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ABSTRACT

Deuterium plasma recycling and chemical erosion behavior of selected graphite materials have been investigated using the PISCES-A facility. These materials include: Pyro-graphite; 2D-graphite weave; 4D-graphite weave; and POCO-graphite. Deuterium plasma bombardment conditions are: fluxes around 7×10^{17} ions $s^{-1}cm^{-2}$; exposure time in the range from 10 to 100 s; bombarding energy of 300 eV; and graphite temperatures between 20 and 120°C. To reduce deuterium plasma recycling, several approaches have been investigated. Erosion due to high-fluence helium plasma conditioning significantly increases the surface porosity of POCO-graphite and 4D-graphite weave whereas little changes for 2D-graphite weave and Pyro-graphite. The increased pore openings and refreshed in-pore surface sites are found to reduce the deuterium plasma recycling and chemical erosion rates at transient stages. The steady state recycling rates for these graphite materials can be also correlated to the surface porosity. Surface topographical modification by machined-grooves noticeably reduces the steady state deuterium recycling rate and the impurity emission from the surface. These surface topography effects are attributed to co-deposition of reemitted deuterium, chemically sputtered hydrocarbon and physically sputtered carbon under deuterium plasma bombardment. The co-deposited film is found to have a characteristic surface morphology with dendritic microstructures.

1. INTRODUCTION

Recycling of hydrogen isotopes from plasma-facing components in a magnetic fusion device is widely recognized as a crucial issue affecting the fueling scenario and the on-site tritium inventory. Particularly, wall pumping effects to reduce fuel particle recycling by graphite components such as a limiter and divertor plate have been recently observed in major tokamaks: TFTR [1]; JET [2]; DIII-D [3]; and TEXTOR [4] and have generated considerable interest. Except for the case of JET, the surface conditioning of these plasma-facing components plays a crucial role in obtaining the wall pumping effect.

In our previous work [5], the helium or argon plasma-activation processes (He, Ar-PAP) were established as new surface conditioning techniques for low-density, isotropic graphite materials. The activated graphite surface showed a sponge-like morphology with significantly increased open pores and a large surface gas adsorption capacity. Using activated graphite, the first non-tokamak wall pumping experiments were conducted for a short plasma exposure duration of 20s to simulate fusion devices operating in the short pulse mode. The increased surface porosity and gas adsorptivity due to He-PAP were found to enhance retention of hydrogen plasma particles and hence provided a significant plasma pumping effect at temperatures between 20 and 480°C.

Here, one must notice that these wall pumping effects are observed only up to the point when the plasma-facing surface becomes saturated with fuel particles. These saturable pumping effect thus will not contribute significantly to the control of the particle balance in future long-pulse fusion devices such as TIBER [6]. For possible applications in the future devices, reinforced graphite materials such as C-C composites are currently under development. Yet little is understood about the hydrogen recycling behavior of these new materials.

In the present work, several possibilities to reduce particle recycling both at the transient stages and at the steady state have been investigated using the PISCES-A facility [7]: (1) helium plasma conditioning to increase surface porosity; and (2) surface topographical modification to induce particle retrapping. Used for this purpose are selected graphite materials including: Pyro-graphite (Pfizer); 2D-graphite weave (BF-Goodrich); 4D- graphite weave (FMI); and POCO-graphite (grade: HPD-1).

2. EXPERIMENTAL

After ultrasonic cleaning in acetone, a disk target of graphite with a diameter of 4.5 cm and with a thickness of 6 mm was placed in steady-state helium and deuterium plasmas in the PISCES-A facility. The plasma diameter was set around 6 cm. The temperature of the graphite target during plasma exposure was measured both by a thermocouple and an infra-red pyrometer. The plasma density and the electron temperature were measured using a Langmuir probe positioned in front of the target. A 1.3m monochromator with an optical multi-channel analyzer (OMA) was aligned in such a way that plasma radiations (e.g. D-alpha) could be monitored, particularly near the specimen surface. Details of the PISCES-A facility and materials experiments in this facility can be found elsewhere [7,8].

The graphite target was first activated in a helium plasma at temperatures around 800°C (He-PAP) and was then exposed to a deuterium plasma for recycling measurements with calibrated D-alpha spectroscopy. The deuterium plasma exposure time was varied from 10 to 100 s. Simultaneously with recycling measurements, chemical erosion behavior due to deuterium plasma bombardment was monitored using a differentially pumped residual gas analyzer (RGA).

Plasma bombardment conditions are generally: flux in the range from 5×10^{17} to 1×10^{18} ions s^{-1} cm^{-2}; bombarding energies of 100 and 300 eV for helium and deuterium plasmas, respectively; plasma density of the order of 10^{11} cm^{-3}; electron temperature in the range from 5 to 15 eV; and neutral pressure of the order of 10^{-5} - 10^{-4} torr. Here, the bombarding energy is controlled by applying a negative DC-bias to the target in addition to the floating potential because the intrinsic ion energy in the PISCES plasma is about a few electron volts. The magnetic field is perpendicular to the target surface and is about 800 gauss. Under these conditions, the ion gyro-radii are 2 to 3 mm. The thickness of the sheath region is around 10μm. Therefore, ions accelerated by the total sheath potential (floating potential plus negative bias) will strike on the surface at the approximately normal incidence.

During plasma bombardment, important experimental parameters: ion saturation current from the Langmuir probe (i.e. plasma bombardment flux to the target surface); D-alpha intensity; signals from RGA; and target temperature were monitored at sampling rates of 1-10 Hz. To avoid complicated effects associated with redeposition, the plasma parameters were controlled to generate conditions in the erosion regime [8] for both physically sputtered carbon and chemically sputtered hydrocarbons (e.g. CD_4).

4. RESULTS AND DISCUSSION

4-1. Basic considerations

In the present work, the deuterium recycling behavior is analyzed using calibrated D-alpha spectroscopy. The plasma density is below 10^{13} cm^{-3}, the brightness of D-alpha light, B, is related to plasma parameters through the relation [9]:

$$B = k\, n_e n_D \langle \sigma v \rangle \qquad (1)$$

where k is the calibration constant, n_e is the plasma density, n_D is the density of atomic deuterium and $\langle \sigma v \rangle$ is the rate coefficient for the reaction: $D + e^- \Rightarrow D^+ + 2e^-$. The rate coefficient is calculated from Lotz's formula [10]. One can evaluate n_D from eq.(1) using the plasma density and electron temperature from Langmuir probe measurements. Also, n_D is considered as an approximate indicator of the density of molecular deuterium reemitted from the surface.

Here, we define the recycling coefficient, R, as

$$R \equiv K n_D / \Gamma_{ion} \qquad (2)$$

where K is the unit conversion constant to make R dimensionless, and Γ_{ion} is the flux. It is important mention here that the effects of the start-up machine time constant and/or fluctuations of the flux on the recycling coefficient are cancelled by definition.

4-2. Effects of He-plasma conditioning on the surface morphology

The graphite target was first bombarded with a helium plasma to a fluence of the order of 10^{21} ions/cm^2 at a bombarding energy of 100 eV and at elevated temperatures around 800°C (i.e. He-PAP). The basal plane of Pyro-graphite and the weave plane of 2D-graphite weave were exposed to the helium plasma. The surface morphology after He-PAP was observed with scanning electron microscopy (SEM).

The results of SEM observations are shown in Fig. 1. Sponge-like surface morphologies with significantly increased pore openings are found for POCO-graphite and

4D-graphite weave. (Not shown here are the surface morphologies of these materials before He-PAP: the surface pores are blanked off by machining dusts or weekly bound particles.) One can notice that POCO-graphite exhibits relatively small but high density pores whereas the opposite is true for 4D-graphite weave. The sponge-like surface morphology observed for POCO-graphite is found to be similar to that observed for graphite limiter tiles used in TFTR for a long term (≈2 years). In our previous work [5], the surface roughness of POCO-graphite after He-PAP was estimated to be about 345 from molecular adsorption measurements. From the observed surface morphology, one may expect a similar or somewhat smaller surface roughness factor for activated 4D-graphite weave. For Pyro-graphite and 2D-graphite weave, in contract, no obvious surface pore openings are observed.

4-3. Recycling and erosion behavior of graphite materials

In this series of experiments, a graphite target specimen was mounted on a water-cooled copper manipulator. One can consider this setup as simulation of an actively water-cooled plasma-facing component. The plasma exposure time was about 10s. The activated graphite target was bombarded with a deuterium plasma at at a bombarding energy of 300 eV. The flux was set at 7×10^{17} ions s^{-1} cm^{-2}. Using the particle reflection coefficient of 0.16 obtained from TRIM code [11], the net incoming flux is calculated to be 6×10^{17} ions s^{-1} cm^{-2}. Due to the heat flux of 0.3 MW/m^2, the target temperature rises up to temperatures around 70°C, as shown in Fig. 2-a. These conditions are believed to be relevant to those for plasma interactions with graphite bumper limiters in TFTR [12].

Typical results of recycling measurements for 4D-graphite weave are shown in Fig. 2-b and those for other materials are summarized in Table 1. The activated surface has yielded a significantly reduced recycling coefficient, relative to the pre-saturated surface. (The pre-saturated surface is defined as the surface previously subjected to He-PAP followed by high-fluence deuterium plasma bombardment [5].) The recycling curve for the activated surface merges the curve for the pre-saturated surface after about 30 second deuterium plasma exposure. Not shown here is the recycling coefficient from the surface before He-PAP, which is essentially the same as the one for Pyro-graphite but is fluence dependent because of the gradual opening of pores under plasma bombardment. The

recycling coefficient for the pre-saturated surface tends to peak first and then gradually decrease to the steady state level, presumably due to ion-impact desorption. The pre-saturated surface can be easily reactivated by helium plasma bombardment to a fluence of the order of 10^{19} ions/cm^2.

It is important to mention here that deuterium recycling apparently reaches a steady state after transient stages of pumping even as the surface temperature changes. Generally, retention of hydrogen isotopes in graphite decreases with increasing surface temperature, and significant thermal desorption is often seen to start at temperatures around 250°C [13]. However, the temperature at the end of plasma exposure is still low enough to retain deuterium.

Notice in Table 1 that the recycling coefficients for activated materials at the steady state are found to decrease with increasing surface porosity (see Fig. 1). This clearly indicates that increased surface pore openings and refreshed in-pore surface adsorption sites by He-PAP play an important role in determining the effective deuterium retention capacity. Also, the recycling coefficients for the pre-saturated surfaces remains below unity. This can be attributed to an in-pore storage mechanism that deuterium temporally resides in pores under plasma bombardment. (Details of the in-pore storage mechanism will be discussed in the next section.) Deuterium, once stored in pores of the pre-saturated surface, may be released after plasma bombardment.

In Fig 2-c, the signal from RGA set for CD_4 is seen to track with the recycling coefficient. Generally, steady state chemical erosion of graphite requires surface saturation with implanted hydrogen isotopes [14]. Consistently, the activated surface with an increased deuterium retention capacity is observed to exhibit significantly retarded erosion behavior to reach the steady state, relative to the pre-saturated surface. It follows that one can avoid hydrocarbon formation by He-PAP in fusion devices having short plasma discharge durations of 2 - 3 sec. A similar result was obtained in our previous work using an in-situ spectroscopic technique [5].

One finds in Table 1 that Pyro-graphite has yielded a higher CD_4 signals than other materials. This may be due either to the fact that the temperature of Pyro-graphite is higher than those for other materials or to the fact that the flat and pore-free surface does not provide any in-pore storage effects. Unfortunately, these possibilities can not be differentiated at present.

From these observations, one postulates that the surface pore openings increase penetration of particles arriving from the plasma and then activated in-pore surface adsorption sites trap these particles after kinematical slowdown due to collisions with the in-pore walls. To understand this process, one must consider the plasma effects: both ions and neutrals are arriving at the surface. Here, deuterium ions are accelerated to the bombarding energy set at 300 eV. The neutrals include molecular deuterium with thermal energies and atomic deuterium with energies ranging a few to 10 eV due to dissociation due to atomic processes (e.g. Frank-Condon process [15]).

Generally, molecular deuterium will not significantly contribute to the surface adsorption process because of its inertness to graphite. Atomic neutrals can be directly transported into pores to an appreciable depth before they come to rest upon adsorption because the particle and energy reflection coefficients maximize at energies around a few electron volts [15]. In contrast, deuterium ions can not directly penetrate deep into pores, as they are, because of the gyration effect (see section 2). However, they will eventually saturate a local area on the in-pore wall and then eject neutral deuterium by ion-impact desorption. Neutral deuterium generated by the ion-impact desorption process generally has energies of a few electron volts and will behave similarly to that transported directly from the plasma. Unfortunately, it is difficult to differentiate these effects individually.

One may extend these arguments to the case when in-pore surface adsorption sites are pre-occupied with deuterium. This case corresponds to the pre-saturated surface. Under deuterium plasma bombardment, atomic deuterium may penetrate even deeper without being trapped due to adsorption and may not be transported outward unless effective backscattering occurs. This effect can be seen reproducibly so long as the surface is porous. Untrapped deuterium will be released, presumably as molecules after the plasma bombardment is stopped or when the flux decreases abruptly. The in-pore storage effect seen for the pre-saturated surface is believed to be explained by this mechanism. The in-pore storage mechanism might in part contribute the wall pumping effect observed in JET [2].

Here, it is important to note that deuterium trapped in the in-pore surface space will be replaced easily with other molecules having more stable adsorption characteristics, for example, with water vapor molecules upon air-exposure. From these arguments, thermal desorption experiments after plasma bombardment may not yield consistent spectra.

4-5. Effect of surface grooves on recycling behavior

As seen in section 4-3, POCO-graphite with the sponge-like surface morphology has yielded the lowest steady state recycling coefficient. As an attempt to further reduce the recycling coefficient by particle retrapping, a POCO-graphite target was topographically modified with machined-grooves. The grooved surface was first subjected to He-PAP and then to deuterium plasma bombardment under similar conditions to those described in the previous section. The deuterium plasma exposure time is extended to 100s to ensure the effect of surface grooves. Towards the end of the extended plasma exposure, the surface temperature rises up to about 120°C, as shown in Fig. 3-a. The groove dimensions are roughly: 4.5 mm deep and 1.5 mm apart. Due to these grooves, the geometrical surface area is increased by a factor of 2.5.

The deuterium recycling behavior of the pre-saturated surfaces with and without machined-grooves is shown in Fig. 3-b. Notice that the surface with machined-grooves yields about 20-25% reduction of the recycling coefficient at the steady state. No saturation behavior is observed. In addition, radiation intensities from CD-band at 4311Å and from C-I at 9095Å measured at the steady state were found to be reduced by about 10% and 15%, respectively. At temperatures around 700°C, similar reduction in recycling of deuterium as well as sputter-induced impurities was also seen, but to a lesser degree. These findings indicate that reemitted deuterium, chemically sputtered hydrocarbon and physically sputtered carbon are continuously co-deposited in the groove structure.

In fact, a yellowish film, indicative of hydrogenated carbon [16], is found on the bottom half surface of the groove partitioning walls and on the groove bottom area. This localized film formation indicates that sputter-emitted carbon from the plasma-front part of the groove partitioning wall dominates the burial process of recycling particles in the bottom part. Since the grooved gap distance is comparable with the gyro-radii of plasma particles (see section 2), most of the ions will strike on the groove partitioning wall mainly in the plasma-front part at a bombarding energy of 300 eV. These energetic deuterium ions will hinder the formation of the co-deposited film. This postulation is consistent with the localized formation of the co-deposited film towards the groove bottom.

Shown in Fig. 4 are the surface morphologies of the groove partitioning wall. A similar but somewhat less porous sponge-like morphology is observed in the plasma-front side (see Fig. 4-a) to that observed for the flat graphite surface after He-PAP (see Fig. 1-a). However, the yellowish co-deposited layer exhibits a completely different surface

morphology with no obvious porosity (Fig. 4-b). Also, notice characteristic dendritic growth of dens-packed microstructures. Unfortunately, no data are available for analysis of incorporated deuterium in the co-deposited film to date.

Here, one must bear in mind that the co-deposition effect will lead to an increase of the on-site tritium inventory in fusion devices. In TFTR, a significant amount of deuterium (of the order of 10^{17}-10^{18} D/cm^2) has been recently found in the gap space between graphite tiles (a few milli meters) and has been attributed to a similar co-deposition effect [17]. However, the deuterium concentration decreases exponentially with increasing distance from the plasma-facing side of the tile. The opposite is true in the present observation. The co-deposited surface in TFTR might serve as a substrate for redepositing particles with relatively low energies and hence one sees only the geometry effect on the reach of the particle in-flow. In contrast, the plasma-front side of the partitioning walls of the groove structure used here acts as the sputter target to supply carbon to bury recycling particles by being bombarded with the deuterium plasma at 300 eV. As a result, the localized co-deposition is observed towards the bottom of grooves.

As demonstrated here, the first attempt to reduce particle recycling by the topographical surface modification has been successful. The partitioning wall of the groove structure is found to act in part as a sputter target to supply carbon to bury recycling particles and to serve in part as a substrate of the co-deposited film under deuterium plasma bombardment. Since obviously the burial probability depends on the in-groove residential time of the particle for a given condition, one might attribute the reduction of deuterium recycling to the plasma effects in such a way that low energy neutrals reside longer than bombarding ions (or neutrals after the first few collisions with the partitioning walls). Further improvement of the groove arrangement is possible. Details will be published separately.

5. CONCULUSIONS AND IMPLICATIONS TO RECYCLING IN FUSION DEVICES

Several possibilities have been investigated, intending to reduce particle recycling from selected graphite materials under high-flux deuterium plasma bombardment. The significance of the plasma effects is pointed out as the crucial issue in understanding the particle recycling behavior.

The increased surface pore openings and refreshed adsorption sites by He-PAP are found to enhance retention of deuterium. This effect leads to a significant reduction of particle recycling and chemical erosion under deuterium plasma bombardment. Also, increasing the surface porosity by appropriate materials selection can reduce the steady state recycling rate due to the in-pore storage mechanism. Due to physical and chemical sputtering, high-fluence hydrogen plasma bombardment will naturally provide the sponge-like surface to low-density, isotropic graphite [18]. Thus, one can expect reduced recycling in fusion devices for the plasma-facing components made of isotropic graphite materials, e.g. POCO-graphite in TFTR, CL5890PL in JET, as the confinement and conditioning plasma discharges are repeated.

Non-saturable deuterium plasma pumping as well as reduced emission of sputter-induced impurity have been observed for a machine-grooved surface. This effect is attributed to continuous co-deposition of particles in the groove space. The co-deposited film is found to have a characteristic surface morphology with dendritic microstructures. The concept of topography-induced co-deposition can be applied to the control of fuel particle recycling as well as impurities in fusion devices, via adjustment to edge-plasma parameters and local magnetic field configuration. Yet the effect of continuous co-deposition on the tritium inventory is crucial. Incorporated tritium might be recovered through thermal decomposition of the co-deposited films. Details are still unclear. Further investigation is necessary to clarify this point.

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Table 1 Summary of recycling coefficients and methane yields at the steady state*.

Material	Recycling coefficient*	CD_4 signal**	Temperature(°C)
POCO-graphite			
Pre-saturated	0.75(0.85***)	0.69	65
Activated	0.72	0.65	60
4D-graphite weave			
Pre-saturated	0.85(1.0***)	0.64	69
Activated	0.8	0.65	65
2D-graphite weave			
Pre-saturated	0.96(1.0***)	0.65	72
Activated	0.94	0.62	65
Pyro-graphite			
Pre-saturated	1.0(1.0***)	0.72	81
Activated	1.0	0.70	87

* The recycling coefficient is taken at t = 10s and normalized to that for Pyro-graphite as a pore-free reference material (see Fig. 1).
** The signal intensity for CD_4 is normalized to that at 600°C (not reported here).
*** The recycling coefficient peak due to ion-impact desorption (see text).

FIGURE CAPTIONS

FIG. 1; Surface morphologies of graphite materials after He-PAP: (a) POCO-graphite; (b) 4D-graphite weave; (c) 2D-graphite weave; and (d) Pyro-graphite. Notice that the surface porosity decreases in order: POCO-graphite, 4D-graphite weave, 2D graphite weave, and Pyro-graphite.

FIG. 2; Typical data from recycling and erosion measurements for plasma-activated and pre-saturated 4D-graphite weave: (a) temperature; (b) recycling coefficient; and (c) methane signal from RGA, each as a function of time. The recycling coefficients shown here are normalized to the steady state value for Pyro-graphite as a pore-free reference material (see Fig. 1).

FIG. 3; Deuterium recycling behavior of the pre-saturated POCO-graphite targets with a flat surface and with the grooved surface: (a) temperature; and (b) recycling coefficient, each as a function of time. The recycling coefficients shown here are normalized to the steady state value observed for the flat surface.

FIG. 4; Surface morphologies of the partitioning wall (a) the plasma-front side with no obvious co-deposited films; and (b) the groove bottom side with the co-deposited film. This particular target was subjected to deuterium plasma bombardment to a fluence of the order of 10^{21} ions/cm^2 at 300 eV.

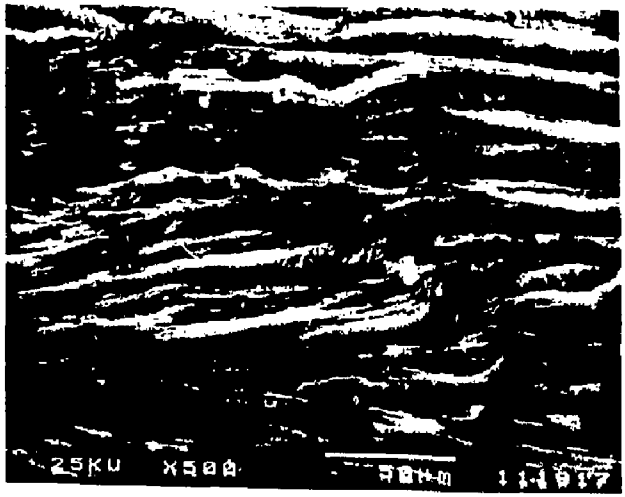
Fig. 1


25KV X500 50μm 111918

(a)


50μm 111912

(b)


25KV X500 50μm 111917

(c)


25KV X500 50μm 111923

(d)

FIG. 2

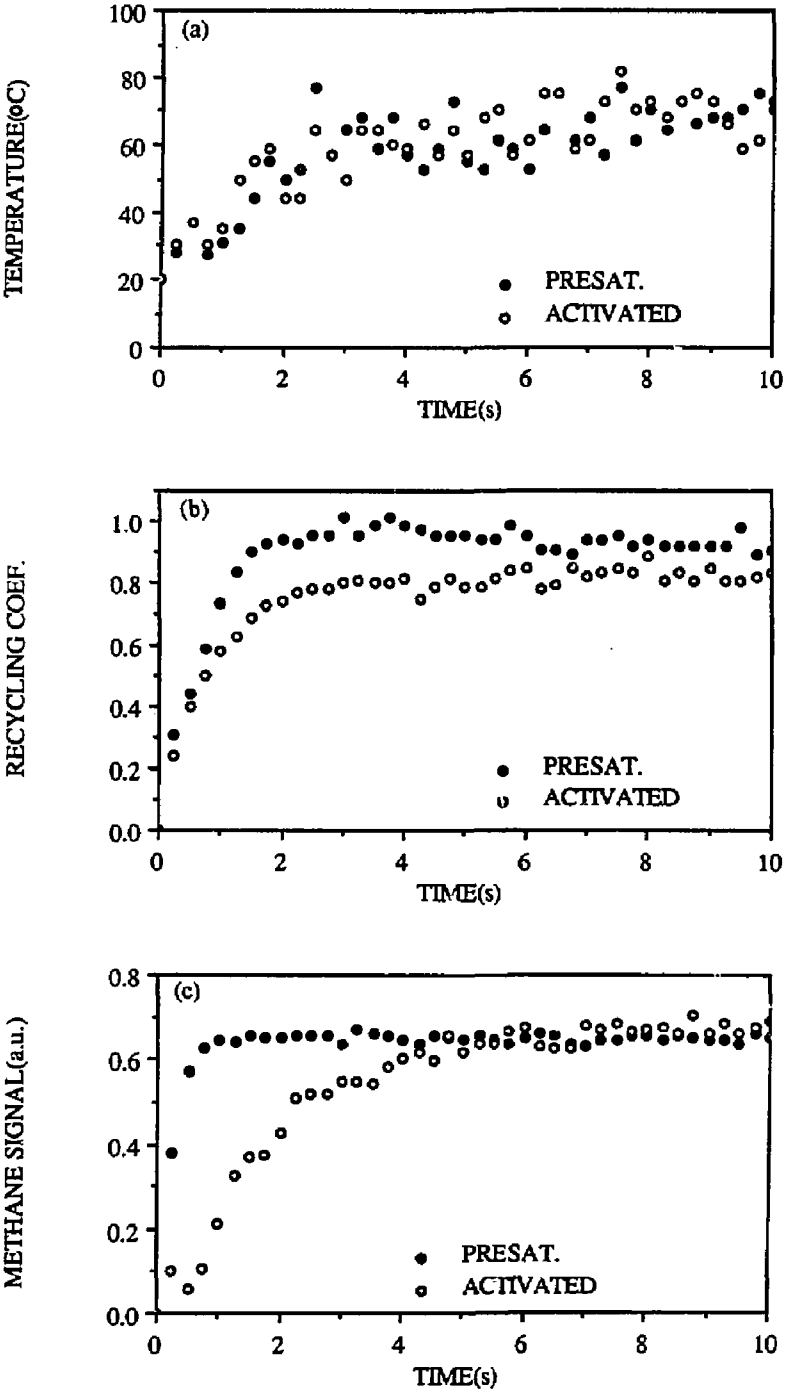

(a)
TEMPERATURE(oC)
100
80
60
40
20
0
0
2
4
6
8
10
TIME(s)
PRESAT.
ACTIVATED
(b)
RECYCLING COEF.
1.0
0.8
0.6
0.4
0.2
0.0
0
2
4
6
8
10
TIME(s)
PRESAT.
ACTIVATED
(c)
METHANE SIGNAL(a.u.)
0.8
0.6
0.4
0.2
0.0
0
2
4
6
8
10
TIME(s)
PRESAT.
ACTIVATED

FIG. 3

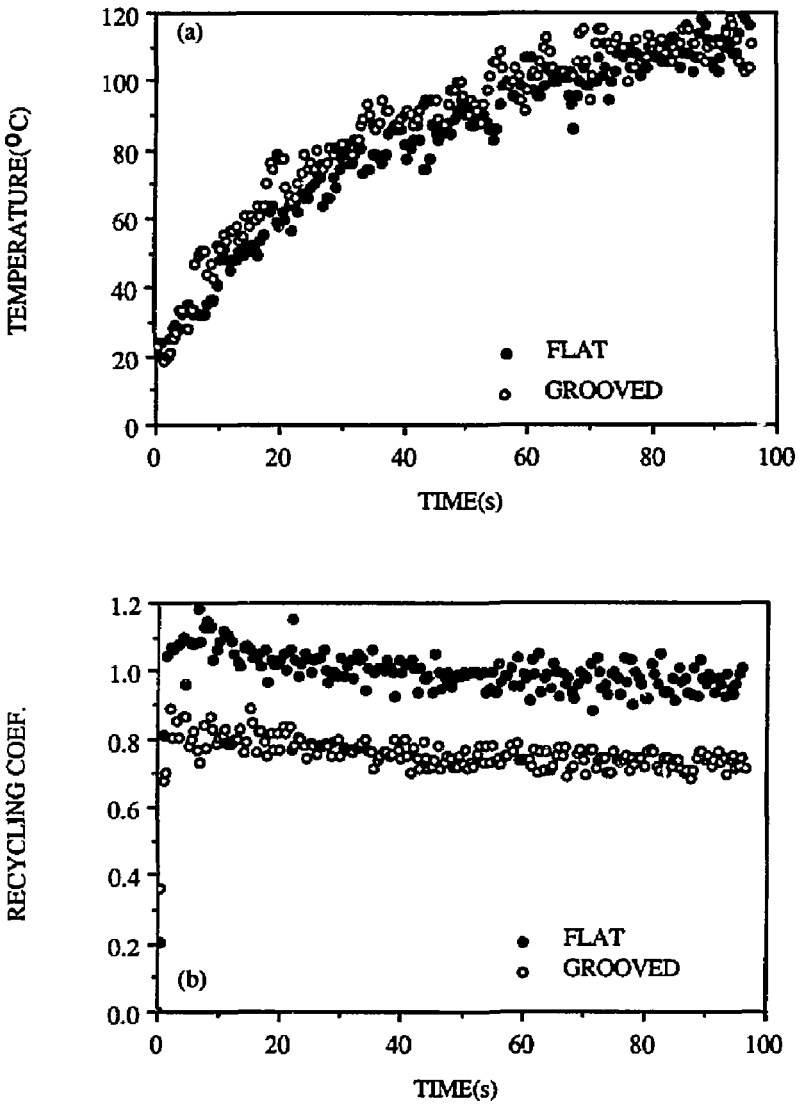
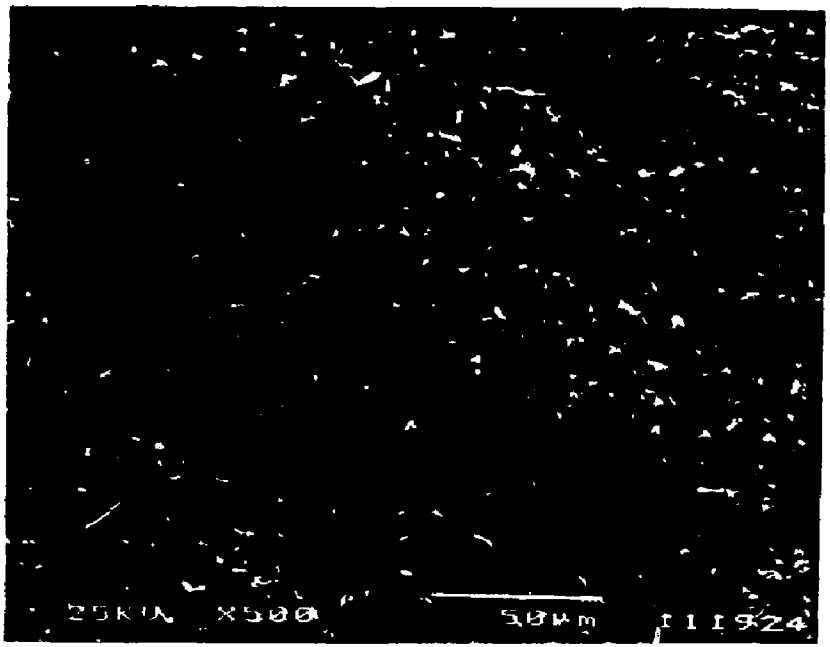

(a)
TEMPERATURE(°C)
120
100
80
60
40
20
0
0
20
40
60
80
100
TIME(s)
FLAT
GROOVED
(b)
RECYCLING COEF.
1.2
1.0
0.8
0.6
0.4
0.2
0.0
0
20
40
60
80
100
TIME(s)
FLAT
GROOVED

Fig. 4

(a)



(b)