Investigation into the effect of Gamma Sterilisation (200kGy)

and

Accelerated ageing on the properties of PEEK-OPTIMA®

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Summary

An investigation was carried out into individual and combined effects of increased gamma sterilization doses and accelerated ageing on the properties of PEEK-OPTIMA®.

PEEK-OPTIMA® samples from extruded rod stock were characterised in terms of their short term mechanical properties, FTIR spectra, GPC data and differential scanning calorimetry following gamma irradiation (200 kGy), accelerated ageing in oxygen (40 days at 5 bar at 70°C) and the combined effects of gamma irradiation followed by accelerated ageing.

Test results suggested that there was no significant change in the properties of treated samples compared to control samples prepared in an identical manner from the same batch.

Cytotoxicity and Implantation Studies confirmed that the biocompatibility of PEEK-OPTIMA® was not altered by the combined effects of gamma sterilization and accelerated ageing.

Contents

1.0 Introduction

An investigation was carried out into individual and combined effects of increased Gamma sterilization doses and accelerated ageing on the properties of PEEK-OPTIMA®. Previous studies have independently considered the effect of irradiation doses of around 75kGy and also the effect of accelerated ageing in oxygen. These reports concluded that the biocompatibility and mechanical performance of PEEK-OPTIMA polymers were not adversely affected by the treatments.

In response to concerns relating to the potential effects of increased gamma radiation doses we have completed a study where PEEK-OPTIMA® samples have been subjected to an increased gamma radiation dose of 200kGy followed by accelerated ageing in an oxygen environment.

1.1 PEEK-OPTIMA® subjected to 200kGy Gamma Radiation

A number of machined ISO test samples were taken from PEEK-OPTIMA extruded rod batch number SSR0047 (PEEK-OPTIMA® LT1R40) and subjected to Gamma Sterilisation at a dose of 200kGy by GAMMASTER® of Germany. These samples were then subjected to a number of physical and chemical tests in accordance with standard Invibio® test procedures to ascertain any changes in properties.

1.2 Gamma Sterilisation Results

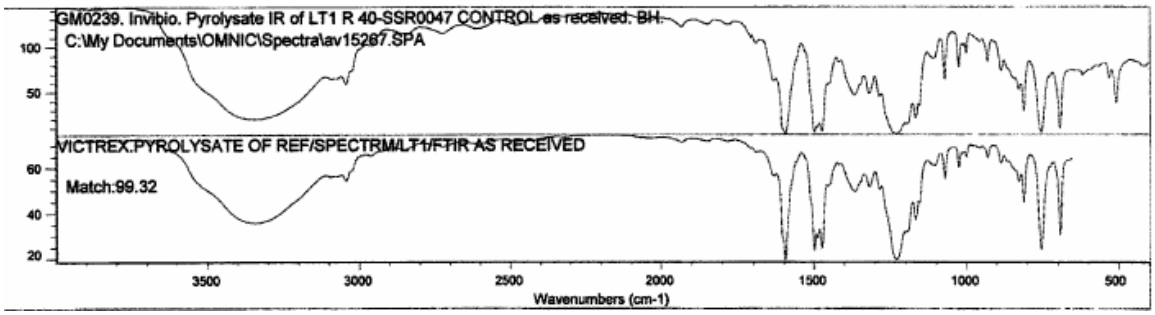
Test	Test Method	Manufacturing Definition	Results Before Sterilisation	Mean After 200kGy Sterilisation	SD
Tensile Strength (MPa)	ISO 527	≥ 90	115	113.9	1.56
Tensile Elongation (%)	ISO 527	≥ 5	17	15.3	0.65
Flexural Strength (MPa)	ISO 178	≥ 150	183	186.4	4.34
Flexural Modulus (GPa)	ISO 178	≥ 3.8	4.3	4.5	0.11
Impact Strength (Notched Izod) ($KJ/m^{2)}$	ISO 180	≥ 4.5	4.6	4.6	0.77
Density (g/cc)	ASTM D792	1.28 – 1.32	1.31	1.31	0

1.3 Infrared Spectroscopy

Infrared spectra were produced for samples before and after gamma irradiation. These samples were compared using Rapra technologies internal procedures using Fourier transform infrared spectroscopy.

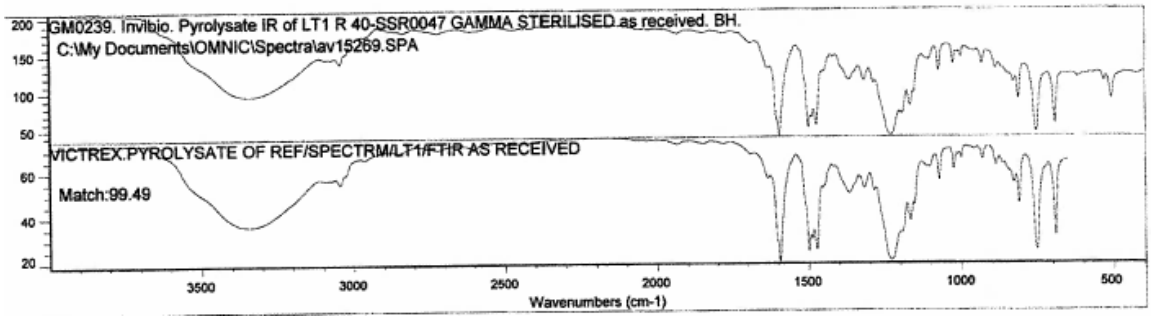
The control samples were initially analysed to ensure that the control sample was identical to reference samples for PEEK-OPTIMA LT1 (Figure 1).

Figure 1



Following this the gamma irradiated samples were compared with the reference sample (Figure 2).

Figure 2



This showed identical traces for the gamma irradiated samples with those of the reference sample and hence those of the control samples for SSR0047.

1.3 Gel Permeation Chromatography (GPC)

Gel permeation chromatography was used to determine the weight and number average molecular weights of the polymer before and after gamma sterilization. All tests were carried out in duplicate.

Sample	Mw	Mn	Polydispersity
LT1R40 SSR0047	108,000 105,000	45,800 44,700	2.4 2.4
LT1R40 SSR0047 – Gamma Sterilised 200kGy	107,000 106,000	41,900 43,500	2.5 2.4

This demonstrated that the molecular weight of PEEK-OPTIMA® polymer was not affected by increased doses of gamma irradiation.

1.4 Conclusion

The results from the mechanical property evaluation, FTIR and GPC analysis demonstrates that there is no significant change in the properties of these samples compared to non-irradiated samples prepared in an identical manner from the same batch.

2.0 PEEK-OPTIMA® subjected to accelerated ageing in Oxygen

These tests were carried out using selected methods described in ASTM F2003. This standard is used to measure accelerated ageing in UHMWPE and describes the ageing of the samples in oxygen at 70°C and 5bar for 15 days to simulate 10 years ageing.

A number of machined ISO test samples were taken from PEEK-OPTIMA extruded rod batch number SSR0047 (PEEK-OPTIMA® LT1R40) and subjected to ageing at 70°C and 5bar for 40 days. These samples were then subjected to a number of physical and chemical tests in accordance with standard Invibio® test procedures to ascertain any changes in properties.

2.1 Accelerated Ageing in Oxygen Results

Test	Test Method	Manufacturing Definition	Results Before Ageing	Mean After Ageing	SD
Tensile Strength (MPa)	ISO 527	≥ 90	115	114.6	1.8
Tensile Elongation (%)	ISO 527	≥ 5	17	15.5	1.6
Flexural Strength (MPa)	ISO 178	≥ 150	183	192.7	0.62
Flexural Modulus (GPa)	ISO 178	≥ 3.8	4.3	4.6	0.03
Impact Strength (Notched Izod) ($KJ/m^{2)}$	ISO 180	≥ 4.5	4.6	4.8	0.08
Density (g/cc)	ASTM D792	1.28 – 1.32	1.31	1.31	0

2.2 Infrared Spectroscopy

Infrared spectra were produced for samples before and after accelerated ageing. These samples were compared using Rapra technologies internal procedures using Fourier transform infrared spectroscopy.

The samples were analysed by pyrolysate IR (**Figure 3**) and Golden Gate IR (**Figure 4**). This resulted in identical FTIR traces for aged and non-aged samples.

Figure 3

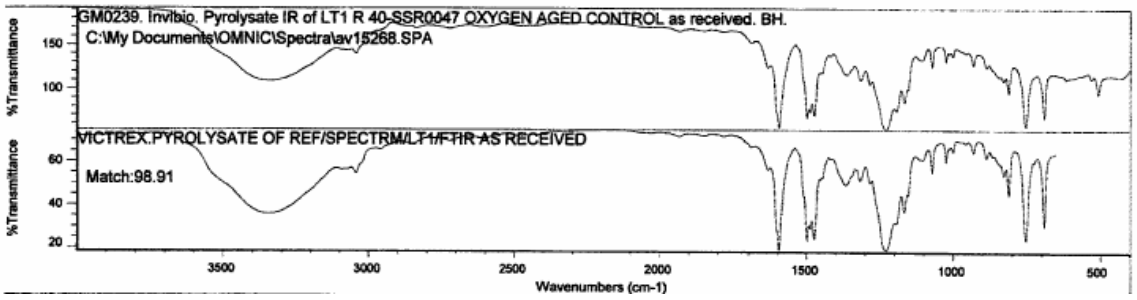
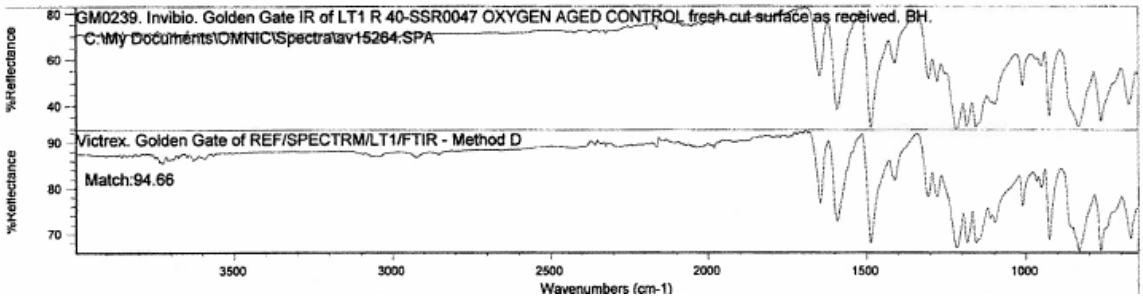


Figure 4



2.3 Gel Permeation Chromatography (GPC)

Gel permeation chromatography was used to determine the weight and number average molecular weights of the polymer before and after accelerated ageing. All tests were carried out in duplicate.

Sample	Mw	Mn	Polydispersity
LT1R40 SSR0047	108,000	45,800	2.4
	105,000	44,700	2.4
LT1R40 SSR0047 – Aged	106,000	44,600	2.4
	110,000	45,300	2.4

This demonstrated that the molecular weight of PEEK-OPTIMA® polymer was not affected by accelerated ageing in oxygen.

2.4 Conclusion

The results from the mechanical property evaluation, FTIR and GPC analysis demonstrates that there is no significant change in the properties of accelerated aged samples compared to non-aged samples prepared in an identical manner from the same batch.

3.0 PEEK-OPTIMA® subjected to 200kGy gamma irradiation followed by accelerated ageing in Oxygen.

A number of machined ISO test samples were taken from PEEK-OPTIMA extruded rod batch number SSR0047 (PEEK-OPTIMA® LT1R40) and subjected to Gamma Sterilisation at a dose of 200kGy by GAMMASTER® of Germany followed by ageing at 70°C and 5bar for 40 days. As previously stated ASTM F2003 describes the ageing of the samples in oxygen at 70°C and 5bar for 15 days to simulate 10 years ageing. These samples were then subjected to a number of physical and chemical tests in accordance with standard Invibio® test procedures to ascertain any changes in properties.

3.1 Gamma Irradiation (200kGy) followed by accelerated ageing in Oxygen

Test	Test Method	Manufacturing Definition	Results Before Irradiation and Ageing	Mean After Irradiation and Ageing	SD
Tensile Strength (MPa)	ISO 527	≥ 90	115	114.3	1.6
Tensile Elongation (%)	ISO 527	≥ 5	17	15.2	1.9
Flexural Strength (MPa)	ISO 178	≥ 150	183	191.0	2.9
Flexural Modulus (GPa)	ISO 178	≥ 3.8	4.3	4.6	0.08
Impact Strength (Notched Izod) ($KJ/m^{2)}$	ISO 180	≥ 4.5	4.6	5.0	0.3
Density (g/cc)	ASTM D792	1.28 – 1.32	1.31	1.30	0

3.2 Infrared Spectroscopy

Infrared spectra were produced for samples before and after gamma irradiation and accelerated ageing. These samples were compared using Rapra technologies internal procedures using Fourier transform infrared spectroscopy.

The samples were analysed by pyrolysate IR (**Figure 5**) and Golden Gate IR (**Figure 6**). This resulted in identical FTIR traces for aged and non-aged samples.

Figure 5

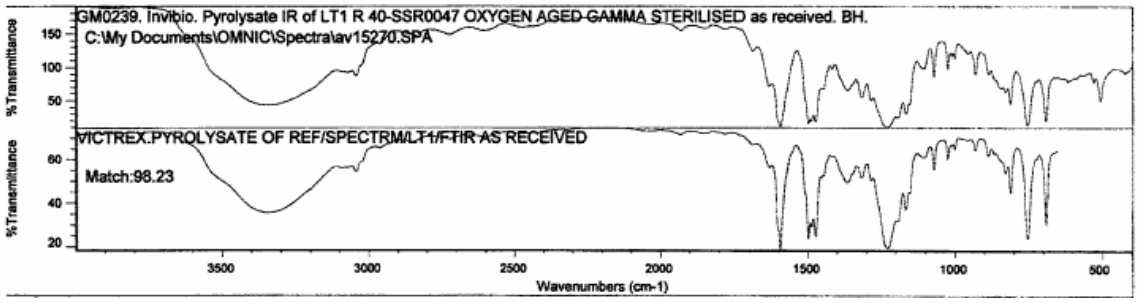
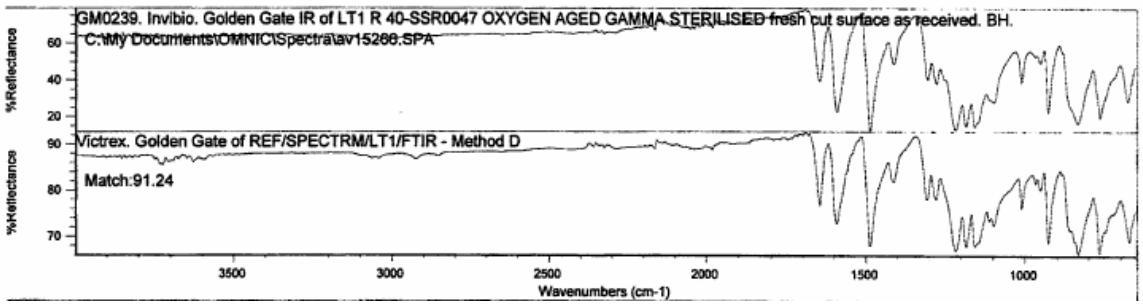


Figure 6



3.3 Gel Permeation Chromatography (GPC)

Gel permeation chromatography was used to determine the weight and number average molecular weights of the polymer before and after gamma sterilization and accelerated ageing. All tests were carried out in duplicate.

Sample	Mw	Mn	Polydispersity
LT1R40 SSR0047	108,000	45,800	2.4
	105,000	44,700	2.4
LT1R40 SSR0047	107,000	43,200	2.5
Irradiated and Aged	107,000	42,500	2.5

This demonstrated that the molecular weight of PEEK-OPTIMA® polymer was not affected by gamma irradiation and accelerated ageing in oxygen.

3.4 Differential Scanning Calorimetry

DSC was used to characterise the melting and crystallisation behaviour of a control sample from SSR0047 (PEEK-OPTIMA® LT1R40) (Figure 7) and a gamma sterilised and oxygen aged sample (Figure 8).

Figure 7

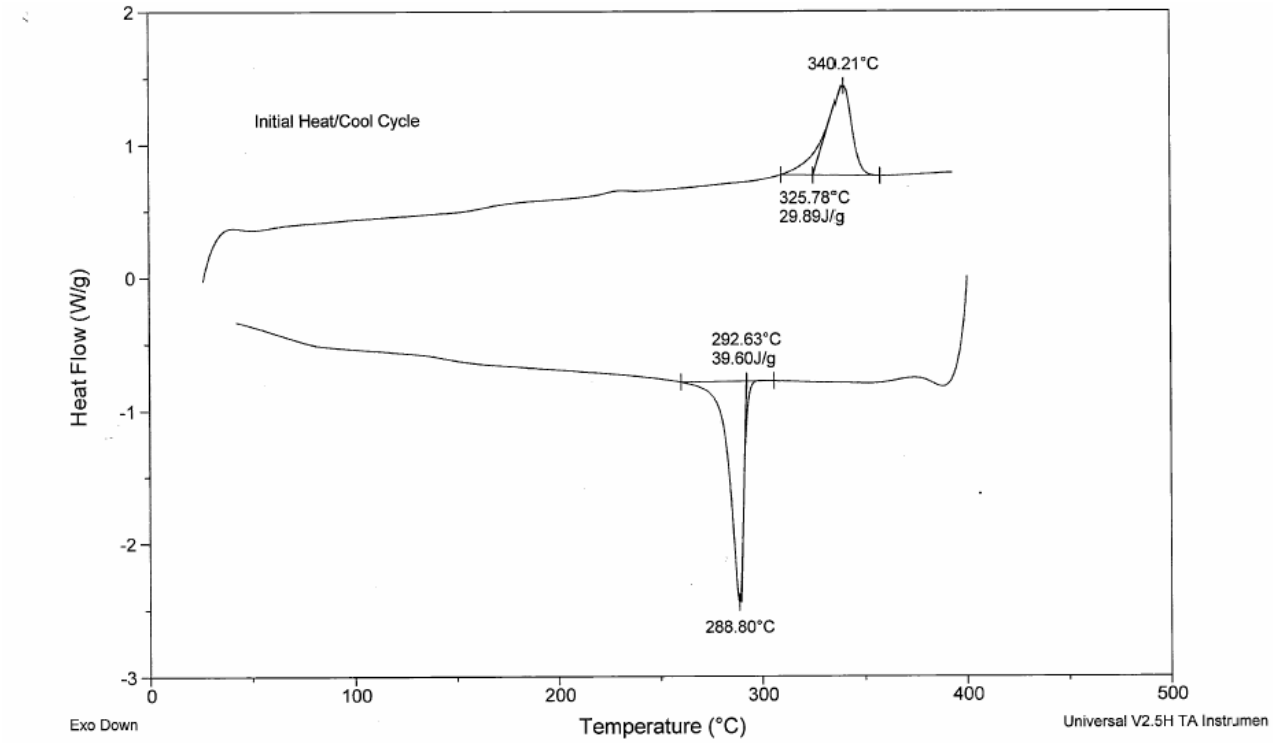
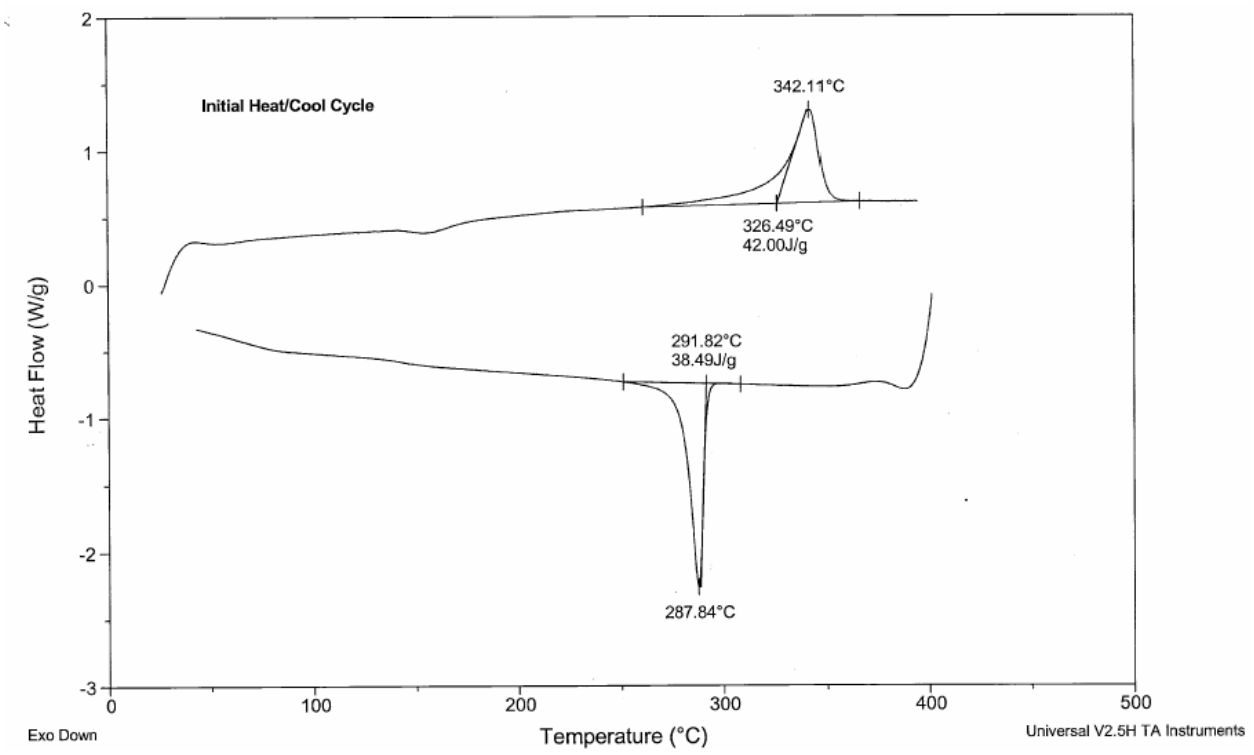


Figure 8



These traces show that the melting and crystallisation characteristics of PEEK-OPTIMA® are not adversely affected by gamma irradiation followed by accelerated ageing in oxygen.

3.5 Conclusion

The results from the mechanical property evaluation, FTIR, GPC and DSC analysis demonstrates that there is no significant change in the properties of gamma irradiated and accelerated aged samples compared to untreated samples prepared in an identical manner from the same batch.

4.0 Cytotoxicity Testing

PEEK-OPTIMA® samples which were gamma irradiated (200kGy), accelerated aged in oxygen (40 days at 5bar pressure at 70°C) and which were subjected to a combination of irradiation and accelerated ageing were tested in accordance with ISO 10993-5 (Cytotoxicity). Biocompatibility testing has shown that all treated samples were non-cytotoxic and similar to untreated samples.

4.1 Implantation Study

Samples of PEEK-OPTIMA LT1 extruded rod were gamma sterilised with a dose of 73.2 kGy and then incubated in physiological saline for 3 months at 90°C (simulation of 10 years real time ageing at 37°C). The PEEK-OPTIMA® samples were implanted in an animal model for a period of 12 months and subjected to cytotoxicity testing (ISO 10993-5), chemical analysis (ISO 10993-18) and a histopathological examination. These tests confirmed the material to be non cytotoxic and histopathology found no muscle degradations, no necroses nor marked inflammatory responses or any significant changes.