genotoxicity effect. According to the manufacturer, Silverfil™ amalgam has been tested for biocompatibility following international guidelines, ISO 10993-10 and has proven to be biocompatible. In recent years, there are some studies has been conducted to further confirmed the free mercury in Silverfil™ amalgam. An electrochemical study was conducted by N.H.A Kasim et al. shown that mercury diffusion rate into silver is higher until no excess Hg after amalgamation process complete (Kassim, Yahya, Radzi, Basirun, & Ghani, 2007). In another study, it has been proven that the mercury used for amalgamation process is completely absorbed by the silver and it does not contain any unreacted

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

mercury (excess mercury) upon setting (Kassim et al., 2007; Ramasindarum, Balakrishnan, Kasim, & Yarmo, 2013).

SCOPE OF WORK

The purpose of this work is to establish a comparison with regards to the evidence of excess Hg in the final amalgam (set amalgam). The comparative materials chosen are as follows:

Properties	**Silverfil amalgam**	**GS80 amalgam**	**Dispersalloy amalgam**
Alloy composition	60 % Ag 40 % Ag_3Hg_2 340 mg powder (Ag + AgHg) 600 mg Hg	40 % Ag 31.3 % Sn 28.7 % Cu 360 mg Hg	69 % Ag 18 % Sn 12 % Cu 1% Zn 400 mg Hg
Manufacturer	Silverfildental Products sdn Bhd, Malaysia	SDI, Australia	Dentsply, USA
Lot No.	SF110801	100222119	100517

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

2. RESULTS

Comparison of the physical properties of Silverfil™ amalgam and two commercially available dental amalgams

XRD analysis

The XRD pattern of Silverfil™ amalgam is shown in Figure 1. The diffractogram shows that this amalgam is in the form of crystalline. All the diffraction peaks with (411) peak having the highest intensity could be readily indexed the value reported in JCPDS11-0067. This suggested that Silverfil™ amalgam is in the form of isometric (cubic)-hexaoctahedral with a unit cell of 10.06 Å which is similar to gamma moschellandsbergite, a natural mineral. All the peaks of Silverfil amalgam are similar to the gamma-moschellandsbergite which has the molecular formula Ag_2Hg_3. This infers that Silverfil is stable because of the fraction of stable compounds as Ag_2Hg_3.

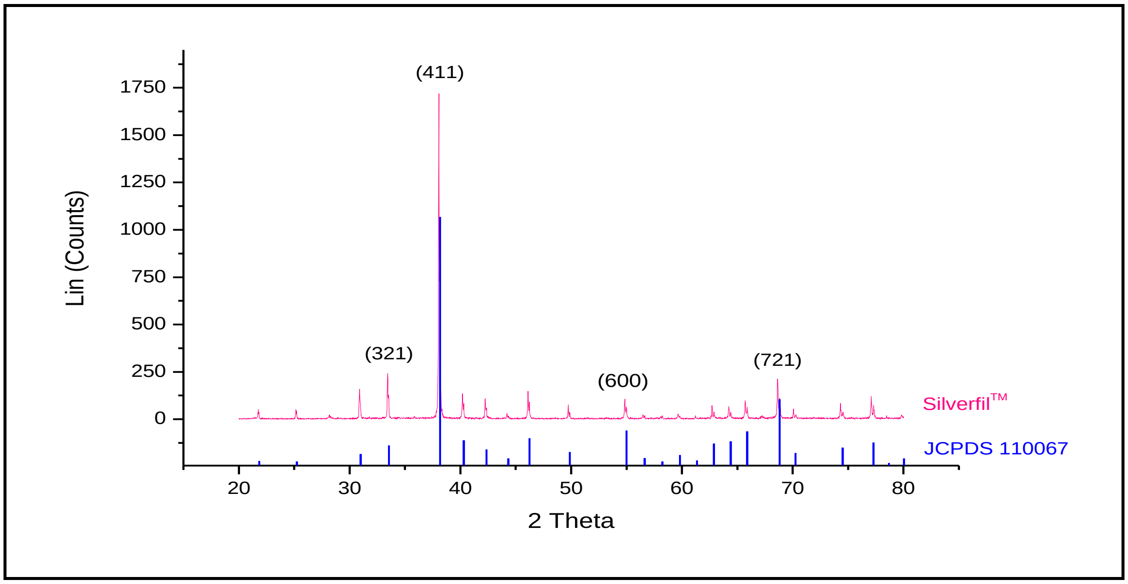

Figure 1: XRD diffraction pattern of Silverfil™ amalgam

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

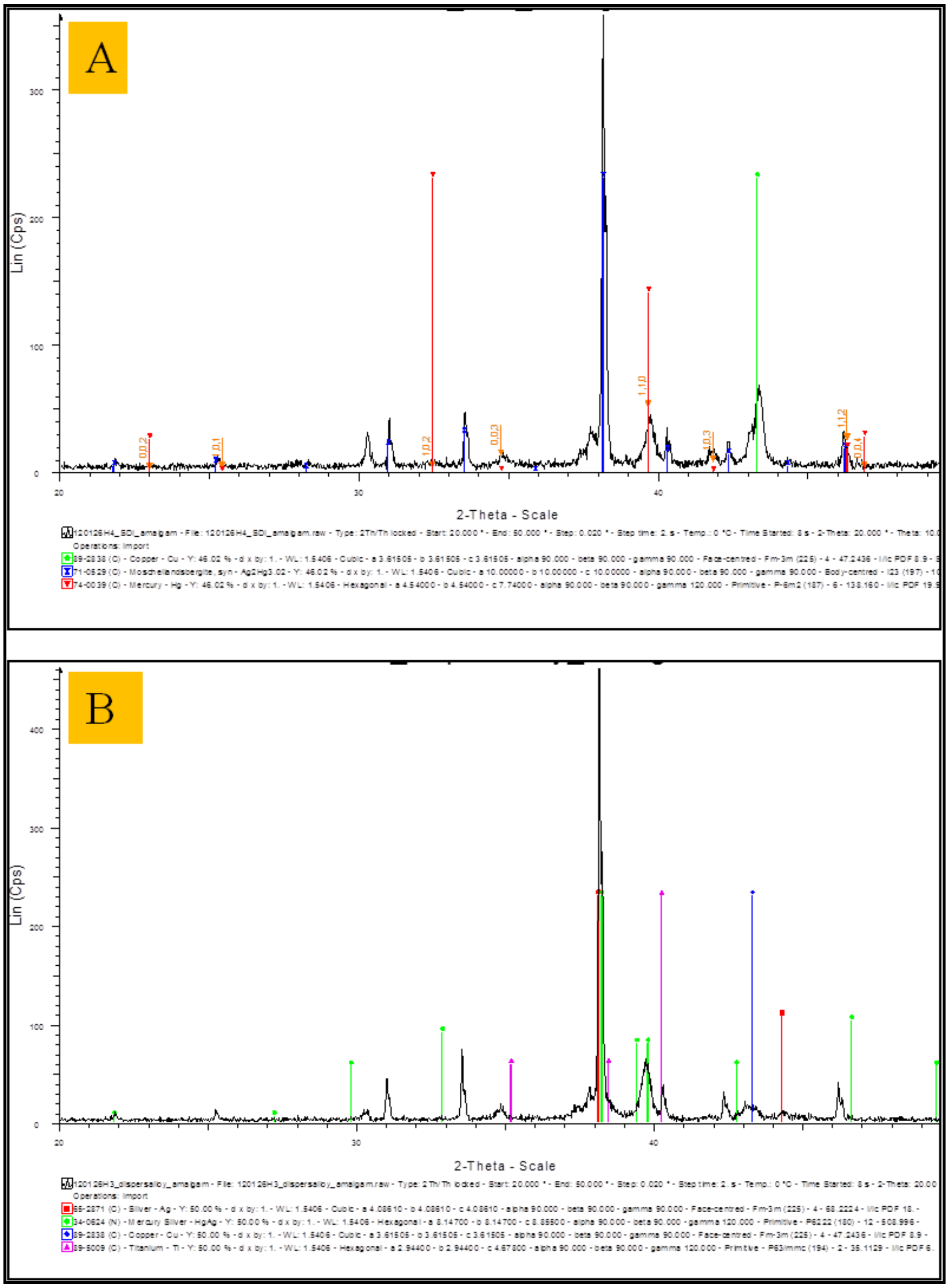

Figure 2: XRD pattern of two commercial amalgams; A. GS80 and B. Dispersalloy

3.

On the other hand, Figure 2 represents the XRD pattern of the two commercial amalgams; GS80 and Dispersalloy. Both amalgams are in the crystalline form. For

GS80 amalgam and the diffraction peaks matches a few value reported in the standard. The major peaks could be readily indexed the value reported in JCPDS 71-0529 which indicated the presences of $Ag_2Hg_{3.02}$. The small intensity peaks matches the value reported in data base JCPDS 74-0039 which represent of Hg and the peak at 43.36^o matches the value reported for copper (JCPDS 89-2838). Similar observation found for Dispersalloy amalgam, the main diffraction peaks matches the peaks value reported in JCPDS34-0624 which indicates the presence of AgHg and a broader peak at 43.3^o matches the value reported for copper (JCPDS 89-2838).

X-Ray Photoelectron Spectroscopy (XPS)

The wide scan spectrum of Silverfil^{TM} is represented in Figure 3 and the relative atomic percentage of the elements is detailed in Table 1. The narrow scan of silver, mercury and tin are shown in Figure 4, 5 and 6 respectively. The wide scan of XPS analysis of Silverfil^{TM} indicates the presence of expected photoelectron peaks of O 1s, C 1s, Sn 3d, Ag 3d and Hg 4f with relative atomic concentration of 24.19 %, 47.85 %, 13.18 %, 10.17 % and 4.60 % respectively.

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

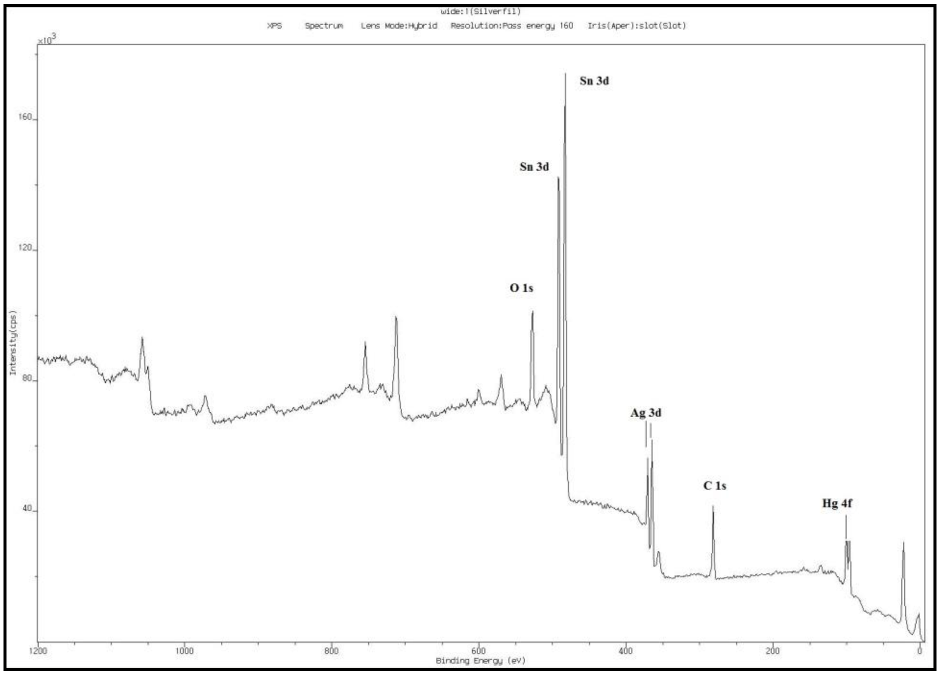

Figure 3: The wide scan XPS spectra of Silverfil^{TM}

Table 1: The relative atomic concentration of Silverfil^{TM}

Element	Peak Quantified	Relative atomic concenteration (%)
Oxygen	O 1s	24.19
Tin	Sn 3d	13.18
Silver	Ag 3d	10.17
Carbon	C 1s	47.85
Mercury	Hg 4f	4.60

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

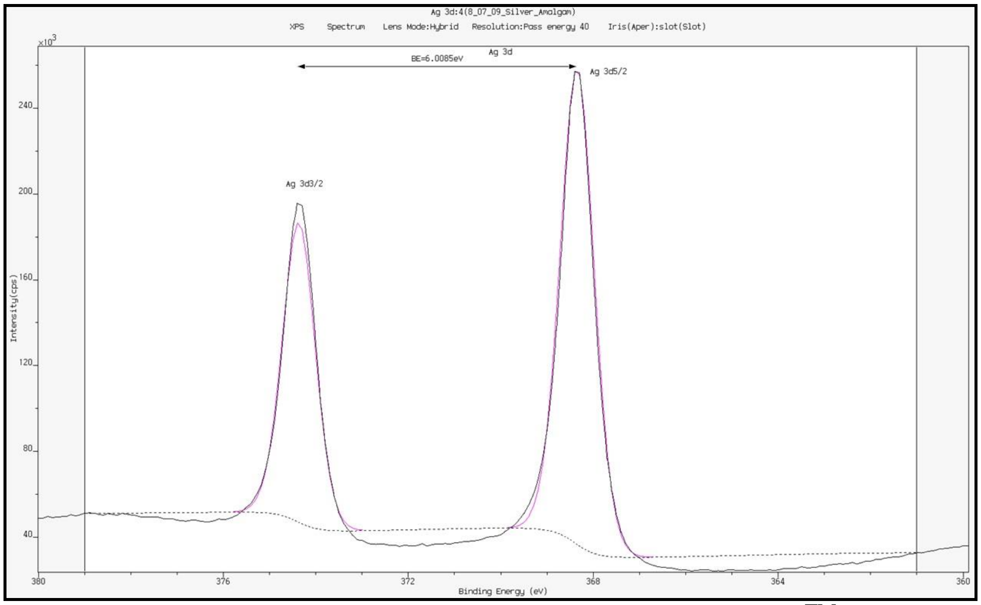

Figure 4: The narrow scan XPS spectra of Silverfil^{TM}
(Ag 3d region)

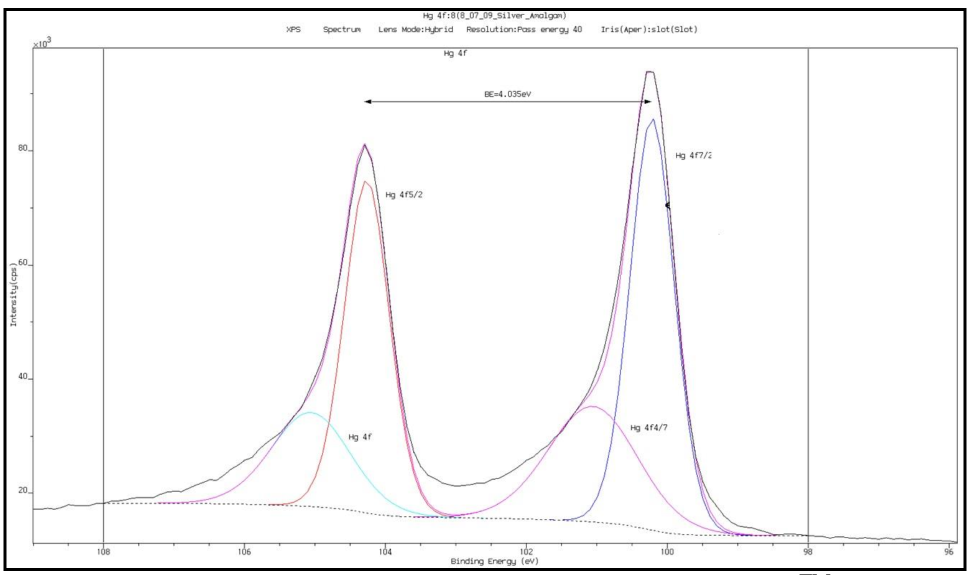

Figure 5: The narrow scan XPS spectra of Silverfil^{TM}
(Hg 4f region)

Narrow scan of Ag 3d (Figure 4) shows doublet pair which arises due to spin-orbital coupling $3d_{5/2}$ and $3d_{3/2}$. The binding energy for the Ag $3d_{5/2}$ and Ag 3d $_{3/2}$ peaks are 368.0 eV and 374.0 eV respectively. The difference between binding energy is 6.0 eV.

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

These peaks at this specific binding energy value confirm the formation of silver as Ag^0. On the other hand, the binding energy for Hg 4f (Figure 5) shows peaks Hg 4f $_{5/2}$ at 104.2eV and Hg 4f $_{7/2}$ at 100.2 eV. The difference in binding energy of 4.03 eV corresponds to the presence of Hg alloy.

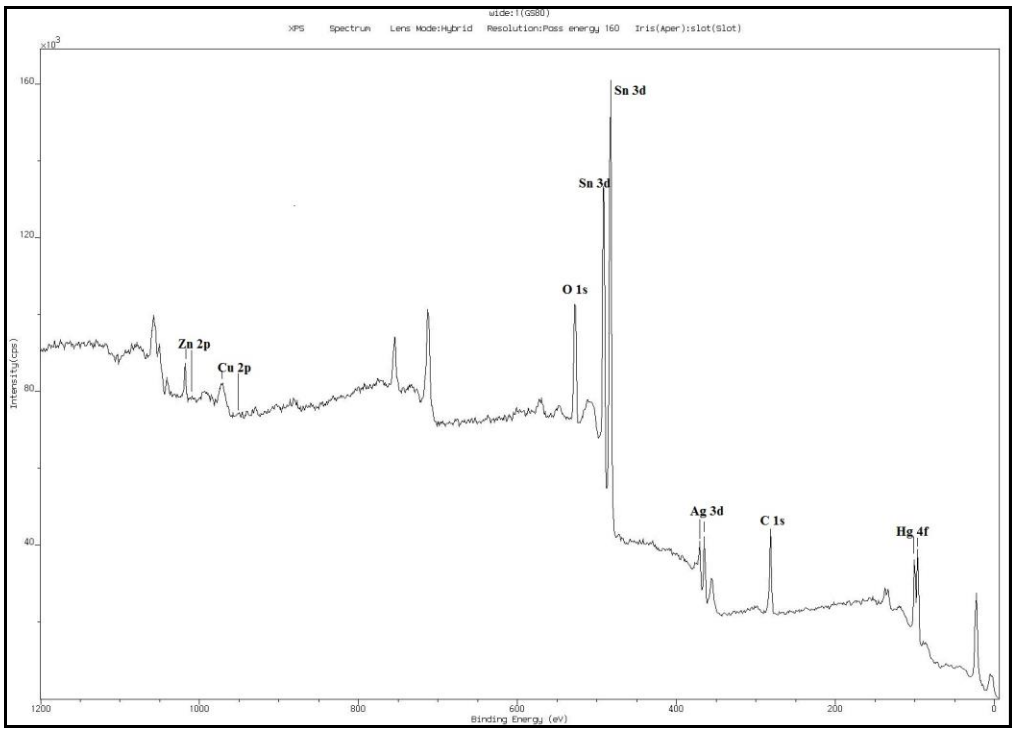

Figure 6: The wide scan XPS spectra of GS80

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

Table 2: The relative atomic concentration of GS80

Element	Peak Quantified	Relative atomic concenteration (%)
Zinc	Zn 2p	1.84
Copper	Cu 2p	0.66
Oxygen	O 1s	26.20
Tin	Sn 3d	12.71
Silver	Ag 3d	4.06
Carbon	C 1s	50.17
Mercury	Hg 4f	4.36

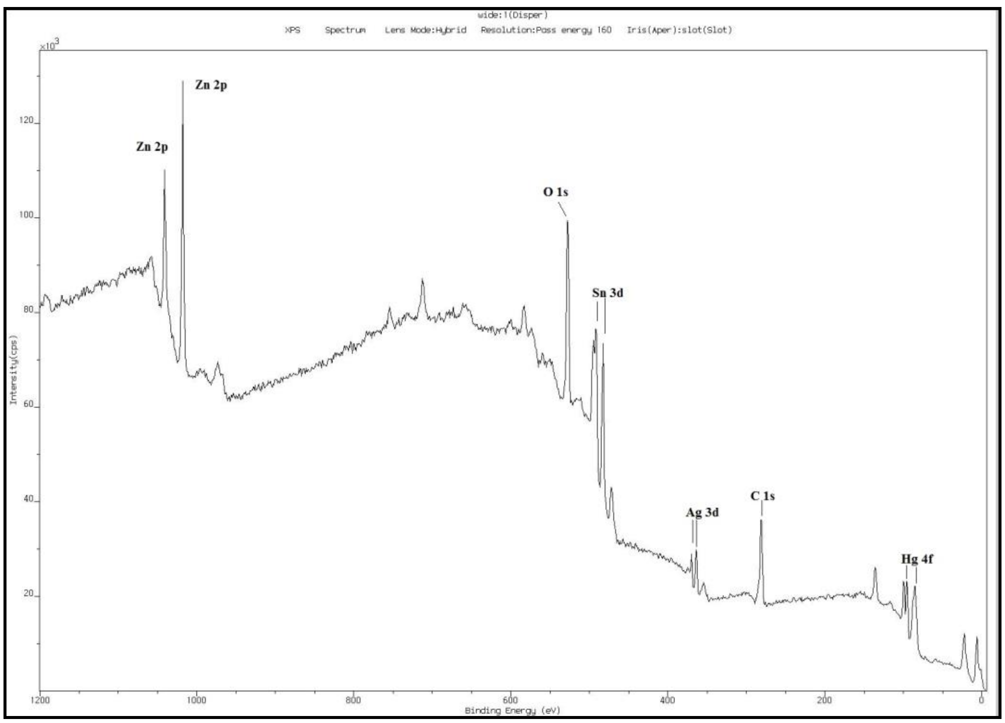

Figure 7: The wide scan XPS spectra of Dispersalloy

CENTRE FOR IONICS
Physics Department, Faculty of Science,
University of Malaya, 50603,
Kuala Lumpur
TEL: +60 3 7967-4406 FAX: +60 3 7967-4146

Table 3: The relative atomic concentration of Dispersalloy

Element	Peak Quantified	Relative atomic concenteration (%)
Zinc	Zn 2p	8.4
Oxygen	O 1s	22.86
Tin	Sn 3d	3.75
Silver	Ag 3d	2.76
Carbon	C 1s	59.87
Mercury	Hg 4f	2.38

The wide scan spectra of GS80 amalgam and Dispersalloy are represented in Figure 6 and 7 respectively. The relative atomic concentration detailed in Table 4.12 and 4.13 for GS80 and Dispersalloy. For GS80 amalgam, the survey spectra (Figure 4.41) demonstrate photoelectron peaks of O 1s, C 1s, Sn 3d, Cu 2p, Ag 3d, Zn 2p and Hg 4f with relative atomic concentration of 26.20 %, 50.17 %, 12.71 %, 0.66 %, 4.06 %, 1.84 % and 4.36 % respectively. On the other hand, Dispersalloy amalgam shows similar photoelectron peaks but with different relative atomic percentage. It was observed that the relative atomic percentage of C 1s is 59.87 %, followed by O 1s 22.86 %, Zn 2p 8.4 %, Sn 3d 3.75 %, Ag 3d 2.76 % and Hg 4f 2.38 %. However, the percentage of copper is too low as it cannot be detected in the survey scan as it shows lots of noise in its narrow scan.

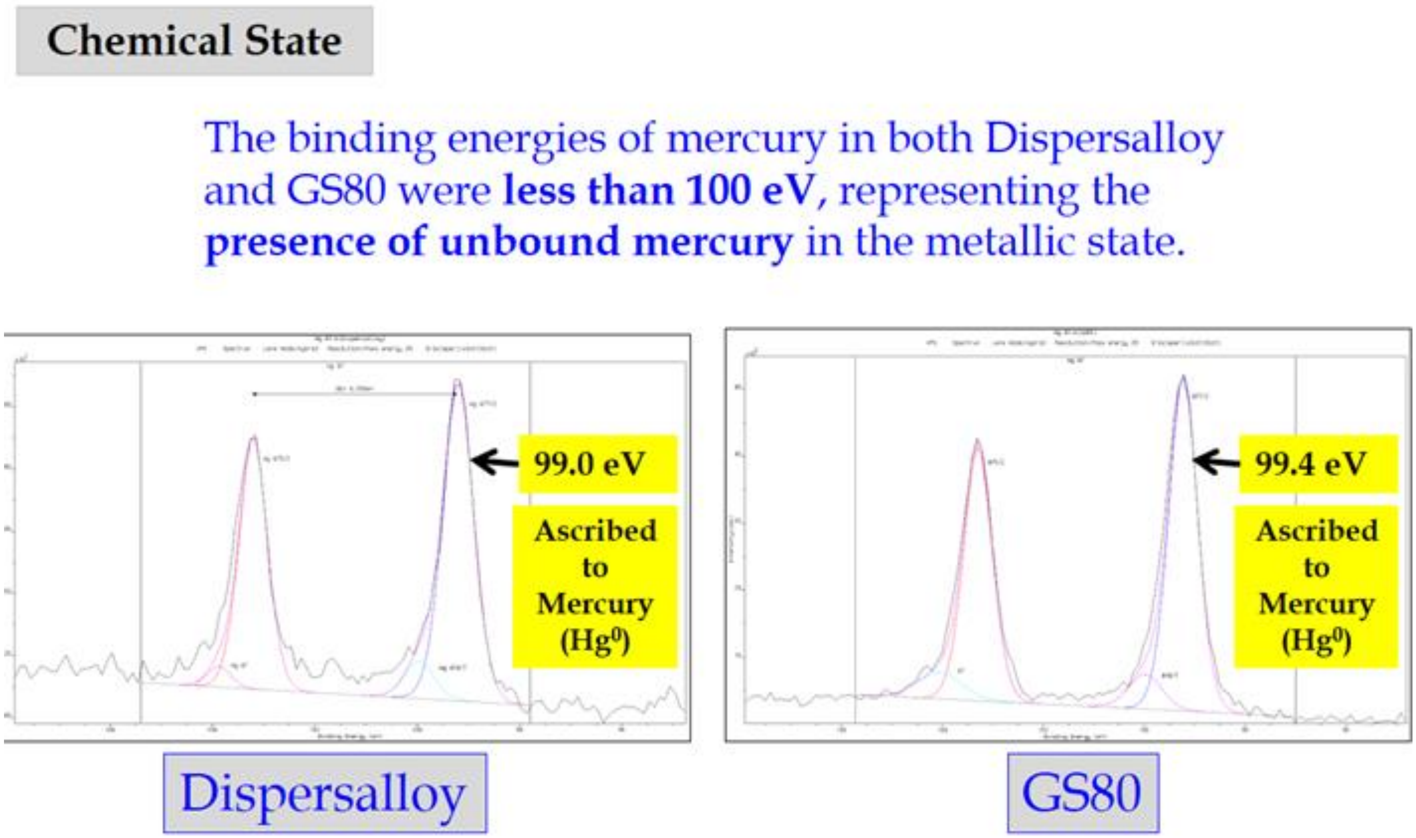

Figure 8: The narrow scan XPS spectra of Dispersalloy and GS 80

The binding energy for Hg (Figure 8) clearly shows peaks Hg^0 at 99.0 eV for Disperse alloy and Hg^0 at 99.4 eV for GS80. These peaks confirm the presence of unbound mercury or presences of excess Hg in both disperse alloy and GS80.

4. CONCLUSIONS

The above studies conclusively establish the following:

- There is no evidence of any excess Hg in the amalgamated SILVERFIL (Final Amalgam).
- In Dispersealloy and GS80 there is clear evidence in the presence of excess mercury in both the amalgams (final amalgam).