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BUDAPEST

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ABSTRACT

Trying to outline the energy dependence of surface deformations such as exfoliation and flaking on candidate CTR first-wall materials, stainless steel and two types of inconels were bombarded by 0.8, 1 and 4 MeV helium ions. All the bombarded spots could be characterized by large exfoliations covering almost the total implanted area. No spontaneous rupture was observed except on one type of inconel where flaking took place right after reaching the critical dose. After mechanical opening of the formations, similar inner morphology was found as in our previous studies on gold [5,6].

АННОТАЦИЯ

Для изучения зависимости от энергии деформаций типа шелушения и флекинга, появившихся на поверхности материалов, предложенных в качестве внутренней стенки термоядерных реакторов, была проведена бомбардировка нержавеющей стали и двух типов инконеля ионами гелия с энергией 0,8, 1 и 4 МэВ. Области поверхности, которые подвергались бомбардировке, характеризуются шелушением, покрывающим почти всю площадь имплантации. За исключением одного образца инконеля, на котором в момент достижения критической дозы произошел флекинг, не наблюдалось спонтанного отрыва слоя от поверхности. При механическом вскрытии слоя можно было наблюдать такую же внутреннюю морфологию, какая наблюдалась раньше на образцах из золота.

KIVONAT

A fuziós reaktorok első falaként javasolt anyagok felületén létrejövő deformációk, ugymint felválás /exfoliation/ és rétegleválás /flaking/ energiafüggésének kideritése céljából rozsdamentes acélt és két fajta inkonelt bombáztunk 0.8, 1 és 4 MeV energiáju hélium ionokkal. Az összes bombázott foltot egy majdnem az egész implantált területet beboritó, nagyméretü felválás jellemezte. Az egyik inkonel minta kivételével, amelyen a kritikus dózis elérésének pillanatában rétegleválás jött létre egyetlen esetben sem figyeltük meg a felválás spontán kiszakadását. Mechanikai felnyitás után az aranyon végzett vizsgálatainkban megfigyelthez hasonló belső morfológiát találtunk [5,6].

1. INTRODUCTION

Surface deformations of the first wall of the future CTR machines, such as blistering, exfoliation and flaking are regarded as a possible main source of plasma contamination. In the low and medium energy region (10-500 keV) these phenomena are extensively studied [1-4] but in the MeV energy region relatively few experiments were performed [5-7].

Therefore a systematic study has been begun to clarify what was going on at high energies. The first experiments were performed on gold which proved to be a good model material in several respects. For example its excellent secondary electron emission property means that much more details could be observed by SEM as compared to the candidate materials. Due to good plasticity it preserves the stages of the evolution of surface deformations caused by high-dose helium implantation. In addition, the good heat conductivity of gold reduces the degree of the beam heating due to increased beam-power at MeV energies. It was concluded that the large exfoliations covering almost the total bombarded spot arised from the lateral coalescence of blisters at the energies used [5,6].

Besides a transition energy was found near 1 MeV below which the surface deformations could be characterized by blistering and above which mainly by exfoliation [6].

This paper offers a qualitative picture of the surface deformations of some candidate materials such as inconels and stainless steel in making an attempt to outline what part of the earlier observations can be generalized and what are specific to gold.

2. EXPERIMENTAL

0.8-4 MeV $^{4}He^{+}$ particles of a 5 MeV Van de Graaff accelerator were used to implant different kind of candidate first wall materials, such as inconels type EP-753 (its nominal composition is 19 Cr, 1.5 Mn, 33 Fe, 41 Ni, 0.5 Nb, 5 Mo) and INCO-625 (21 Cr, 3.5 Fe, 63 Ni, 3.5 Nb, 9 Mo) and stainless steel type OKH16N15M3B (16 Cr, 0.5 Mn, 65 Fe, 15 Ni, 0.5 Nb, 13 Mo). Samples were irradiated after having been cleaned with pure alcohol only. The ions hit the target perpendicularly on a spot size of Ø 2 mm. The bombarding dose was measured by standard secondary electron supression and current integration. The vacuum was kept at $5x10^{-5}$ Pa. A special cold trap system minimized the hydrocarbon deposition onto the target surface.

To determine the critical dose, a binocular system with a magnification of ten was applied to in-situ observation during bombardment. This system was able to detect surface deformation which were larger than 50 μm. The dose rate was kept as low as $4-8x10^{13}$ ions/cm^2s but the estimated temperature rise was between 100-250°C. All these parameters together with the critical doses are shown in Table I.

The exfoliations were investigated in a JEOL-JSN-35 type scanning electron microscope with a lateral resolution of 10 nm. To investigate their inner morphology the exfoliations were opened mechanically by a tungsten pin.

3. RESULTS

The first observation has been that the critical dose values do not follow any tendency as opposed to gold [5,6]. All the bombarded spots could be characterized by large exfoliations which covered almost the total implanted area.

No spontaneous rupture was observed among the present experimental conditions except for EP-753 at 1 MeV where upon reaching the critical dose flaking took place instantly (*Fig. 1*). On the flaked skin several holes could be observed and the missing material was found to be sticked to the bulk of the material (*Fig. 2*). Besides the skin exhibited a two-layer structure as it had been observed on gold [5] (*Fig. 3*). However, in contrast to gold, both the skin and the bottom were relatively smooth and no structure has been observed.

At 4 MeV bombardment the implanted area exfoliated without rupture (*Fig.4*). According to the in-situ observations right after the exfoliation appeared the middle part of the skin became red hot. This might explain that the central part of the skin was broken along grain boundaries (*Fig. 5a*). After mechanical opening a zone structure could be seen on the otherwise smooth bottom (*Fig. 5b,c*) which were presumably connedted with the lateral coalescence of blisters and/or exfoliations of smaller size [5,6].

Inconel type INCO-625 exhibited a different behaviour. At both 0.8 and 1 MeV irradiations only shrinked exfoliated surface has been observed with several holes on it but no rupture took place applying the same dose than that of EP-753 (*Fig. 6*). The surface coverage factor was only 30% while on EP-753 almost the total spot flaked. The different type of surface deformations might be the consequence of the different composition. A smooth bottom without any structure was found after opening both formations.

Table I.

Samples and their implantation circumstances

Material	Energy [MeV]	Dose rate [x10^{13} $\frac{ions}{cm^2 s}$]	Dose [x10^{18} $\frac{ions}{cm^2}$]	Critical dose for exfoliation [x10^{18} $\frac{ions}{cm^2}$]	$T^{a/}$ [^oC]	$T^{b/}$ [^oC]
EP-753	1	5	1.0	0.9	100	300
	4	8	0.9	0.6	250	800
INCO-625	0.8	4	1.2	1.2	100	300
	1	4	1.0	0.9	100	300
	4	7	2.0	1.8	250	500
OKH16N15M3B	0.8	5	1.9	0.9	100	300
	1	5	1.5	1.1	100	300
	4	6	0.9	0.8	200	450

a/ Estimated temperature rise of the bulk during irradiation.

b/ Estimated maximum temperature of the exfoliated skin.

The 4 MeV implantation resulted in a total exfoliation of a dome like shape without any observable hole (*Fig. 7*). The border part of the formation was not uniform, several infolding could be found. This observation emphasizes the role of local mechanical properties of the material in the evolution of such big formations. The additional dome - shaped bulge on the middle part of the exfoliation is due to plastic deformation caused by the high internal pressure of the gas and the local temperature rise together.

Inside, both on the inner side of the skin and on the bottom a rough structure consisting of lamellae of different size could be observed. This might be the reason why we cannot see bottom zones observed on gold (*Fig. 8*).

In the case of stainless steel both the 0.8 and 1 MeV helium implantation produced a fully exfoliated layer again. The skin of exfoliations was shrinked and several holes could be observed on them. The character of these exfoliations was quite similar to INCO 625, only the surface coverage factor increased to 90% because of the higher fluence (*Fig. 9*).

The structure inside of the lower energy formations showed a different morphology from inconels. First of all on the inner side of the skin many secondary ruptured blisters of size 1-4 μm were found (*Fig. 10*). This observation is similar to those on gold. Besides, just like for any other materials the skin consisted of two layers (*Fig. 11*) and has no uniform thickness. Some of the micrographs markedly show that at many spots a part of the skin sticked to the bottom (e.g. *Fig. 12*).

Fig. 13 shows the exfoliation which was obtained after 4 MeV bombardment. According to the micrograph its shape and size are quite a similar to that observed on INCO 625 at this particular energy. On the central part the grains became recognizable (*Fig. 14*), presumably in correlation with the higher skin temperature induced by the beam. The bottom region inside was smooth but on the inner side of the cover at the central region a sponge-like structure has been clearly observed (*Fig. 15a*). Going outward on the skin this structure turns into blisters of 0.5-2 μm size (*Fig. 15b*).

4. SUMMARY AND DISCUSSIONS

The observations can be summarized and interpreted as follows.

The bombardment in all cases results in large exfoliations without spontaneous rupture except inconel type EP-753 where reaching the critical dose flaking took place instantly. So the transition energy at which the blistering process turns into exfoliation [6] is lower than 0.8 MeV in the case of materials investigated.

From the shape of exfoliations and from the existence of rough zones on the bottom indicating earlier stages of the evolution we can conclude that the formation mechanism of exfoliation on these candidate materials is again the lateral coalescence of blisters of smaller size.

The puffed shape of high energy formations clearly indicates the role of gas pressure inside. The escape of helium through holes in every case resulted in a shrinked skin.

The exfoliated skin consists of two clearly distinguishable layers. The reason for this is the following. The implanted helium causes damage and accumulates in a relatively thin depth interval which splits off into two parts during the exfoliation process. Its outer part generally can be found on the inner side of the lid and the inner one on the bottom, both as distinct layers. Obviously in these layers the helium concentration is high at the surface, too.

The helium rich layers on the bottom and on the exfoliated skin try to swell resulting in large integrated lateral stresses. Presumably these stresses, which are regarded as moving forces of the blistering process [8,9] are responsible for the appearance of a lot of secondary formations observed inside exfoliations, such as split lamellae, ruptured and/or unruptured blisters and thin secondary exfoliated layers on the bottom and/or on the inner side of the exfoliated skin.

If the temperature of the exfoliated skin during prolongated bombardment is high enough grains develope on the skin, moreover they can separate from one another along their boundaries. In addition, the inner helium rich layer can transform into a sponge-like structure.

It is difficult to make a comparison between critical doses for exfoliation because of the different surface roughness and temperature rise of the implanted samples, but the INCO-625 seems to be the most resistant materia .

5. CONCLUSIONS

On the base of present experiments we can conclude that our earlier observations on gold can be generalized to a relatively large extent.

The most exciting result is the general appearance of secondary surface deformations of different kind inside the exfoliations. They clearly demonstrate that surface layers containing high amount of helium beginning right from the surface can relatively easily separate from the bulk of the material. Similar layers will be developed on the first-walls of future CTR devices due to the broad distribution of energy and impinging direction projectiles.

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Fig. 1

Flaking on [illegible] type EP-[illegible] after 1 MeV implantation

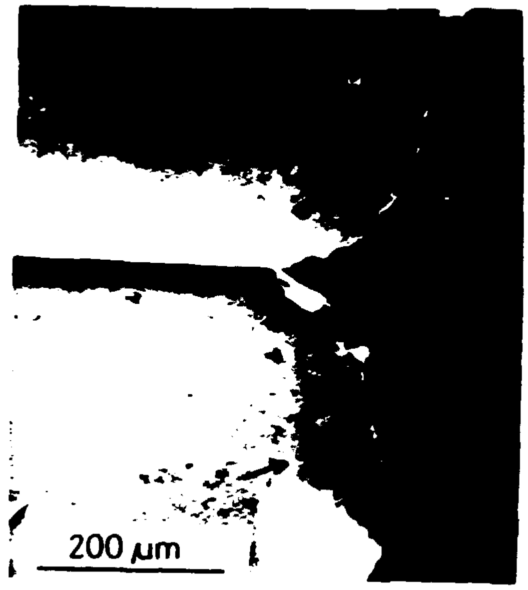

Fig. 2

1 MeV implanted EP-[illegible]. Arrows indicate a hole on the flaked skin and the missing material on the bottom

Fig. 3

1 MeV implanted EP-[illegible]. Side view of the flaked skin with double layer structure

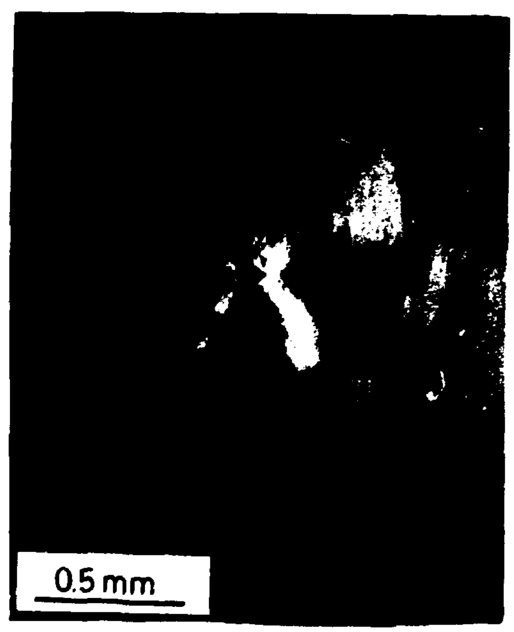

Fig. 4

Exfoliation on 4 MeV implanted EP-756


100 µm


100µm


10 µm

Fig. 6

Exfoliation on inconel type INCO-625
after 1 MeV bombardment

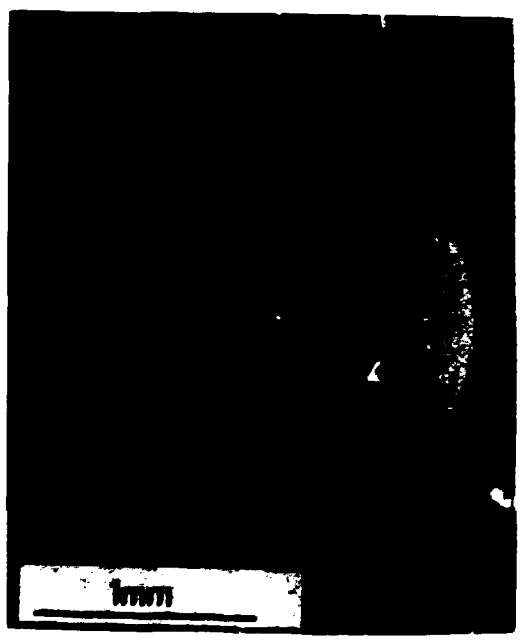

Fig. 7

Exfoliation on 4 MeV bombarded
INCO-625

Fig. 8a

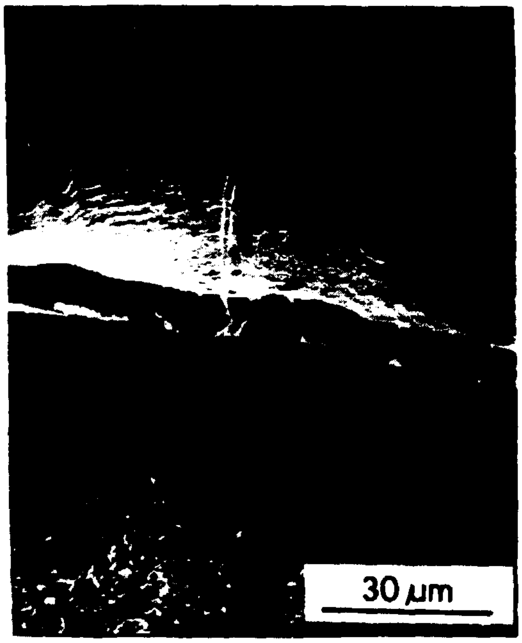

Fig. 8b

Fig. 8c

The inner structure of exfoliation on 4 MeV bombarded INCO-625. An overall picture of mechanically opened formation /a/. Splitted off lamellae on the outer /b/ and central region /c/ of the bottom

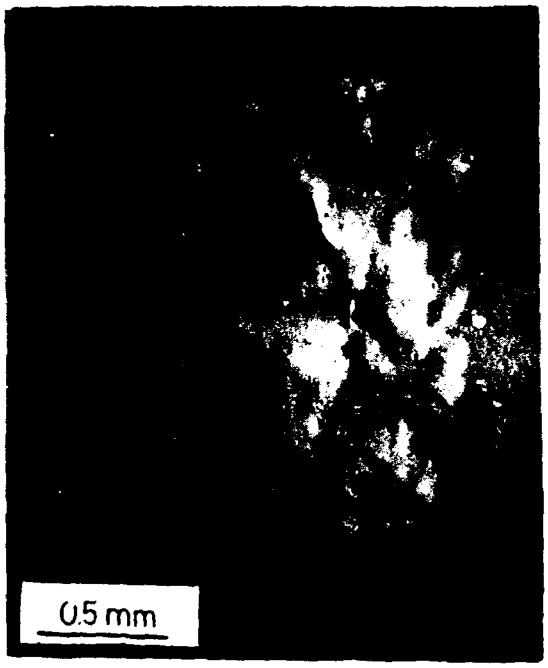

Fig. 9a

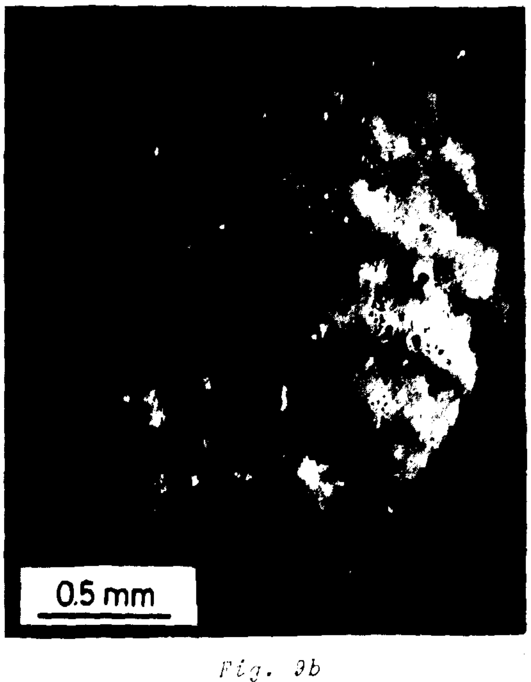

Fig. 9b

Exfoliations on stainless steel type 0KH16N15M3B after 0.5 /a/ and 1 MeV /b/ implantation

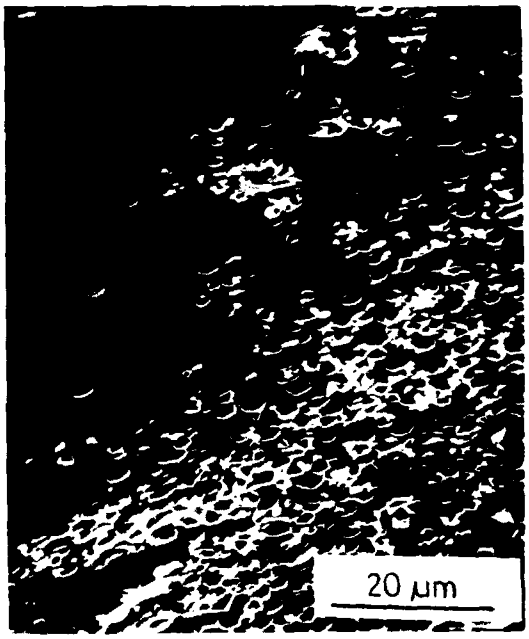

Fig. 10a

Fig. 10b

Fig. 10c

Ruptured secondary blisters on the central part of the inner side of the exfoliated skin on 0KH16N15M3B after 0.[illegible] (a,b[illegible] and 1 MeV (c) implantation

Fig. 11

[illegible] MeV implanted OKH16N15M3B the [illegible] layer structure of the skin

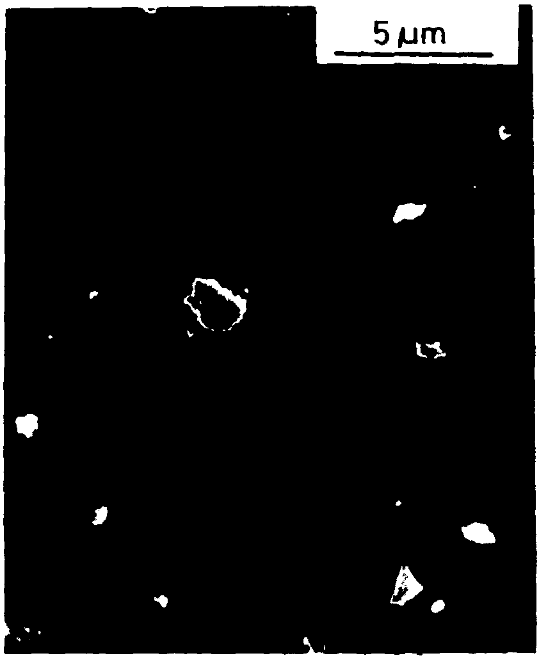

Fig. 12

0.4 MeV implanted OKH16N15M3B. Lamellae from the inner side of the skin sticked to the bottom

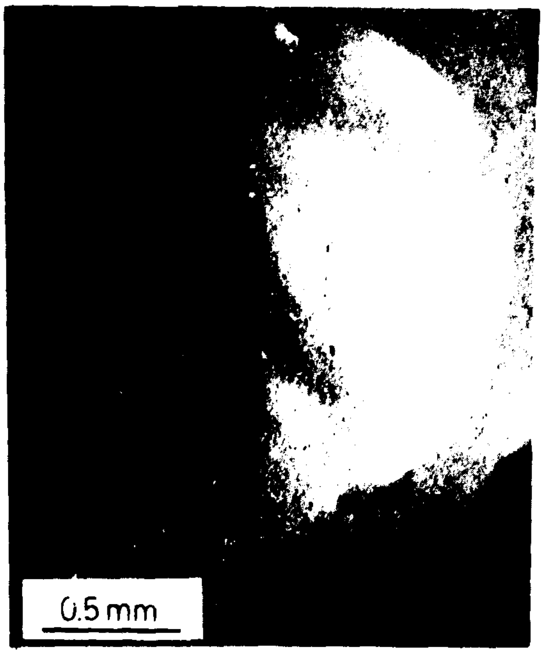

Fig. 13

[illegible] 4 MeV implanted [illegible]

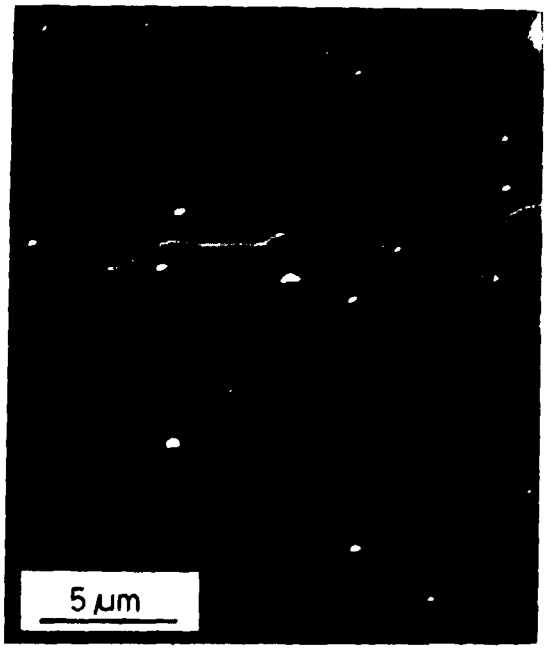

Fig. 14

4 MeV implanted OKH16N15M3B. Recognizable grains on the central part of the [illegible] skin

Fig. 15a

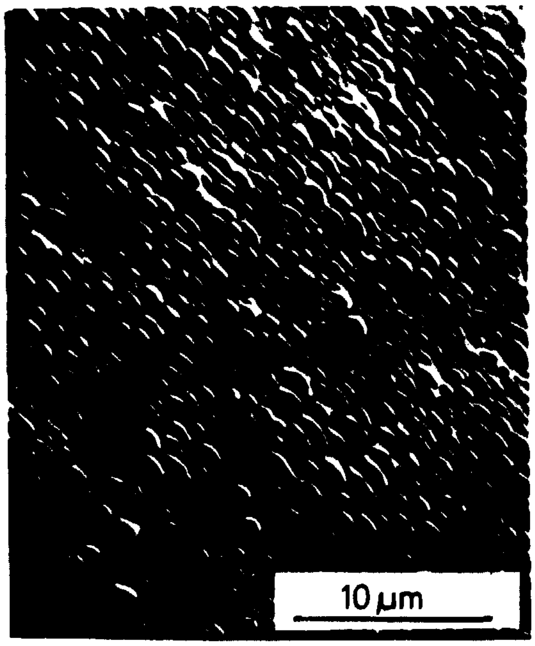

Fig. 15b

4 MeV implanted OKH16N15M3B. Sponge-like structure on the central region of the inner side of exfoliated skin /a/ rounded by a region of secondary blisters /b/

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