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RESULTS OF PRELIMINARY EXPERIMENTS ON TRITIUM
DECONTAMINATION BY UV IRRADIATION

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Yasuhisa OYA, Wataru SHU, Shigeru O'HIRA,
Takumi HAYASHI and Masataka NISHI

日本原子力研究所
Japan Atomic Energy Research Institute

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Results of Preliminary Experiments on Tritium Decontamination
by UV Irradiation

Yasuhisa OYA, Wataru SHU, Shigeru O'HIRA,
Takumi HAYASHI and Masataka NISHI

Department of Fusion Engineering Research
(Tokai Site)

Naka Fusion Research Establishment
Japan Atomic Energy Research Institute
Tokai-mura, Naka-gun, Ibaraki-ken

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In the point of view of protection of workers from the radiation exposure and the limitation of the contamination with radioactive materials, it is important to decontaminate mobile tritium from plasma facing components of a nuclear fusion reactor at the beginning of their maintenance work. It is considered that the heating is the most effective method for decontamination. However, it is important to develop new decontamination method of adsorbed hydro-carbon based substances from the materials that cannot be heated or the inner pipe of double pipes.

This report presents results of preliminary experiments performed for the development of the effective tritium decontamination technique pursuing under US/Japan collaborative program on technology for fusion-fuel processing (Annex IV). In the experiments, the effects of Ultra Violet (UV) irradiation on tritium removal from some kinds of materials, such as poly vinyl chloride $-(\text{CH}_2\text{CHCl})_n-$ film, polyethylene film and graphite samples coated by C_2H_2 plasma were examined. As the result of UV irradiation, it was confirmed that hydrogen and carbon based compounds could be released from the specimen during UV irradiation. It is concluded that UV irradiation is one of the hopeful candidates for effective tritium decontamination.

Keywords: Tritium, Decontamination, UV Irradiation, Nuclear Fusion

紫外線によるトリチウム除染に関する予備試験結果

日本原子力研究所那珂研究所核融合工学部
大矢 恭久・洲 亘・大平 茂・林 巧・西 正孝

(2000年1月25日受理)

核融合炉においてプラズマ対向機器等の保守・点検作業前にトリチウム除染を行うことは、作業者の被ばく防護や放射能汚染低減の観点から非常に重要である。現在、加熱法による除染手法が考案されているが、加熱できない材料や二重管になっている配管から効率的にメタン系の付着物を取り除く除染方法の開発が重要である。そこでこれらの要求を満たす新しい効率的なトリチウム除染技術開発が重要である。

本報告では、日米核融合研究協力協定（附属書 4）の下で実施予定のトカマク核融合試験炉トリチウム除染技術開発に向けて行った予備試験の結果について報告する。実験ではポリクロロビニルやポリエチレンシート及びアセチレンプラズマでコーティングしたグラファイトの試料に紫外線を照射し、紫外線によるトリチウム除染の可能性について検討した。その結果、紫外線を照射することにより試料表面からの水素や炭化水素等の放出が確認でき、紫外線照射が有望なトリチウム除染方法のひとつになる見通しを得た。

那珂研究所（東海駐在）：〒311-1195 茨城県那珂郡東海村白方白根 2-4

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1. Introduction

In a fusion reactor using tritium as a fuel, tritium is retained in the materials, especially near the surfaces of the materials. The retention of tritium makes their maintenance difficult. The rudimentary decontamination method should be developed to protect workers from the risk of excess tritium exposure and to lower the tritium contamination in the maintenance area.

Some decontamination treatments, like heating and gas purge methods had been proposed [1-4]. But, there are problems that how to decontaminate from some components which cannot be heated or double pipe etc., and that how to remove hydrocarbons from these components. Therefore new effective technique for tritium decontamination should be developed. Ultra violet (UV) irradiation method had been proposed as one of the convenient methods to decontaminate tritium by some researchers [5]. In the study of Kraszna and Mowat [5], the specimen was treated by UV and ozone and water was made positively. It seems that a new technique without ozone, should be developed. This decontamination technique has a few advantage. First of all, the contaminated materials can be treated in ambient temperature and pressure. In addition, this method needs shorter processing time than other methods, such as heating and gas purge. Although the UV acts only on the surface of the material, the surface decontamination of the materials is very important to handle it.

This report presents results of preliminary experiments performed for the development of the tritium decontamination technique pursuing under Japan-US collaboration on technology of fusion-fuel processing (Annex IV).

2. Experimental

In this study, the poly vinyl chloride film, the polyethylene film and the graphite specimens were used. The effects of UV (172nm) irradiation on tritium removal from poly vinyl chloride $-(\text{CH}_2\text{CHCl})_n-$ film, polyethylene $-(\text{CH}_2\text{CH}_2)_n-$ film and graphite samples coated by C_2H_2 plasma were examined. The bond energies of hydrocarbons are summarized in Fig.1 [6],

which shows that the energy of excimer lamp (172nm) is high enough to cut C-H, C-Cl or C-OH bonds. Tritium contamination on the surface of materials can be cleaned using this technique.

Figure 2 shows the schematic view of the UV irradiation system and Figure 3 depicts this system. Specimens are located on the specimen holder, which is movable for focusing in the center of the chamber. Distance from the UV source to the specimen is about 100 mm. The UV light is collimated by a collimator to concentrate the radiation on the specimen. A turbo molecular pump (70 l/sec) and a scroll pump (250 l/min) are connected to the chamber to keep it in high vacuum range. N₂ gas flowed through the UV irradiation system. The UV irradiation experiment was performed under vacuum less than 3×10^{-5} Pa. A quadrupole mass spectrometer was installed on the vacuum chamber to analyze the released gases during the UV irradiation experiment.

To clarify if UV light decomposes materials; it is convenient to analyze chloride release from the poly vinyl chloride film, which is one of the most common films containing chloride. In the second step, the polyethylene film was irradiated, because polyethylene is pure hydrocarbon materials and it is important to study the possibility of the decomposition of C-H bond. In the Tokamak Fusion Test Reactor (TFTR) at Princeton Plasma Physics Laboratory (PPPL), formations of some hydrocarbon deposition layers on plasma-facing graphite tiles were found. To study the hydrogen release from the deposited layers; similar film was made on graphite by glow discharge using a quick coater system with acetylene gas.

2.1 UV irradiation experiment using poly vinyl chloride wrap

To confirm the decomposition of chemical bonds in carbon-based compounds (C-H:435kJ/mol C-Cl:284kJ/mol), observation of species released from the UV-irradiated wrap materials that was composed of poly vinyl chlorine resin (Asahikasei Co. Ltd.) was performed. The sample covered the specimen holder and the size of the sample was 100×100mm.

The mass spectra were measured from $m/e=1$ to $m/e=100$, before, during and after UV irradiation.

2.2 UV irradiation experiment using polyethylene film

To confirm the hydrogen release from pure hydro-carbon material, UV irradiation experiment was performed with polyethylene film, the size of which was 50×50 mm. Mass spectra were measured before, during and after irradiation.

2.3 UV irradiation experiments with graphite specimens exposed to acetylene glow discharge

The deposited layer (about $1 \mu\text{m}$) was formed on the graphite (ETP-10) specimen ($10 \times 10 \times 1$ mm) by acetylene glow discharge to simulate the co-deposited layer, which was deposited with hydrogen and carbon when deposited layer was formed. The gas pressure of glow discharge was 1.4×10^{-4} Pa and voltage was 1kV. The current density of the glow discharge was about $2\text{mA}/\text{cm}^2$. The duration of the discharge was 30 minutes. Figure 4 depicts the pictures before and after the glow discharge and it is found that the thin layer was formed like circle on the specimen after the discharge. The graphite specimen with deposited layer was then irradiated by the UV irradiation system.

To distinguish between the co-deposited hydrogen and the adsorbed hydrogen from the atmospheric circumstance, the following experiments were performed. After the first UV irradiation experiment, the chamber was kept under atmospheric circumstance for about one hour. Then, the chamber was evacuated again and the second irradiation experiment was performed. These experimental procedures were continued for three times to evaluate the source of hydrogen release.

3. Results and discussion

3.1 UV irradiation experiment to the poly vinyl chloride wrap

Figure 5(a) and 5(b) show the pictures of the wrap material after UV irradiation. It is found that the color of the wrap was changed from Fig.5(a). Figure 5(b) shows the specimen holder with the wrap. These pictures make us understood the irradiation area of UV irradiation system.

Figure 6 shows the mass spectra before, during and after UV irradiation, respectively. The intensities of major peaks are summarized in Table1. The intensity of the mass peak of $m/e=2$, H_2 , during UV irradiation is two and half times as high as that before UV irradiation. Great increase of the mass peaks of 35, 36, 37 and 38 was observed during UV irradiation. It is known that the isotope ratio of ^{35}Cl to ^{37}Cl is 3:1 in nature. The ratio of these elements was consistent with the ratio of residual gas and the mass ratio of $m/e=36$ against $m/e=38$ was also about 3:1. This result indicates that chlorine was released as the form of HCl^+ . From these results, it is concluded that chlorine was released from the wrap and UV light decomposed not only the adsorbed species (water etc.) but also the substances (chlorine etc.) on the surface. The time dependence of the mass peak intensity through the experiment was displayed in Fig.7 for masses, 2, 35, 36, 37 and 38. When the sample was irradiated by UV, hydrogen was released immediately, but chlorine ($m/e=36$ and 38) was gradually released and it took five minutes for chlorine release to reach its maximum amounts during UV irradiation as seen in Fig.8.

3.2 UV irradiation experiment to the polyethylene film

Figure 9 shows the mass spectra before, during and after UV irradiation and typical values are listed in Table2. Before UV irradiation, main element in the chamber was water, namely mass number, 18. When UV light was irradiated to the polyethylene film, hydrogen gas was released simultaneously. The released hydrogen was so much that the intensity of

mass number 2 was overscaled. Some peaks were found at the mass numbers 28, 29 and 30, which correspond to $C_2H_3^+$, $C_2H_4^+$, $C_2H_5^+$. High mass number species, which had mass number over 50, were also released. Mass numbers, 55, 56 and 57 correspond to $C_4H_7^+$, $C_4H_8^+$, $C_4H_9^+$, respectively. Weak peaks are also found in mass range of 69, 70 and 71, corresponding to $C_5H_9^+$, $C_5H_{10}^+$ and $C_5H_{11}^+$. These results imply that the polyethylene film was decomposed into small fragments of hydrocarbons.

3.3 UV irradiation to the graphite specimens exposed to the acetylene glow discharge

Figure 10 show the mass spectra with graphite specimen and the typical mass peak intensities are summarized in Table3. Amount of H_2 released during UV irradiation was about four times as large as that before irradiation. From the mass spectra, the elements, whose mass numbers were 28, 29 and 44, were released. Figure 11 shows time dependence of hydrogen release (mass number 2). Scan-1 represents the first UV irradiation experiment and Scan-2 means the second UV irradiation after one hour exposure to the atmosphere and subsequent evacuation posterior to Scan-1. Scan-3 is the third UV irradiation after one hour exposure to the atmosphere and subsequent evacuation posterior to Scan-2.

Since adsorbed hydrogen from air moisture was removed in all scans, namely Scan-1, 2 and 3, it may be concluded that some deposited hydrogen with carbon was released in Scan-1 and that the difference between scan-1 and scan-2&3 suggests the amount of decontaminated hydrogen. The amount of hydrogen released in first scan was twice as large as that of background.

From these results, the hydrogen release from co-deposited layer formed by acetylene glow discharge can be distinguished from the hydrogen release from the adsorbed gases.

3.4 Evaluation of cutting efficiency by UV irradiation

The decontamination efficiency was calculated using the ionization probability of mass spectrometer, as listed in Table4 [7]. The ion existence

rate of hydrogen and hydrogen chloride released from wrap materials was 1.1×10^{-8} and 1.3×10^{-8} , respectively and it is found that the cutting efficiency of C-H bond is the same as that of C-Cl bond. These results suggest that the chemical bond whose energy is below UV energy will be cut with same efficiency by irradiating the UV of 172nm.

In case of graphite exposed to the acetylene glow discharge, the ion existence of hydrogen is 3.8×10^{-9} . As the cutting efficiency of the C-H and C-Cl was the same, the difference between the ion existence of hydrogen on wrap materials and that on the graphite surface would be resulted from the amount of hydrogen-carbon bonds.

4. Conclusion

To develop a new effective technique for tritium decontamination from surface of materials, the preliminary experiments for UV irradiation were performed, using poly vinyl chloride wrap, polyethylene film and graphite specimens discharged in acetylene glow.

At first, hydrogen based polymers were irradiated by UV light to confirm the possibility of UV light for tritium decontamination. Release of hydrogen and some hydrocarbon and hydrogen chloride were observed by UV irradiation.

In the experiment with poly vinyl chlorine wrap, chlorine gas was released from wrap and it is confirmed that UV light decomposes not only adsorbed species but also wrap substrate layers.

In case of polyethylene film, many kinds of hydrocarbons were released, including high mass number species, with the number of over 50. These species correspond to the C_4H_x ($X=7-9$) or C_5H_y ($Y=9-11$) based hydrocarbon.

The bond cutting efficiency is the same for 172nm UV to cut C-H and

C-Cl bonds. The deposited hydrogen on the graphite surface with carbon was liberated by the UV irradiation.

These results show that the UV light irradiation is a hopeful approach to decontaminate tritium from the surface of plasma-facing materials or other components of D-T fusion reactors.

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Table1 Typical mass peak intensities of wrap material

m/e	Before irradiation	During irradiation	After irradiation
2	1.86E-09	4.93E-09	1.14E-09
14	1.30E-10	3.22E-10	1.09E-10
15	2.30E-10	6.59E-10	1.91E-10
16	1.90E-10	3.74E-10	1.36E-10
17	2.78E-09	3.20E-09	1.96E-09
18	1.53E-08	1.76E-08	1.05E-08
27	1.80E-10	5.85E-10	1.00E-10
28	1.23E-09	2.94E-09	8.73E-10
29	3.80E-10	1.11E-09	3.36E-10
35	4.00E-11	2.01E-09	2.09E-10
36	6.00E-11	2.09E-08	2.42E-09
37	3.00E-11	6.50E-10	1.36E-10
38	4.00E-11	6.26E-09	7.36E-10
41	2.80E-10	7.01E-10	2.27E-10
43	3.70E-10	1.02E-09	3.73E-10
44	4.20E-10	1.26E-09	2.91E-10

Table2 Typical mass peak intensities of polyethylene film

m/e	Before irradiation	During irradiation	After irradiation
2	1.14E-09	1E-8 >	5.90E-10
14	5.90E-11	1.25E-10	4.50E-11
15	1.16E-10	3.60E-10	8.60E-11
16	6.50E-11	2.50E-10	4.80E-11
17	1.02E-09	9.07E-10	6.97E-10
18	5.48E-09	4.87E-09	3.80E-09
27	7.60E-11	7.44E-10	5.50E-11
28	6.29E-10	1.71E-09	4.29E-10
29	1.73E-10	1.06E-09	1.39E-10
30	3.80E-11	1.63E-10	2.00E-11
41	1.48E-10	1.04E-09	1.28E-10
42	5.50E-11	3.59E-10	2.50E-11
43	2.32E-10	1.28E-09	2.06E-10
44	1.79E-10	4.48E-10	1.48E-10
55	4.80E-11	3.64E-10	5.70E-11
56	5.30E-11	3.26E-10	2.80E-11
57	1.06E-10	5.40E-10	1.35E-10
69	2.20E-11	1.41E-10	2.40E-11
70	2.10E-11	1.21E-10	2.10E-11
71	3.10E-11	1.28E-10	3.80E-11

Table3 Typical mass peak intensities of graphite specimen

m/e	Before irradiation	During irradiation	After irradiation
2	4.19E-10	1.69E-09	2.62E-10
15	4.40E-11	1.14E-10	3.70E-11
16	2.20E-11	1.13E-10	2.80E-11
17	2.01E-10	3.40E-10	2.68E-10
18	1.10E-09	1.86E-09	1.47E-09
28	3.13E-10	6.63E-10	2.79E-10
29	4.00E-11	1.25E-10	3.70E-11
41	2.40E-11	5.44E-11	2.10E-11
42	2.00E-11	3.01E-11	1.90E-11
43	4.70E-11	7.81E-11	2.80E-11
44	6.90E-11	3.97E-10	9.80E-11
55	2.10E-11	2.51E-11	1.40E-11
56	1.70E-11	2.31E-11	1.70E-11
57	1.90E-11	2.88E-11	1.80E-11

Table4 Ionization probability table [7]

Substance	Formula	Relative ionization sensitivity
Hydrogen	H ₂	0.44
Hydrogen Chloride	HCl	1.6
Nitrogen	N ₂	1.0
Water	H ₂ O	1.0

Wave length / nm

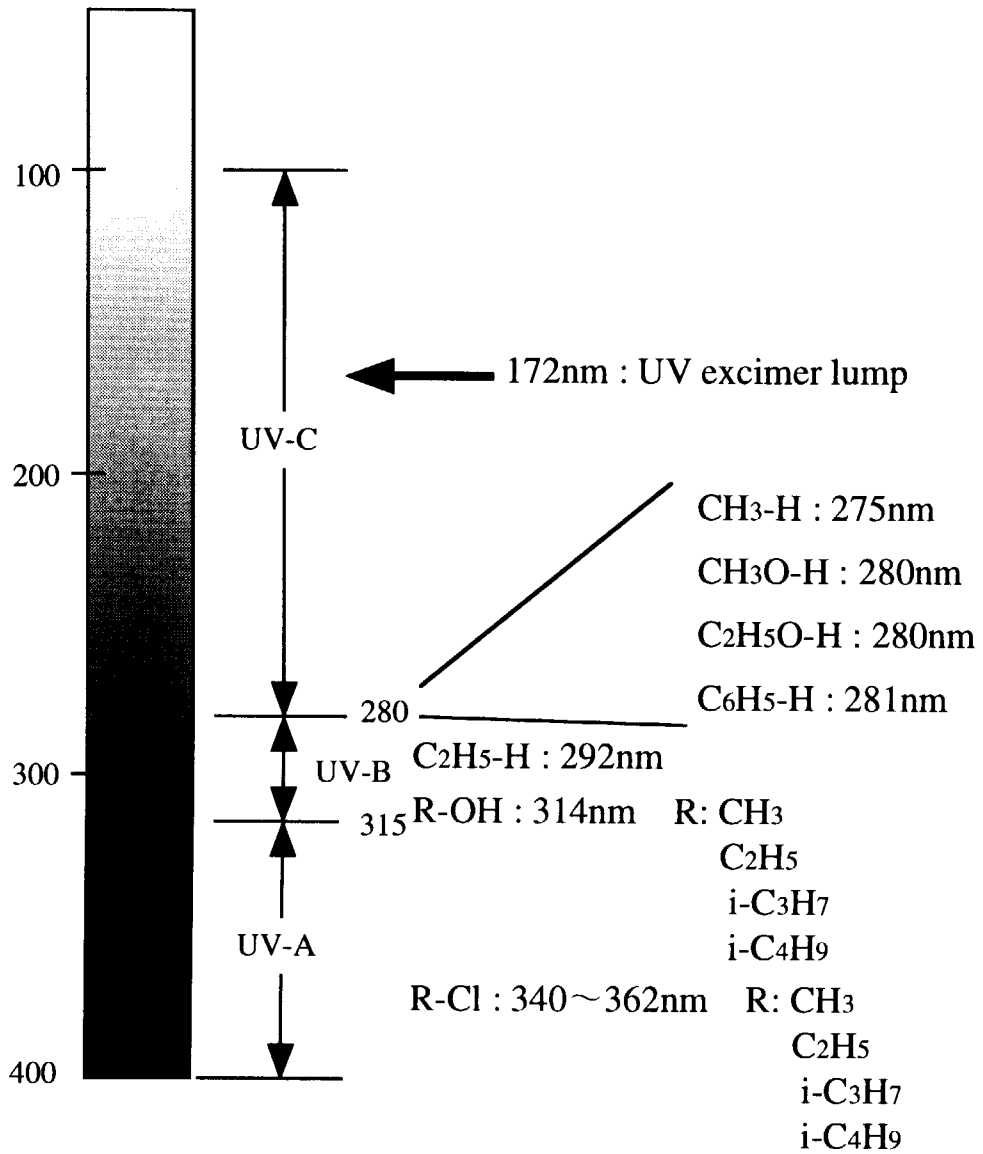


Fig.1 Bond dissociation energies for hydro-carbon compounds

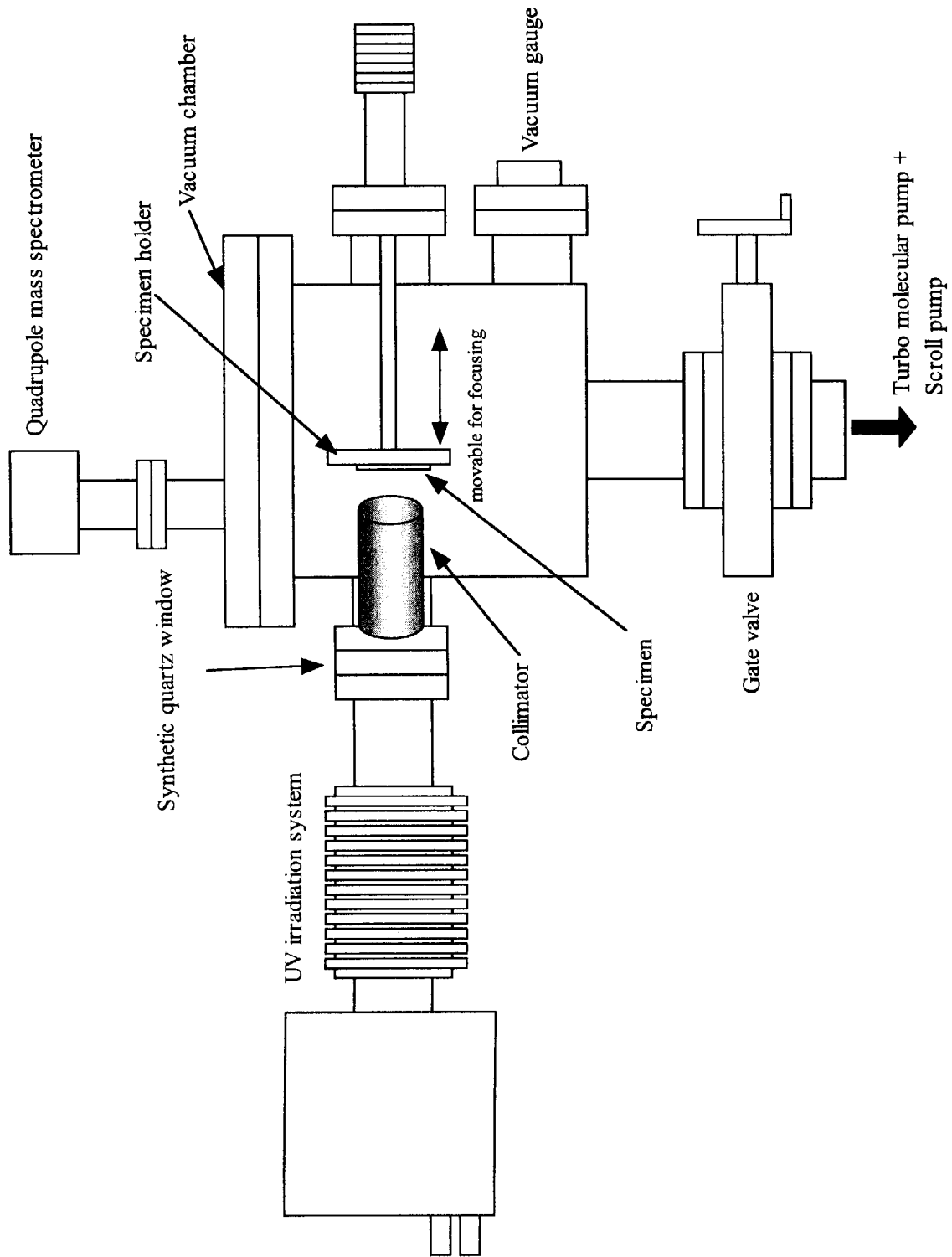


Fig.2 The schematic view of UV irradiation system

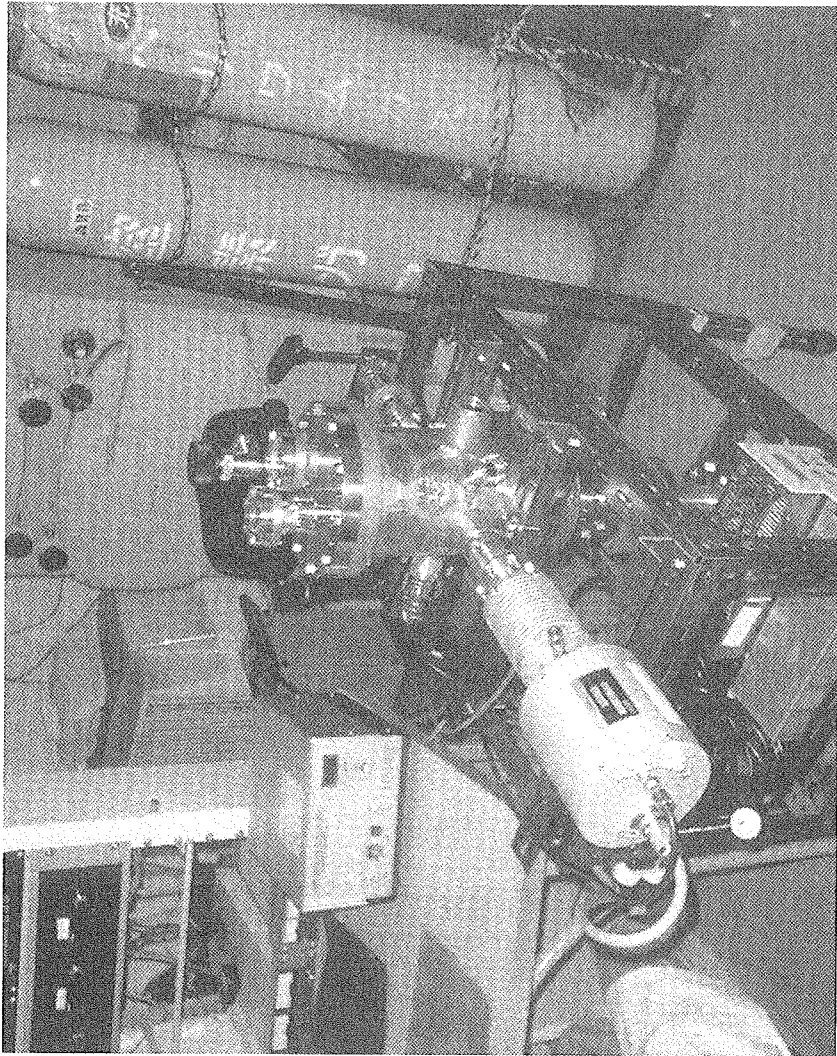


Fig.3 Picture of the experimental system

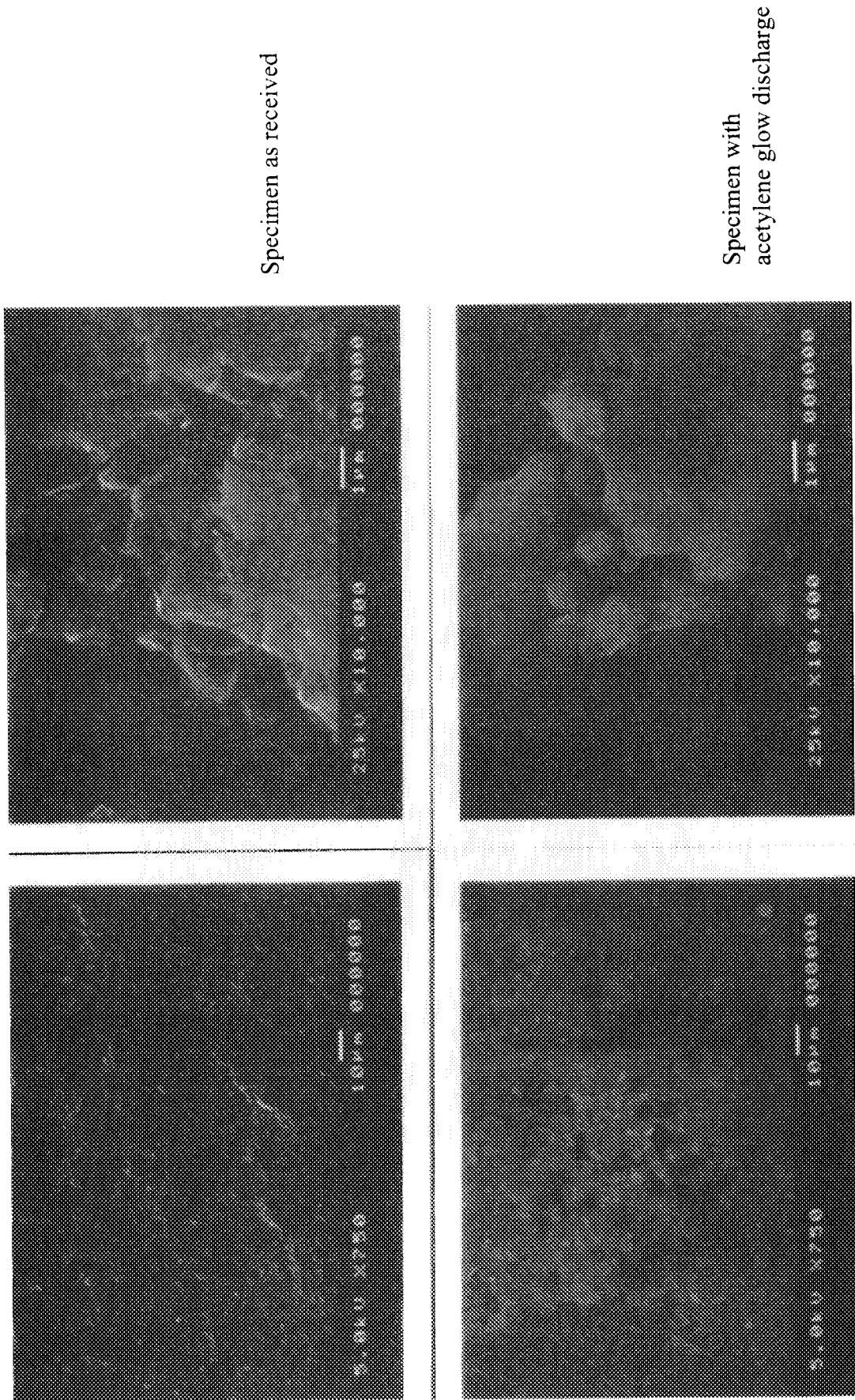


Fig.4 Pictures of graphite before and after the glow discharge

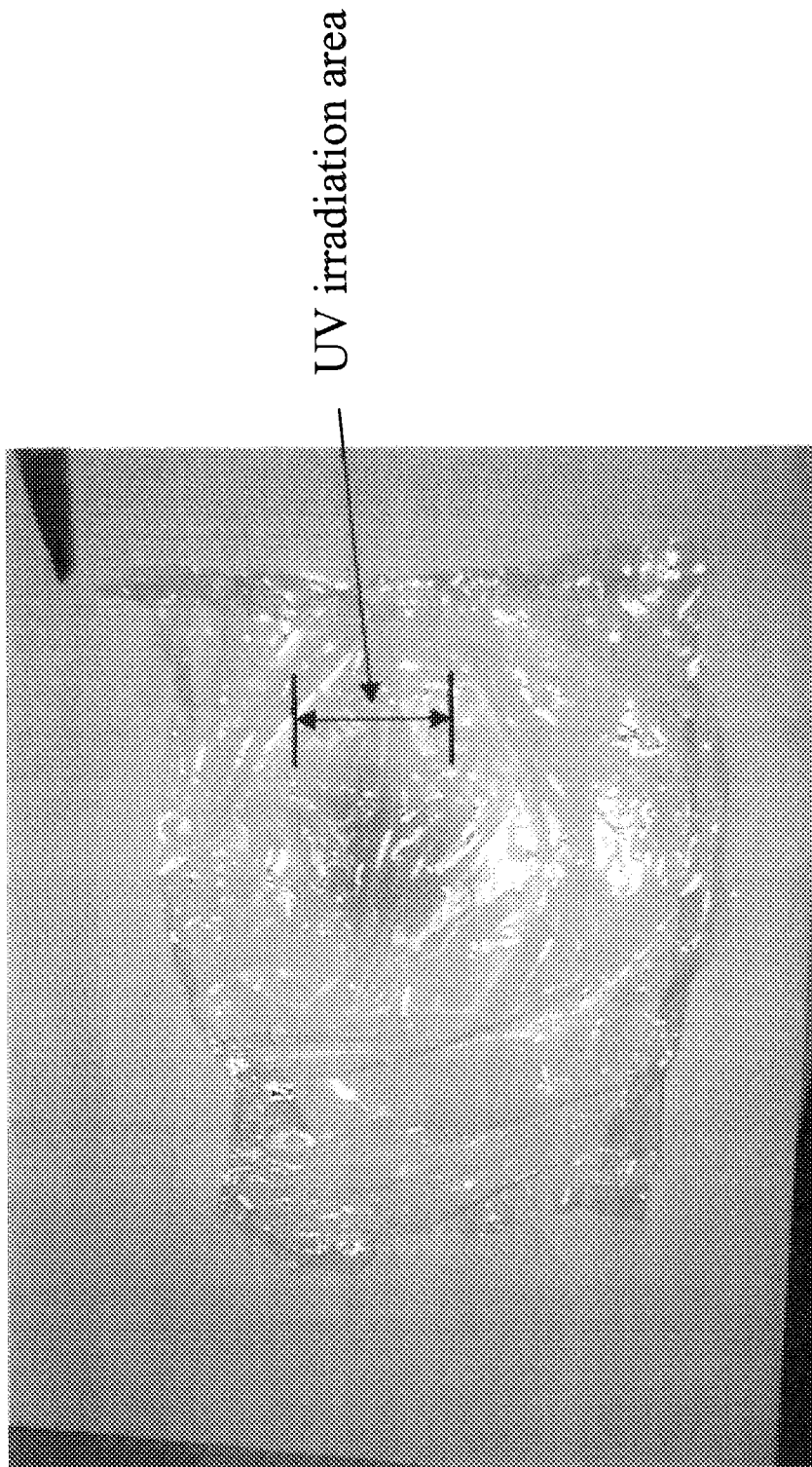


Fig.5 Pictures of the warp materials after UV irradiation

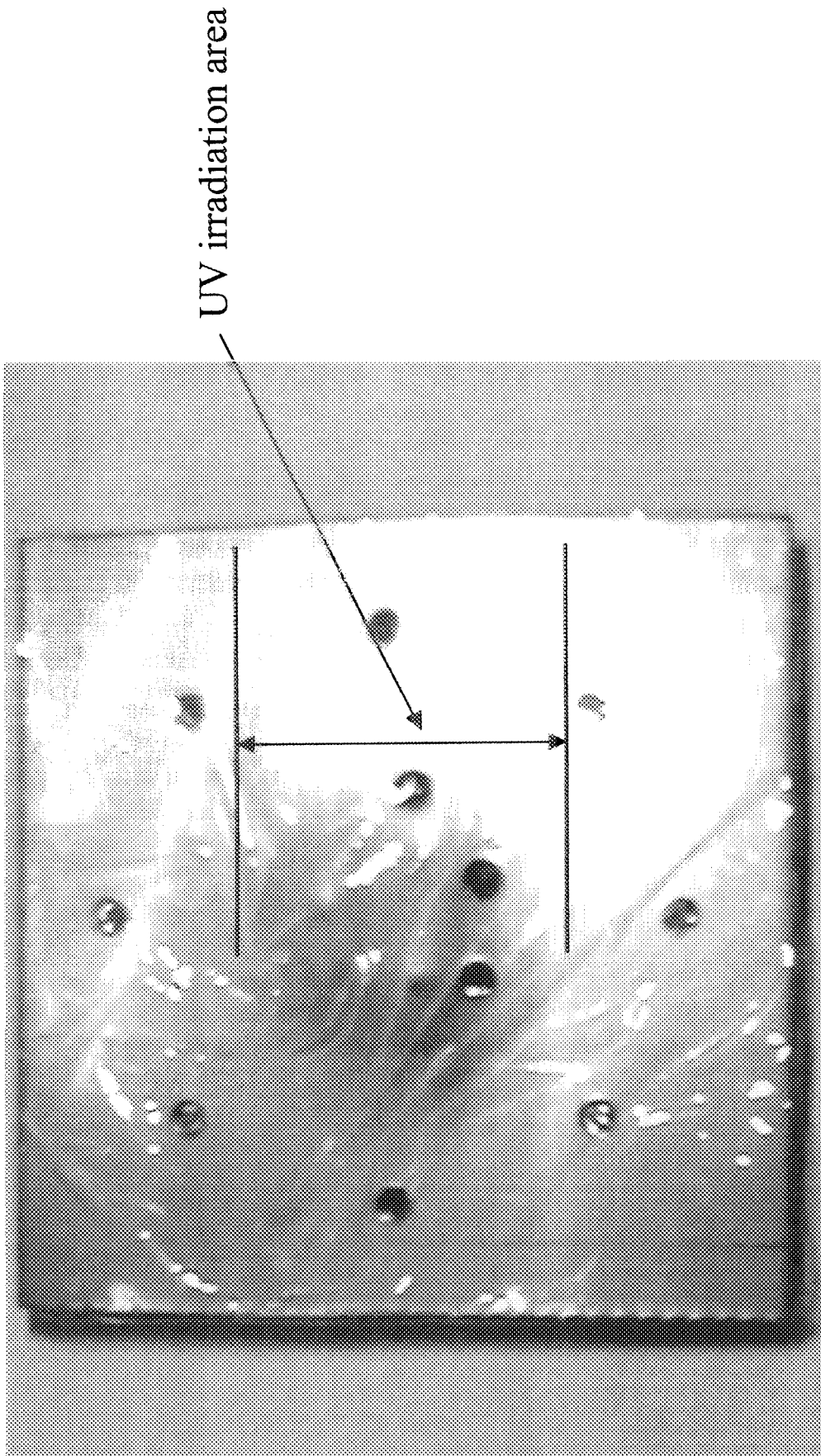


Fig.5 Pictures of the warp materials after UV irradiation

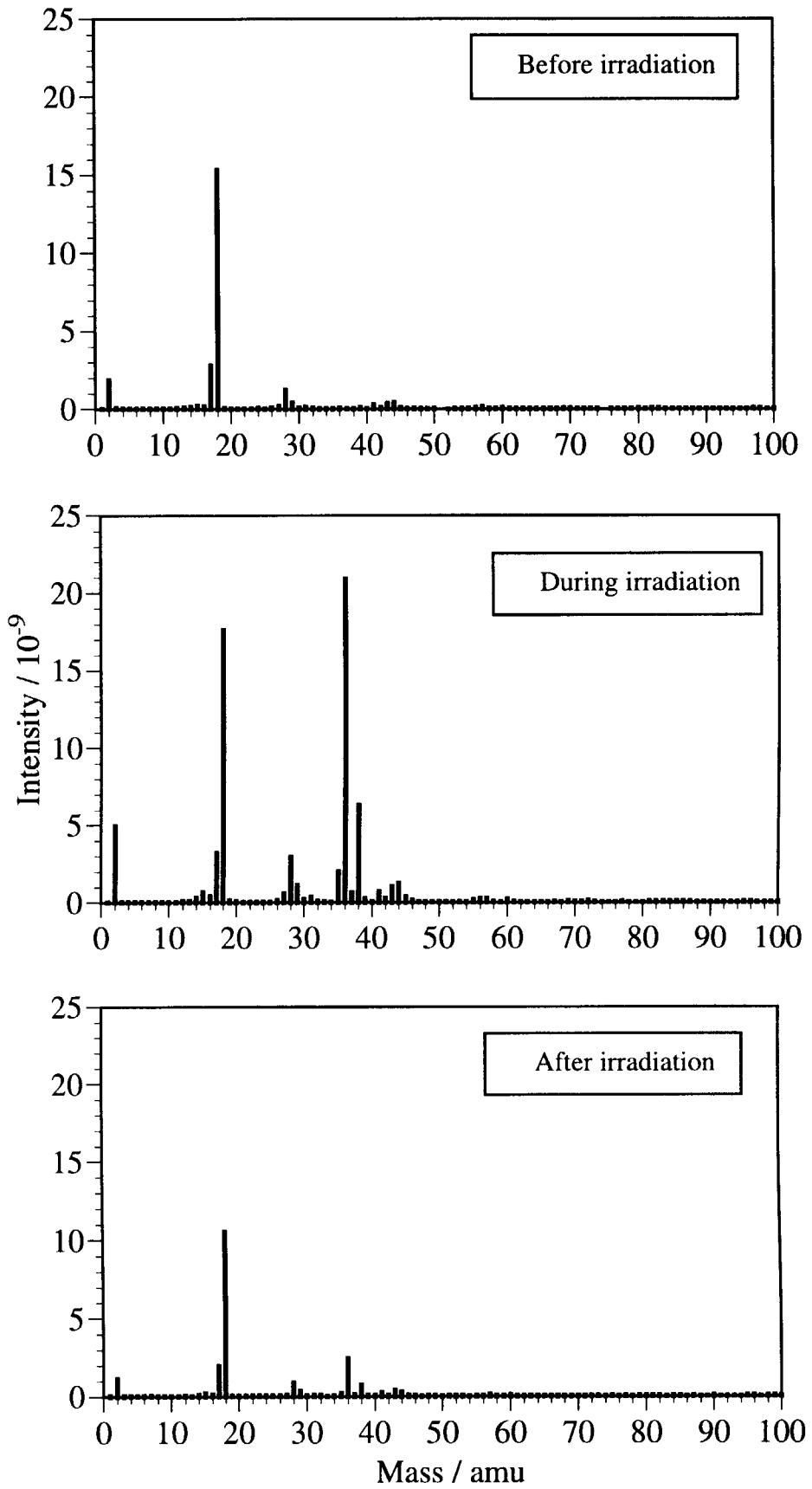


Fig.6 Mass spectra with UV irradiation to wrap material

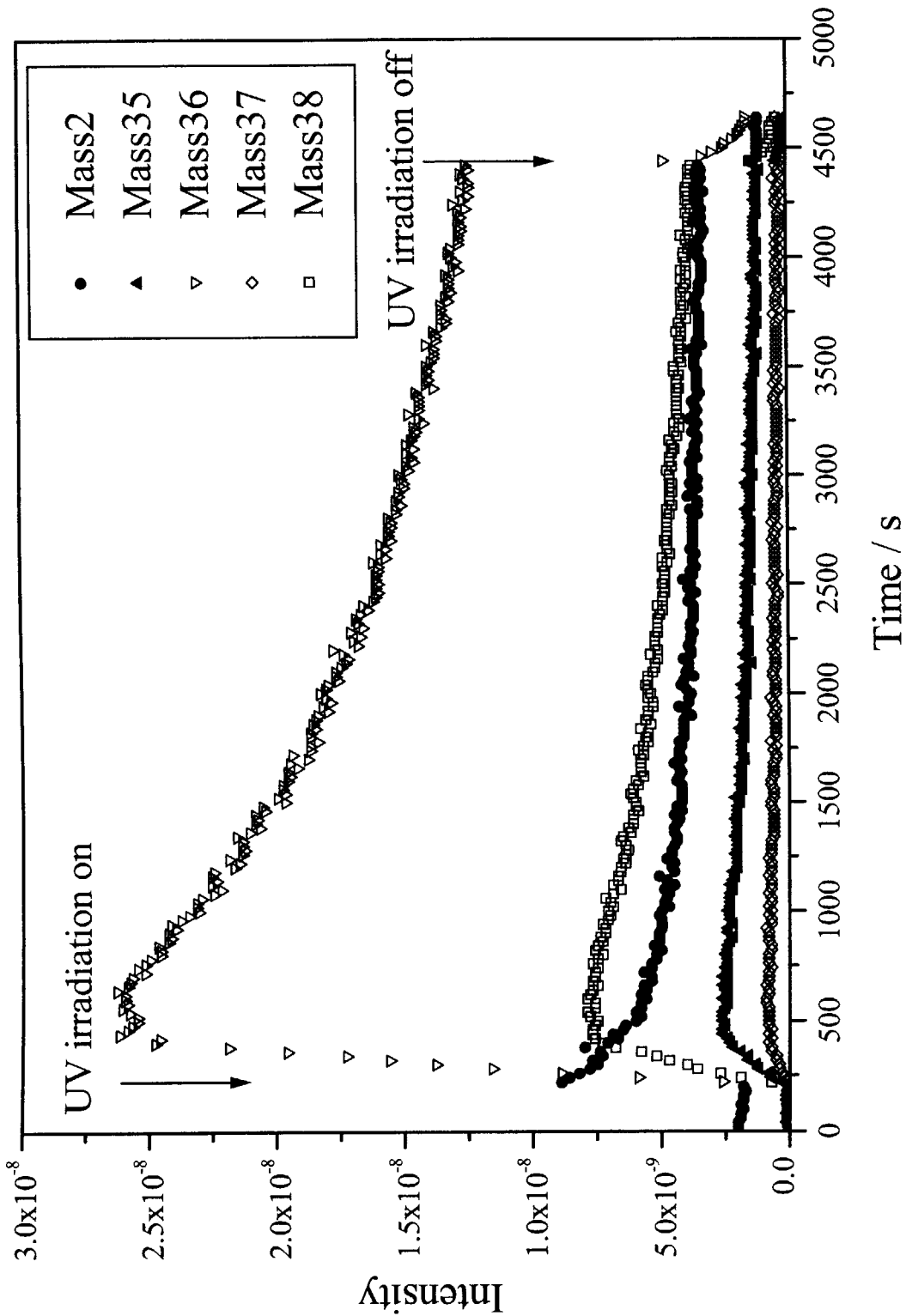


Fig.7 Time distribution of mass 2, 35, 36, 37 and 38 with UV irradiation (wrap material)

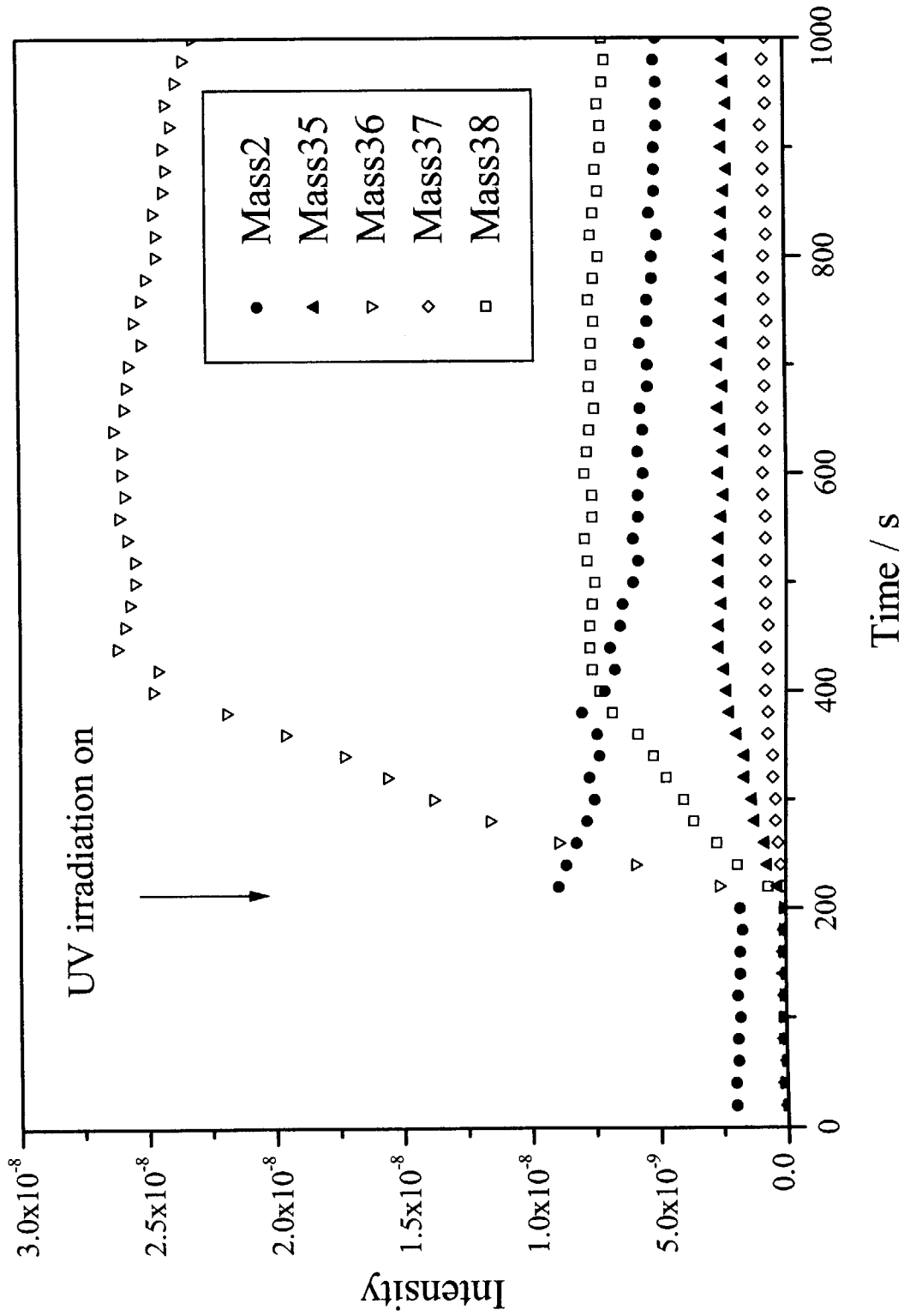


Fig.8 Time distribution of mass 2, 35, 36, 37 and 38 with UV irradiation expanded initial irradiation period of Fig.6.

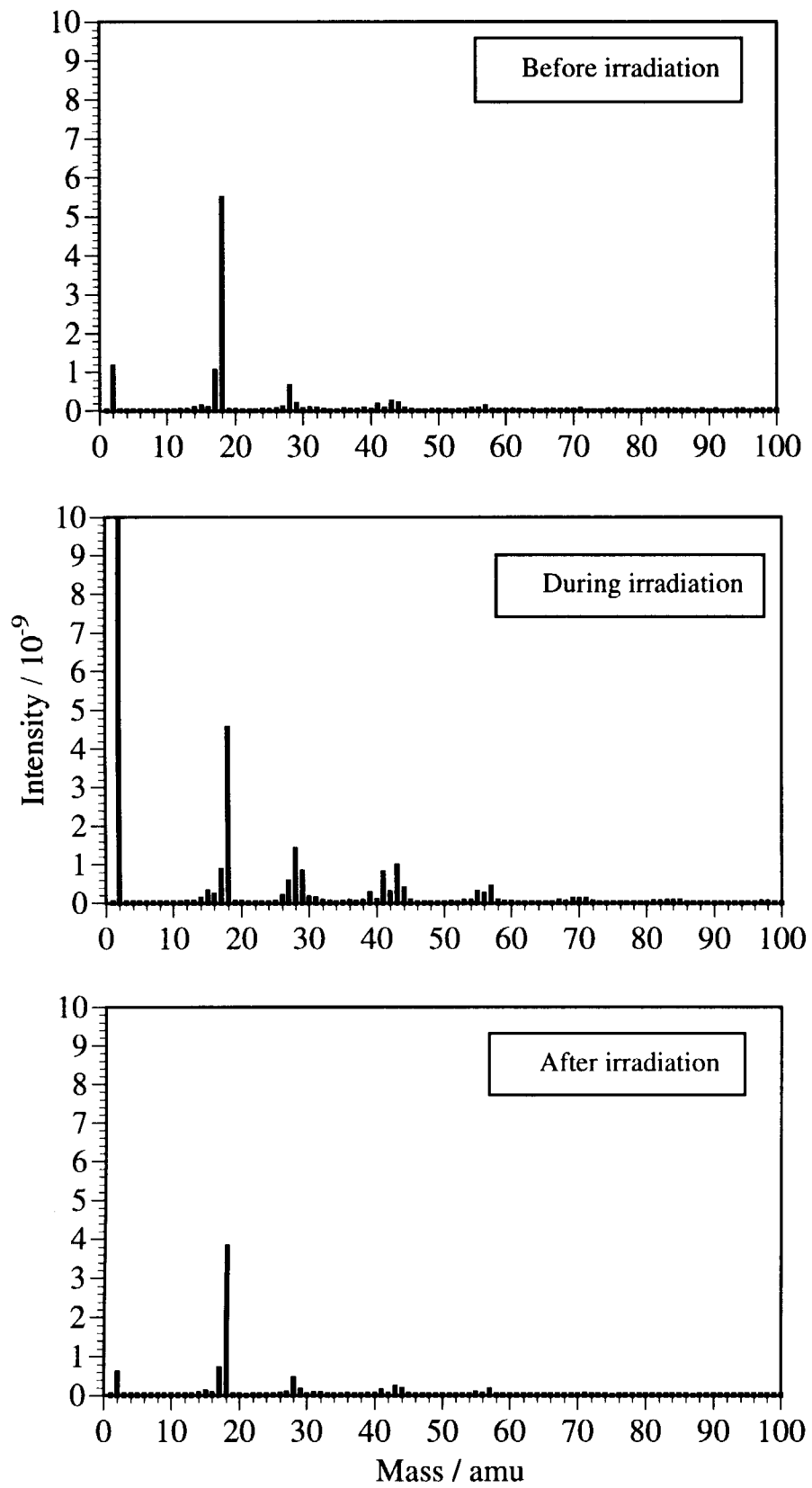


Fig.9 Mass spectra with UV irradiation to polyethylene film

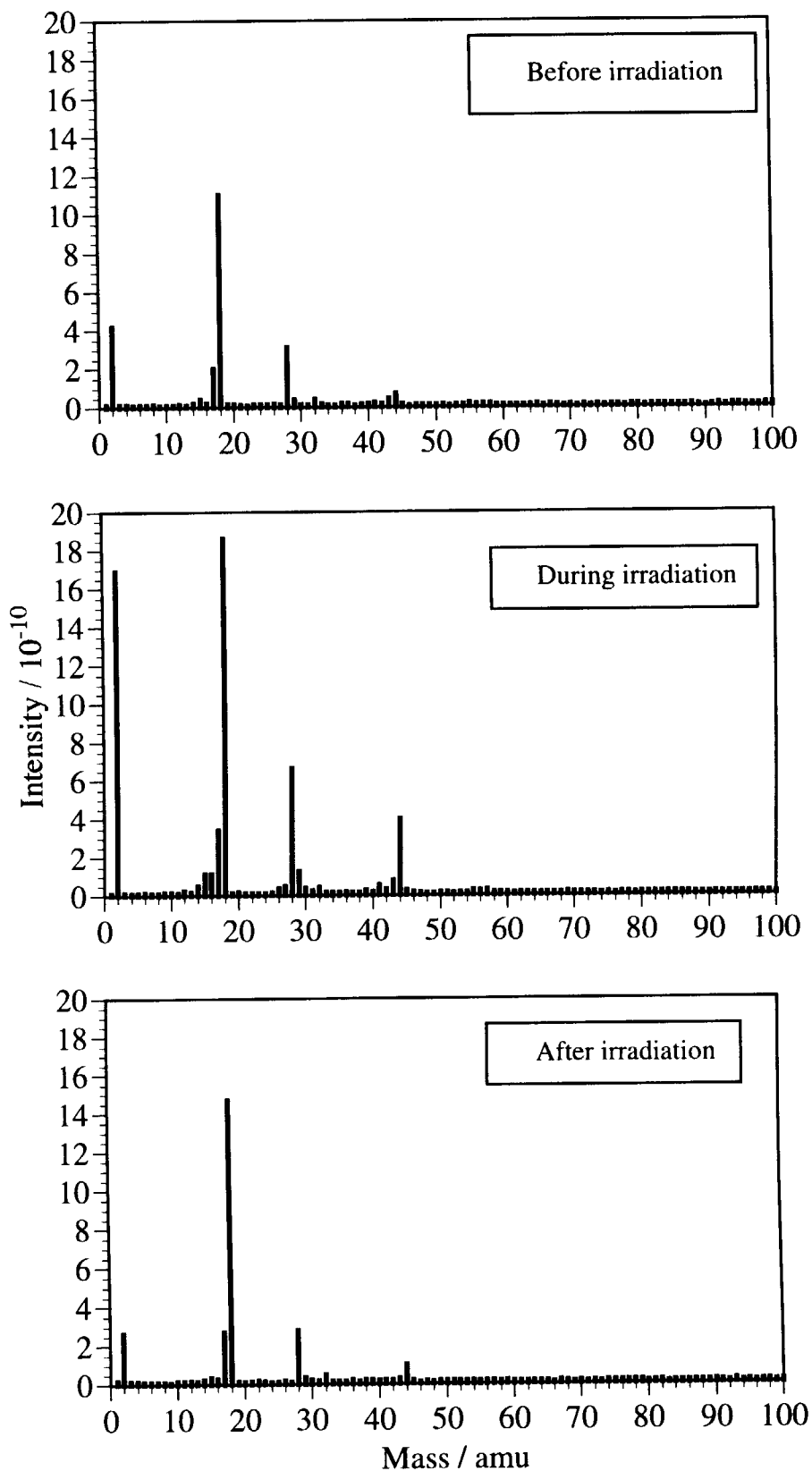


Fig.10 Mass spectra with UV irradiation to graphite specimen

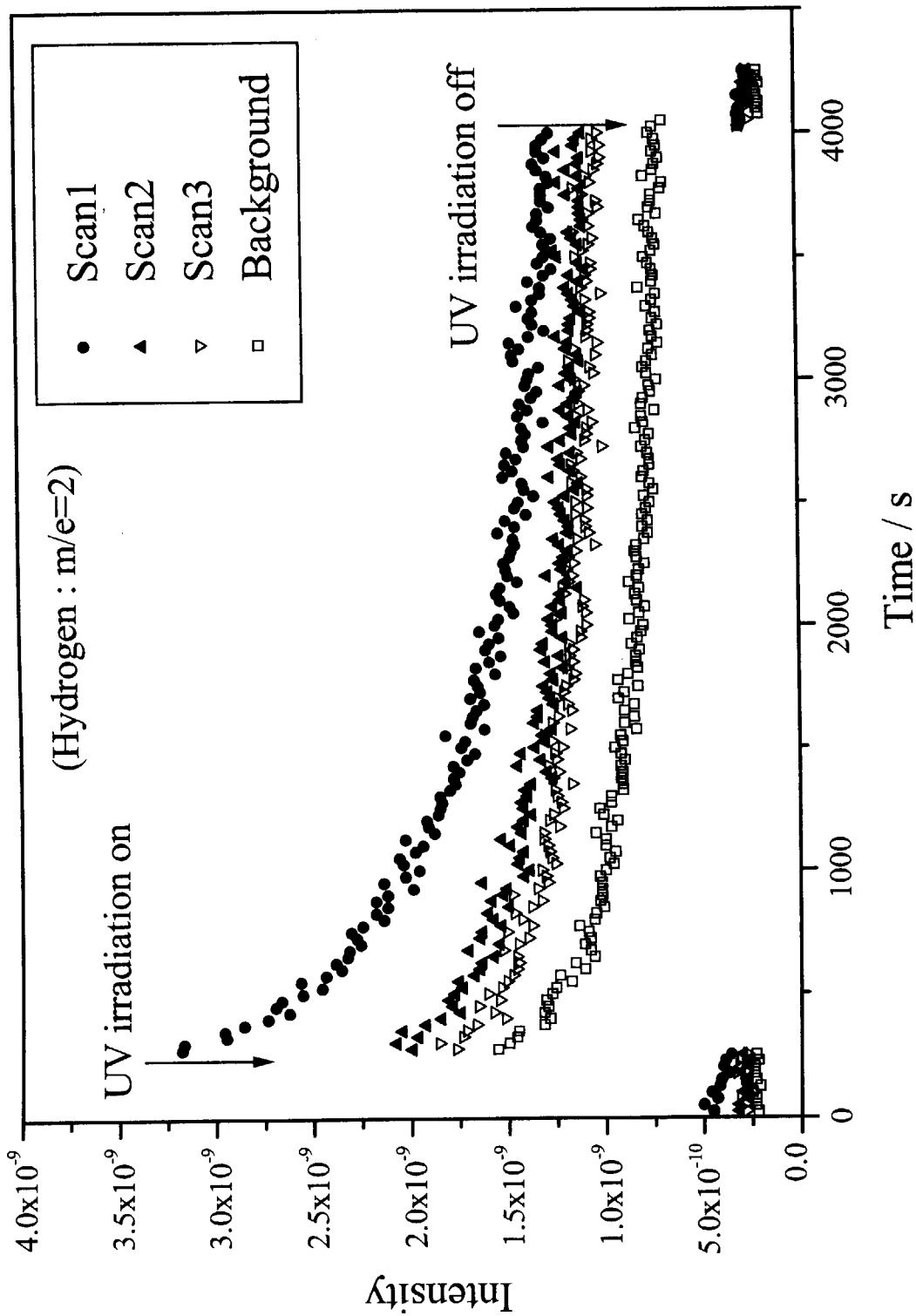


Fig.11 Time distribution of hydrogen with UV irradiation (graphite specimen)

国際単位系 (SI) と換算表

表1 SI基本単位および補助単位

量	名称	記号
長さ	メートル	m
質量	キログラム	kg
時間	秒	s
電流	アンペア	A
熱力学温度	ケルビン	K
物質質量	モル	mol
光度	カンデラ	cd
平面角	ラジアン	rad
立体角	ステラジアン	sr

表3 固有の名称をもつSI組立単位

量	名称	記号	他のSI単位による表現
周波数	ヘルツ	Hz	s ⁻¹
力	ニュートン	N	m·kg/s ²
圧力, 応力	パスカル	Pa	N/m ²
エネルギー, 仕事, 熱量	ジュール	J	N·m
工率, 放射束	ワット	W	J/s
電気量, 電荷	クーロン	C	A·s
電位, 電圧, 起電力	ボルト	V	W/A
静電容量	ファラド	F	C/V
電気抵抗	オーム	Ω	V/A
コンダクタンス	ジーメンス	S	A/V
磁束	ウェーバ	Wb	V·s
磁束密度	テスラ	T	Wb/m ²
インダクタンス	ヘンリー	H	Wb/A
セルシウス温度	セルシウス度	°C	
光束	ルーメン	lm	cd·sr
照射度	ルクス	lx	lm/m ²
放射線量	ベクレル	Bq	s ⁻¹
吸収線量	グレイ	Gy	J/kg
線量当量	シーベルト	Sv	J/kg

表2 SIと併用される単位

名称	記号
分, 時, 日	min, h, d
度, 分, 秒	°, ', "
リットル	l, L
トン	t
電子ボルト	eV
原子質量単位	u

1 eV = 1.60218 × 10⁻¹⁹ J
1 u = 1.66054 × 10⁻²⁷ kg

表4 SIと共に暫定的に維持される単位

名称	記号
オングストローム	Å
バ	b
バール	bar
ガリ	Gal
キュリ	Ci
レントゲン	R
ラド	rad
レム	rem

1 Å = 0.1 nm = 10⁻¹⁰ m
1 b = 100 fm = 10⁻²⁸ m²
1 bar = 0.1 MPa = 10⁵ Pa
1 Gal = 1 cm/s² = 10⁻² m/s²
1 Ci = 3.7 × 10¹⁰ Bq
1 R = 2.58 × 10⁻⁴ C/kg
1 rad = 1 cGy = 10⁻² Gy
1 rem = 1 cSv = 10⁻² Sv

表5 SI接頭語

倍数	接頭語	記号
10 ¹⁸	エクサ	E
10 ¹⁵	ペタ	P
10 ¹²	テラ	T
10 ⁹	ギガ	G
10 ⁶	メガ	M
10 ³	キロ	k
10 ²	ヘクト	h
10 ¹	デカ	da
10 ⁻¹	デシ	d
10 ⁻²	センチ	c
10 ⁻³	ミリ	m
10 ⁻⁶	マイクロ	μ
10 ⁻⁹	ナノ	n
10 ⁻¹²	ピコ	p
10 ⁻¹⁵	フェムト	f
10 ⁻¹⁸	アト	a

(注)

- 表1-5は「国際単位系」第5版, 国際度量衡局 1985年刊行による。ただし, 1 eV および 1 uの値は CODATA の 1986年推奨値によった。
- 表4には海里, ノット, アール, ヘクタールも含まれているが日常の単位なのでここでは省略した。
- bar は, JISでは流体の圧力を表わす場合に限り表2のカテゴリーに分類されている。
- EC閣僚理事会指令では bar, barn および「血圧の単位」mmHgを表2のカテゴリーに入れている。

換算表

力	N (=10 ⁵ dyn)	kgf	lbf
	1	0.101972	0.224809
	9.80665	1	2.20462
	4.44822	0.453592	1

粘度 1 Pa·s (N·s/m²) = 10 P (ポアズ) (g/(cm·s))

動粘度 1 m²/s = 10⁴ St (ストークス) (cm²/s)

圧	MPa (=10 bar)	kgf/cm ²	atm	mmHg (Torr)	lbf/in ² (psi)
	1	10.1972	9.86923	7.50062 × 10 ³	145.038
力	0.0980665	1	0.967841	735.559	14.2233
	0.101325	1.03323	1	760	14.6959
	1.33322 × 10 ⁻⁴	1.35951 × 10 ⁻³	1.31579 × 10 ⁻³	1	1.93368 × 10 ⁻²
	6.89476 × 10 ⁻³	7.03070 × 10 ⁻²	6.80460 × 10 ⁻²	51.7149	1

エネルギー・仕事・熱量	J (=10 ⁷ erg)	kgf·m	kW·h	cal (計量法)	Btu	ft·lbf	eV
	1	0.101972	2.77778 × 10 ⁻⁷	0.238889	9.47813 × 10 ⁻⁴	0.737562	6.24150 × 10 ¹⁸
	9.80665	1	2.72407 × 10 ⁻⁶	2.34270	9.29487 × 10 ⁻³	7.23301	6.12082 × 10 ¹⁹
	3.6 × 10 ⁶	3.67098 × 10 ⁵	1	8.59999 × 10 ⁵	3412.13	2.65522 × 10 ⁶	2.24694 × 10 ²⁵
	4.18605	0.426858	1.16279 × 10 ⁻⁶	1	3.96759 × 10 ⁻³	3.08747	2.61272 × 10 ¹⁹
	1055.06	107.586	2.93072 × 10 ⁻⁴	252.042	1	778.172	6.58515 × 10 ²¹
	1.35582	0.138255	3.76616 × 10 ⁻⁷	0.323890	1.28506 × 10 ⁻³	1	8.46233 × 10 ¹⁸
	1.60218 × 10 ⁻¹⁹	1.63377 × 10 ⁻²⁰	4.45050 × 10 ⁻²⁶	3.82743 × 10 ⁻²⁰	1.51857 × 10 ⁻²²	1.18171 × 10 ⁻¹⁹	1

1 cal = 4.18605 J (計量法)
= 4.184 J (熱化学)
= 4.1855 J (15 °C)
= 4.1868 J (国際蒸気表)
仕事率 1 PS (仏馬力)
= 75 kgf·m/s
= 735.499 W

放射能	Bq	Ci
	1	2.70270 × 10 ⁻¹¹
	3.7 × 10 ¹⁰	1

吸収線量	Gy	rad
	1	100
	0.01	1

照射線量	C/kg	R
	1	3876
	2.58 × 10 ⁻⁴	1

線量当量	Sv	rem
	1	100
	0.01	1

RESULTS OF PRELIMINARY EXPERIMENTS ON TRITIUM DECONTAMINATION BY UV IRRADIATION