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SPUTTERING AND SURFACE DAMAGE OF TFTR PROTECTIVE
PLATE MATERIALS (Mo, TZM, GRAPHITE) BY ENERGETIC
 D^+ ION IRRADIATION

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SUMMARY

Studies have been conducted in accordance with a service request from the Plasma Physics Laboratory (PPPL) of Princeton University to determine the total sputtering yields and the surface damage of molybdenum [a candidate material for the neutral beam injector protective plate for Princeton's Fusion Test Reactor (TFTR)] caused by the impact of D^+ ions at 120-keV, 60-keV and 40-keV at fluxes and total doses agreed upon between PPPL and ANL (see Appendix A, my letter to Dr. Joseph Cecchi, PPPL). The irradiations have been conducted in a specific pulsed mode as well as in a d.c. mode. The irradiations have been conducted over a period of fifteen weekends (48 working hours per weekend, plus 4 hours of set up time on Fridays) during the months of June, July, August and September of 1977. The material TZM (a molybdenum alloy) was included in some tests of surface damage, but not for the full complement of doses planned for molybdenum. According to a request by PPPL the target temperature was not to be controlled (targets were allowed to reach a temperature determined by the beam power deposition and the conductive and radiative heat losses), but the target temperature was to be monitored. The irradiations were conducted at pressures ranging from 4×10^{-9} torr to 2×10^{-8} torr.

A determination of the total sputtering yield S of Mo for energetic deuteron bombardment was made from the total number of deuterons striking the Mo surface, and from the measurement of the thickness of the sputtered Mo deposit on monocrystalline Si (111) substrate by the Rutherford backscattering technique in an ultrahigh vacuum environment. Since the yield values were expected to be very small ($\sim 10^{-3}$ atoms/ion), very careful calibrations of the Rutherford backscattering technique were required together with a careful handling of targets and substrates. For polycrystalline Mo bombarded at normal incidence by deuterons with energies of 40-keV, 60-keV and 120-keV, we determined yield values of $(1.10 \pm 0.11) \times 10^{-3}$ atoms/ion, $(0.75 \pm 0.07) \times 10^{-3}$ atoms/ion, and $(0.4 \pm 0.05) \times 10^{-3}$ atoms/ion, respectively. These values agree within 20% with values we have calculated using a modified Sigmund theory and by fitting the calculated value for ~ 2 keV D^+ -Mo to the corresponding experimental value published recently by Bay et al., JAP (1977). For an estimate of the number of Mo atoms released per cm^2 and per deuterium pulse (with 1 sec pulse length) one needs to multiply the above mentioned yield values with the deuterium fluxes typical for the three energies mentioned above. If one uses the flux values estimated by Dr. J. Cecchi (for a list, see Appendix A) one obtains for all three deuterium components together a release rate of 8.9×10^{12} atoms $\text{cm}^{-2} \text{sec}^{-1}$. If $\sim 5.5\%$ of the first wall area (e.g., Mo armor plate area struck by beam $\sim 6.0 \times 10^4 \text{ cm}^2$) is being bombarded, approximately 5.3×10^{17} atoms per armor plate area struck by the beam are being released per 1 sec pulse. If these impurity atoms would have a chance to diffuse uniformly through the plasma in TFTR, an average Mo impurity concentration n_i of $\sim 1.5 \times 10^{10}$ atoms cm^{-3} would be built up during a 1 sec pulse. Then, for a plasma density $n_p = 1 \times 10^{14} \text{ cm}^{-3}$, an impurity fraction $f = n_i/n_p = 1.8 \times 10^{-4}$ would result. Such an f -value for Mo impurities would cause, for example, an increase in the bremsstrahlung losses of a hydrogenic plasma of $\sim 32\%$ for a fully stripped Mo atom. Of course, other radiation loss mechanisms such as line radiation will have to be considered too [e.g., see R. V. Jensen, et al., Nucl. Fusion, 17, 1187 (1977)]. An estimate of the surface erosion of the Mo protective plates caused by sputtering, reveals that the thickness loss one can expect for the atom release

rate of 8.9×10^{12} atoms $\text{cm}^{-2} \text{sec}^{-1}$ and for the long period of 10,000 deuterium pulses is negligibly small (~ 150 Å).

The analysis of the irradiated samples by scanning electron microscopy reveals surface damage due to blistering in the Mo samples irradiated with 40-keV and 60-keV D^+ ions for dose values ranging from $\sim 0.14 \text{ C/cm}^2$ to $\sim 1.3 \text{ C/cm}^2$ [corresponding to a total number of pulses ranging from 500 pulses (1 sec pulse length) to 2500 pulses, or in d.c. runs, to the doses mentioned] at target temperatures ranging from 23°C to 100°C for the 40-keV run, and from 23°C to 120°C for the 60-keV run. For the 120-keV D^+ ion irradiations, however, very little surface damage due to blistering could be observed for either the pulsed mode or the d.c. mode operation for dose values which were much higher than those used for the 40-keV and 60-keV D^+ ion irradiations (the dose values ranged from 0.73 C/cm^2 to 3.70 C/cm^2 for a total number of pulses ranging from 500 pulses to 2500 pulses, or for the equivalent length of d.c. runs). This surprising result was thought to be due to higher target temperatures reached in the 120-keV runs, temperatures which would enhance the permeation of deuterium through Mo and prevent the build-up of large bubbles [temperatures ranged from $\sim 280^\circ\text{C}$ in the pulsed operation to 430°C in the d.c. operation]. In order to test if higher target temperatures would also help to reduce or eliminate the surface damage of Mo by blistering for the lower D^+ ion energies of 40-keV and 60-keV, irradiations at target temperatures of 300°C were conducted. These irradiations were conducted for both the pulsed mode (2500 pulses - dose $\sim 1.2 \text{ C/cm}^2$ for 60-keV D^+ , and 0.7 C/cm^2 for 40-keV D^+) and the d.c. mode operation (1.2 C/cm^2 for 60-keV D^+ , and 0.7 C/cm^2 for 40-keV D^+). Scanning electron microscope analysis of the irradiated samples revealed no detectable surface damage at the highest magnification, 50K (resolution ~ 100 Å).

This is a very encouraging result since it suggests that if it should be possible in TFTR operations to maintain a neutral beam injector protective plate, made out of molybdenum, at 300°C , then it should be possible to eliminate surface damage by blistering even at the high fluxes used ($1.7 \times 10^{15} \text{ D}^+ / (\text{cm}^2 \text{ sec})$ for 40-keV, $2.9 \times 10^{15} \text{ D}^+ / (\text{cm}^2 \text{ sec})$ for 60-keV, and $\sim 9.3 \times 10^{15} \text{ D}^+ / (\text{cm}^2 \text{ sec})$ for 120-keV and for the highest total doses reached in these experiments. [It should be noted that the back target temperatures quoted here are average target temperatures (as measured with a thermocouple on the back-side of a 0.005" thick Mo-sample, mounted on a 3/4" thick Cu-block and are not actual surface temperatures)]. Surface temperatures higher than 280°C were measured with an infrared pyrometer. Furthermore, for identical irradiation conditions, TZM alloy showed less blistering than Mo for both pulsed and continuous irradiation with 40- and 60-keV D^+ . A special request was made by PPPL during the course of these experiments to look for the effect of the D^+ ion impact on ATJ Graphite and the Reactor Grade Graphite (UT6FT). The results of these studies are included in this report.

GOAL OF STUDIES

The design of Princeton University's Tokamak Fusion Test Reactor (TFTR) calls for additional plasma heating by neutral beam injection. Since the injected neutral beam (e.g. deuterium) is only partially trapped in the plasma, a protective plate is required to collect this transmitted beam.

We were asked by Dr. Joseph L. Cecchi to determine, by experimental studies, if polycrystalline molybdenum (a potential protective plate material) would experience detectable surface damage and erosion under the impact of intense D^+ ions in both a pulsed mode and d.c. mode operation at 120-keV, 60-keV or 40-keV at fluxes and total doses specified by Dr. Cecchi. In addition, we were asked to determine the sputtering yields of polycrystalline Mo under D^+ ion bombardment for these three energies. The fluxes were estimated under the assumption that 10% of the 120-keV neutral beam component would not be absorbed by the plasma. The appropriate fluxes for the 60-keV and 40-keV components were determined from their relative intensities upon exiting the beam lines and suitable scaling of effective ionization cross sections with beam energy (see Appendix A, item 1a). It was also agreed to include some test runs of TZM as a potential protective plate material, but not for the full complement of doses planned for molybdenum. The surface damage studies were to be conducted over the period of twelve weekends during the months of June, July and August 1977. An Interim Report of some of the pertinent results of these studies was issued during the fall of 1977. During the course of these experiments, Dr. Cecchi expressed the desire to include into the program additional materials such as ATJ Graphite and Nuclear Grade Graphite (UT6FT). While some irradiations of these materials have been included in the twelve weekend runs, the time was too short to analyze the irradiated samples to an extent that meaningful results could be included in the Interim Report. However, the results available are included now in this Final Report.

Three weekends during the month of September were used for D^+ ion irradiations of Mo needed for sputtering yield determinations. Some repeat irradiation runs were conducted during December 1977 and January 1978. The evaluation of the sputtering yield data was completed in February 1978, and this Final Report is scheduled to be issued in March 1978.

EXPERIMENTAL PROGRAM PLAN

A. Surface Damage and Erosion

(a) The first set of irradiation runs was used to determine if, for the maximum number of pulses (2500) specified by PPPL, and for the flux levels agreed upon $[(1.7 \pm 0.2) \times 10^{15} D^+ \text{ ions}/(\text{cm}^2 \text{ sec})$ at 40-keV, $(3.0 \pm 0.5) \times 10^{15} D^+ \text{ ions}/(\text{cm}^2 \text{ sec})$ at 60-keV, and $(9.0 \pm 0.5) \times 10^{15} D^+ \text{ ions}/(\text{cm}^2 \text{ sec})$ at 120-keV] surface damage due to blistering could be observed on polycrystalline molybdenum surfaces. It should be noted that the flux levels reached here are significantly higher than those guaranteed by ANL (see Appendix A). Since the pulse-width was 1 sec and the repetition rate 1 pulse per minute, a single irradiation run of 2500 pulses took approximately 41.7 hours, or nearly one entire weekend.

(b) The second set of irradiation runs was used to determine if surface damage due to blistering could be observed on polycrystalline molybdenum under d.c. mode irradiations with D^+ ions at 40-keV, 60-keV and 120-keV at the flux levels mentioned above and at total doses which were nearly identical to those obtained for the three D^+ ion energies during the 2500 pulse mode irradiation. A comparison between the surface damage caused by the pulsed mode and the d.c. mode operation was to be made.

(c) Irradiation runs were conducted for Mo for each of the three D^+ ion energies for the following number of pulses: 100, 500, 1000 and 2500 pulses at the fixed flux levels mentioned above. This allowed a determination of the dependence of the degree of surface damage (e.g., blister density) on the dose.

(d) Irradiation runs were conducted for Mo for each of the three D^+ ion energies in a d.c. mode operation for dose values which correspond for each ion energy to the dose values reached for the range of total pulses obtained in the experiments mentioned under (c), i.e., 100, 500, 1000, 2500 pulses.

In anticipation of low sputtering yields ($\sim 10^{-3}$ atoms/ion), and in order to facilitate the determination of sputter deposit thickness by Rutherford backscattering, it was decided to irradiate polycrystalline Mo surfaces for each of the three D^+ ion energies in a d.c. mode operation to relatively high doses in the range from 27.7-62.3 C/cm². The D^+ ion fluxes ranged from 2 to 4 x 10¹⁵ ions/(cm² sec).

EXPERIMENTAL ARRANGEMENT AND PROCEDURE

A mass analyzed beam of D^+ ions was produced by an accelerator system consisting of a duoplasmatron source, a beam profiler, an electrostatic beam steerer, an analyzing magnet and a 150-KV d.c. supply. Beam shutters allowed measurements of the "total" ion beam (unbiased) in front of the analyzing magnet, and of the analyzed beam (unbiased) following the analyzing magnet. As shown in Figure 1, the analyzed D^+ ion beam passed through a quadrupole doublet for focussing and through two liquid nitrogen beam line traps to reduce carbon contamination on the irradiated targets. The focussed and analyzed beam then passed through a set of collimating apertures before striking either the target or a pneumatically operated shutter (latter was used for (a) beam tuning, and (b) for beam pulsing). The collimating aperture closest to the target was biased negatively with respect to the target to suppress secondary electrons from the target in order to obtain a more correct ion beam current signal.

For measurements of the total sputtering yield S a monocrystalline Si (111) collector was placed at a distance of 4.75 mm from the surface of the target which was bombarded by the incident beam. The collector had a 3 mm hole to pass the beam through. A tantalum aperture with a 2 mm hole was placed in front of the collector side facing the incident beam. Another aperture was placed between the collector and the target which was biased (-100 V) with respect to the target. This aperture had a sufficiently large slot to allow the sputtered material to pass through and to be deposited on the collector.

As shown in more detail in Figure 2, the target chamber consisted of a stainless steel cross with metal seal ports for the mounting of the stepping motor-target micromanipulator arrangement, the viewing port, the pneumatically operated shutter, an ion-sublimation pump combination, and a front flange

with beam entrance port plus smaller viewing windows (the window was used for the infrared pyrometer). During a typical run the pressure in the target chamber was $4-6 \times 10^{-9}$ torr, and only in two runs was the pressure as high as 1.8×10^{-8} torr (note, ANL guaranteed a pressure of 2×10^{-8} torr, see Appendix A). The beam line of the accelerator section was pumped with three oil diffusion pumps which were baffled and provided with liquid nitrogen traps. The pressure in the beam line in front of the target chamber was $2-3 \times 10^{-8}$ torr.

The details of the stepping motor-target micromanipulator arrangement are shown in Figures 3 and 4. The stepping motor allowed the precise rotation of the target assembly block shown in Figure 5 in two hundred steps (1.8° per step). The $\sim 3/4$ " diameter Cu-block had ten faces to allow the mounting of target foils with dimensions of approximately 1.2 cm x 2.0 cm and thicknesses varying from 0.005" to 0.030". With the aid of the x,y micrometer drive and the z-axis drive, different target areas could be exposed to the beam.

In order to be able to irradiate more than one target during the period of the pulse repetition time of 60 sec (the pulse length being only 1 sec) a microprocessor was used to activate automatically the stepping motor every six seconds to rotate a new target (one of the 10 targets mounted on the target block) into the beam position. A Pro-log microprocessor system was used containing an Intel 8080 CPU (central processing unit), 4K of RAM (random access memory) for storage of data. The processor was interfaced to the stepping motor, the pneumatically operated shutter, and the current integrator scaler via an 8255 programmable interface chip. Input and output were made through a Texas Instruments silent 700 terminal. The microprocessor system would store the number of counts (the charge) each target obtained during the period of one pulse. Every 15 (or 30 minutes) a read-out was provided, listing each target which had been irradiated, the total number of pulses desired, the actual number of pulses accumulated, the total number of counts (the charge) collected for the number of pulses accumulated, and the number of counts for the last pulse each target had received prior to the particular print-out (a recorded check of D^+ ion beam stability). Also, if the flux fell below a minimum value the microprocessor would stop the irradiation until the current was back up again. The design and construction of this advanced target and data handling system was started in July and completed in August. It has helped greatly to increase the output of irradiated targets under controlled conditions.

For one set of irradiation runs the target temperatures were not controlled, in accordance with PPPL's request, but monitored. For another set of irradiation runs the target temperature was held at 300°C . Thermocouples were clamped between the foil and the Cu-block and an "average" target-block temperature was measured. In some irradiation runs thermocouples were spot welded to the target surface edge near target center. The temperature readings varied by $60^\circ\text{C} - 80^\circ\text{C}$ for these two methods of T.C. mounting for otherwise identical irradiation conditions, an effect which can be understood by the difference in heat transfer for the two cases. Surface temperatures could only be monitored for temperatures $>280^\circ\text{C}$ with an infrared pyrometer. Under normal operating conditions the target assembly would be placed into the vacuum chamber on a Friday evening and be pumped down for the beginning

of the run on Saturday morning. The ion source tuning would begin on Saturday morning at 8:00 AM (the Friday evening was used to place a new filament into the source and to replace the W aperture plug with a 0.006" opening). A sufficiently intense analyzed beam was normally obtained by 10:30 AM and a focussed and collimated beam on target by 11:30 AM. After completion of a run the targets were removed from the chamber, demounted from the holder, and stored in plexiglass containers before the analysis with the scanning electron microscope was started.

A description of the target preparation and characterization procedures is given below. The molybdenum targets were obtained from Materials Research Corporation, Orangeburg, New York, as 1" wide and 0.005" thick foils. The TZM foils of the same dimensions as the Mo foils were obtained from Amax Specialty Metals Corporation, Cleveland, Ohio. The chemical composition of the foils as received from the manufacturer are given in Table 1. Both Mo and TZM foils were received in the stress relieved condition and no further annealing was done. The targets (1" x 1/2") were first given a fine metallographic polish and then electroplated in a bath containing 87.5% methanol and 12.5% sulphuric acid at a temperature of 5°C.

Table 1: Chemical Composition of the Materials Used

Material	Major Elements Present (ppm)						
	Ti	Zr	O	C	Si	Fe	Ni
Mo	<10	<10	25	20	<5	60	80
TZM	5000	1040	9	200	10	23	10
ATJ Graphite	Total impurity content 100 ppm, density - 1.9 g/cm ³						
Reactor Grade Graphite UT6FT	Total impurity content <10 ppm, density - 1.8 g/cm ³						

The electropolished targets were characterized before and after irradiation using scanning electron microscopy and scanning Auger spectroscopy. The grain size was determined from the scanning electron micrographs of the electrolytically etched surfaces using Hilliard's method.¹ The average grain size for both Mo and TZM was measured to be $\sim 4 \mu\text{m}$, which is finer than ATSM grain size No. 12. Auger spectra were taken from some typical electropolished Mo targets to determine the impurities present on the surface and Figure 5a shows a typical Auger spectrum. The major surface impurities are O, C and N (Figure 5a). After sputtering the surface with 2-keV Ar^+ ions, the C and O peaks are greatly reduced (Figure 5b) and the C and O impurities are roughly estimated to be ~ 20 monolayers.

Two types of graphite samples were obtained from Dr. Ulrickson of PPPL for this initial study. One was extrusion molded ATJ graphite manufactured by Union Carbide and had a total impurity content of 100 ppm (Table 1); the other was high purity reactor grade graphite (UT6FT), obtained from Ultra Carbon, Bay City, Michigan, having an impurity content of < 10 ppm. The latter graphite was baked at 2400° to drive off the impurities. Both graphite targets were given only a fine metallographic polish.

The polished targets were irradiated in the facility described above. Following the irradiation, the targets were examined in a Cambridge Stereoscan S4-10 scanning electron microscope equipped with an energy dispersive x-ray spectrometer. The size distribution of the blisters observed in the irradiated samples were measured from the micrographs using a Zeiss particle size analyzer.

For a determination of the total sputtering yield S it is necessary to determine the total mass M which has been emitted from the irradiated target spot. Since the sputtered Mo is emitted with an angular distribution which appears to follow to a good approximation a cosine distribution, the deposit thickness will not be uniform over the deposit area but will follow to a good approximation a cosine distribution, too. For a target spot of radius r , a distance l between the center of the deposit and the center of the target spot, a distance R measured from the center of the deposit along a radial direction of the deposit spot, the deposited material density ρ , and the deposit thickness t , the emitted Mass M is given by

$$M = 2 \pi \rho r^2 \frac{t}{\sqrt{1 - \frac{1 + \left(\frac{R}{l}\right)^2 - \left(\frac{r}{l}\right)^2}{1 - \left(\frac{R}{l}\right)^2 + \left(\frac{r}{l}\right)^2 + 4 \left(\frac{R}{l}\right)^2}}} \quad (1)$$

For a determination of (a) a center line of the deposit spot, and (b) of the function $t(R)$, the Rutherford backscattering technique was used with a 750-keV $^4\text{He}^+$ beam, a beam diameter of $0.3 \mu\text{m}$ (note, $\sim 99\%$ of the Mo deposit was in a circular spot with a diameter of $\sim 20 \mu\text{m}$), and a scattering angle (lab. syst.) of 154° .

1. *Metals Handbook*, vol. 8 (Amer. Soc. for Metals, Metals Park, O. p.42, 1973)

Figure 6 illustrates a Rutherford backscattering spectrum of 4.5 monolayer (ML) thick molybdenum deposit on a Si(III) substrate obtained by the scattering of 750-keV $^4\text{He}^+$ ions under an angle of 154° . One can readily distinguish the Mo peak from the broad Si spectrum. The area A under the Mo peak is a measure of the deposit thickness t. A quantitative correlation between A and t (in units of ML) was obtained by calibration standards. These standards had been prepared by vapor depositing known amounts of Mo on a Si(III) substrate. The thickness of such deposits was measured independently by the Rutherford backscattering technique and by depth profiling with Auger spectrometry. The Rutherford backscattering technique allowed measurements of a Mo deposit thickness of only 0.001 ML.

Figure 7 shows the function t (R) vs R as calculated from equation (1), and the experimentally determined values of t (R) for Mo sputtered by 60-keV D^+ ions have been included (see points marked †). The agreement between calculated and experimental values appears to be good.

RESULTS AND DISCUSSION

A. Surface Damage

a. Molybdenum

Figures 8(a)-8(d) show scanning electron micrographs of polycrystalline molybdenum surfaces irradiated with 40-keV D^+ for 100, 500, 1000 and 2500 pulses as indicated in Figure 8. The total dose accumulated for 100, 500, 1000 and 2500 pulses are 1.7×10^{17} , 8.7×10^{18} and 4.3×10^{18} ions/cm², respectively. No blisters could be detected for irradiation to a dose of 100 pulses (Figure 8a). For irradiation to a dose of 500 pulses, blisters with diameters ranging from 1 to 10 μm are observed, and the average blister diameter is 2.8 μm . The value for the average blister density is found to be approximately 3.1×10^4 blisters/cm². For the higher dose of 1000 pulses, the blister diameters range from 1.4 to ~ 20 μm and the average blister diameter is 4.6 μm . The blister density for this 1000 pulse-irradiation is 5.7×10^4 blisters/cm². On further increasing the dose to 2500 pulses the blister diameters range from 2.5 to 20 μm , the average blister diameter is 4.6 μm and the blister density increases to 7.1×10^4 blisters/cm².

The scanning electron micrographs in Figures 8e, 8f, 8g and 8h show polycrystalline molybdenum surfaces after irradiation with 60-keV D^+ for 100 (dose: 3.2×10^{17} ions/cm²), 500 (1.6×10^{18} ions/cm²), 1000 (dose: 3.2×10^{18} ions/cm²) and 2500 (dose: 8.1×10^{18} ions/cm²) pulses, respectively. Again, no blisters could be detected for irradiation for 100 pulses (Fig. 8e). The average blister diameters for irradiation for 500, 1000, and 2500 pulses are 3.5†, 5.2†, and 6.6 μm , respectively. The values for the blister density are 3.5×10^4 †, 3.9×10^4 † and 7.3×10^4 blisters/cm² for 500, 1000 and 2500 pulses, respectively. Thus, for both 40-keV and 60-keV D^+ irradiation, both the blister density and average blister diameter increase with the increasing number of pulses. The above results also show that for equivalent numbers of pulses the average blister diameter is higher for the 60-keV D^+ irradiation than the 40-keV D^+ irradiation, but the blister density does not appear to change much with projectile energy.

† These numbers are based on only one set of irradiations.

Figure 8i, 8j, 8k and 8l show micrographs of molybdenum surfaces irradiated with 120-keV D^+ ions for 100 (dose: 8.7×10^{17} ions/cm²), 500 (dose: 4.3×10^{18} ions/cm²), 1000 (dose: 8.7×10^{18} ions/cm²) and 2500 (dose: 2.2×10^{19} ions/cm²) pulses, respectively. No blisters could be detected for irradiation for 100 pulses (Figure 8i), and even for irradiation for 500, 1000 and 2500 pulses, only a few blisters with diameters ranging from 3 to 5.5 μ m could be seen over the entire bombarded area. (Figures 8j-8l).

The irradiation of polycrystalline molybdenum with 40-, 60- and 120-keV D^+ ions in the continuous mode for total doses of 1.7×10^{17} , 3.2×10^{17} and 8.7×10^{17} ions/cm², respectively, showed no blisters on the surface, a result similar to the pulsed irradiation. Figures 9a, 9b and 9c show polycrystalline molybdenum surfaces irradiated in a continuous mode with 40-keV D^+ for total doses of 8.7×10^{17} (equivalent of 500 pulses), 1.7×10^{18} (equivalent of 1000 pulses) and 4.3×10^{18} (equivalent of 2500 pulses) ions/cm², respectively. The average blister diameters for irradiation for doses equivalent to 500, 1000 and 2500 pulses are 1.6, 1.8 and 1.8 μ m, respectively. The blister densities for these three doses equivalent to 500, 1000 and 2500 pulses, are 1.6×10^5 , 2.1×10^5 and 2.8×10^5 blisters/cm², respectively. Figures 9d, 9e and 9f show scanning electron micrographs of polycrystalline molybdenum surfaces irradiated in continuous mode with 60-keV D^+ ions for total doses of 1.6×10^{18} (equivalent of 500 pulses), 3.2×10^{18} (equivalent of 1000 pulses) and 8.1×10^{18} (equivalent of 2500 pulses) ions/cm², respectively. The average blister diameters for the doses equivalent to 500, 1000 and 2500 pulses are 1.7, 1.6 and 1.1 μ m, respectively, and do not appear to be much different from the average diameters corresponding to 40-keV D^+ irradiations. The blister densities for the doses equivalent to 500, 100 and 2500 pulses are 1.6×10^5 , 1.8×10^5 and 1.7×10^5 blisters/cm², respectively. For irradiation with 120-keV D^+ ions, no blisters could be detected for total doses of 4.3×10^{18} (equivalent of 500 pulses, see Figure 9g), 8.7×10^{18} (equivalent of 1000 pulses, see Figure 9h) and 2.2×10^{19} (equivalent of 2500 pulses, see Figure 9i) ions/cm².

The results for pulsed and continuous irradiations are compared in Figures 10a-g for 40-, 60- and 120-keV D^+ irradiation for a dose of 2500 pulses or its equivalent. It can be seen that for all the three energies there is a reduction in the average blister diameter for the continuous irradiation (Figures 10b, 10e and 10g) as compared to the pulsed irradiation (Figures 10a, 10d and 10f). For the sake of comparison an unirradiated area is shown in Figure 10c. The distributions of blister diameters for the pulsed and continuous irradiation are compared in Figure 11, in which the ordinate is the percentage of blisters in a class of blisters whose diameters fall within the range included in the width of each vertical bar. In the case of 40-keV D^+ pulsed irradiation, the blister diameters range up to $\sim 20 \mu$ m (Figure 11a), whereas for the continuous irradiation, for the same total dose, the maximum blister diameter is less than 5 μ m (Figure 11c). Similar results are observed for 60-keV D^+ irradiation (Figures 11b and 11d).

Table 2 summarizes the quantitative data on average blister diameter and blister density for both pulsed and continuous irradiations, together with

† These numbers are based on only one set of irradiations. The repeat runs have yet to be done.

Table 2: Summary of Average Blister Diameter and Blister Density for Mo Irradiated With D^+ Under Various Conditions.

Energy (keV)	No. of Pulses or its Equivalent	Total Dose Ions/cm ²	PULSED			CONTINUOUS		
			Average Blister Dia. (μm)	Blister Density (no./cm ²)	Average Target Temp. (°C)	Average Blister Dia. (μm)	Blister Density (no./cm ²)	Average Target Temp. (°C)
40	500	8.7×10^{17}	2.8	3.1×10^4	Bulk Temp-38,	1.6	1.6×10^5	Surface Temp. ~260
	1000	1.7×10^{18}	4.6	5.7×10^4	Surface Temp. ~100	1.8	2.1×10^5	
	2500	4.3×10^{18}	4.6	7.1×10^4		1.8	2.8×10^5	
60	500	1.6×10^{18}	3.5	3.5×10^4	Bulk Temp-~44,	1.7	1.6×10^5	Surface Temp. 260-295
	1000	3.2×10^{18}	5.2	3.9×10^4	Surface Temp. ~120	1.6	1.8×10^5	
	2500	8.1×10^{18}	6.6	7.3×10^4		2.1	1.7×10^5	
120	500	4.3×10^{18}	Only Few Blisters of 3-5.5μm in diameter in the entire bombarded area.		Bulk Temp ~70, Surface Temp ~280	No Blisters Detected		Surface Temp. 360-400
	1000	8.7×10^{18}						
	2500	2.2×10^{19}						

the target temperatures measured during the irradiation. It can be seen that for an equivalent number of pulses, the average blister diameter is higher for 60-keV D^+ than for 40-keV D^+ irradiation for pulsed mode of irradiation, but for the continuous mode of irradiation the blister diameters do not change significantly. However, for 120-keV D^+ irradiation, the blistering is greatly reduced as compared to 40- and 60-keV D^+ irradiation even though the total doses for an equivalent number of pulses were higher for the former case than for the latter two cases (see Table 2). This reduction in blistering for 120-keV D^+ irradiation as compared to 40- and 60-keV D^+ irradiation can be partly attributed to the higher target temperature during the 120-keV D^+ irradiation. Since the target temperatures were not controlled (as requested by PPPL) during these irradiations, higher power deposition for the 120-keV D^+ irradiation caused the surface temperature to rise to $\sim 280^\circ\text{C}$ (and the bulk temperature, measured behind the irradiated surface, to $\sim 70^\circ\text{C}$) for the pulsed mode and to $360\text{--}400^\circ\text{C}$ for the continuous mode of irradiation. Similarly, the reduction in blistering in the continuous irradiation with 40- and 60-keV D^+ as compared to the pulsed irradiation can, in part, be attributed to the higher target temperature in the continuous irradiation ($\sim 260\text{--}295^\circ\text{C}$) as compared to the pulsed irradiation (surface temperature of $100\text{--}120^\circ\text{C}$).

In order to test that the observed reduction in blistering is, in fact, due to higher target temperatures, several irradiations were done with target temperatures controlled at 300°C . Figures 12a and 12b show scanning electron micrographs of polycrystalline molybdenum surfaces irradiated at 300°C with 40-keV D^+ in continuous and pulsed modes, respectively, to a total dose of 2500 pulses or its equivalent. During continuous irradiation the heating current was adjusted so as to keep the target temperatures at $\sim 300^\circ\text{C}$ during the 1 sec pulse. No blisters could be detected either for the continuous (Figure 12a) or for the pulsed (Figure 12b) irradiation and the irradiated area does not appear to be different from the unirradiated area (Figure 12c). Similarly, for 60-keV D^+ irradiation at 300°C , no blisters could be detected for both continuous (Figure 12d) and pulsed (Figure 12e) irradiation for a dose of 2500 pulses or its equivalent. This reduction in blistering in Mo at 300°C can be attributed in part to increased permeability of D^+ in Mo at 300°C as compared to room temperature. Thus, it appears that if the molybdenum surfaces can be maintained at 300°C or above, blistering due to D^+ irradiation can be reduced for the dose range studied.

b. TZM-Alloy

In order to compare the results for molybdenum with TZM-alloy, several irradiations were performed only for a few selected energies and doses. Since very little blistering was observed for Mo irradiated with 120-keV D^+ , the irradiation of TZM was done only for 40- and 60-keV D^+ bombardment. Figures 13a and 13b show scanning electron micrographs of polycrystalline TZM irradiated in a continuous mode with 40-keV D^+ to a dose of 4.3×10^{18} ions/cm² (equivalent of 2500 pulses) and with 60-keV D^+ to a dose of 8.1×10^{18} ions/cm² (equivalent of 2500 pulses), respectively. For 40-keV D^+ irradiation only a few blisters (estimated blister density $\lesssim 1 \times 10^3$ blisters/cm²) could be seen over the entire bombarded area (Figure 13a), whereas for 60-keV D^+ irradiation no blisters could be detected (Figure 13b). Figures 13c and 13d show

micrographs of TZM surfaces irradiated in pulsed mode for 2500 pulses with 40- and 60-keV D^+ , respectively. For both 40- and 60-keV irradiations, only a few blisters (estimated density of $\leq 3 \times 10^3$ blisters/cm²) were observed over the entire irradiated area. Comparing these results with those obtained for Mo [see Figures 8d, 8h, 9c, 9f, 10a and 10e] one can see that blistering is reduced in TZM alloy for both pulsed and continuous irradiation with 40- and 60-keV D^+ . The largest blister diameter observed for TZM irradiated with 60-keV D^+ in the pulsed mode is only $\sim 9 \mu\text{m}$ as compared to $\sim 20 \mu\text{m}$ for the case of Mo irradiated under similar conditions. The reason for this reduction in blistering in TZM alloy as compared to Mo is not clear at present, but it may be related to the increased yield strength of TZM as compared to molybdenum.

c. Graphite

As mentioned earlier, at the special request of PPPL, a few selected D^+ irradiations were done on two types of graphite, namely ATJ graphite and reactor grade (UT6FT) graphite. Figures 14a and 14b show scanning electron micrographs (at two different magnifications) of surfaces of ATJ graphite irradiated with 60-keV D^+ to a dose of 3.2×10^{18} ions/cm² (equivalent of 1000 pulses). For comparison, two micrographs of the unirradiated areas at comparable magnifications are shown in Figures 14c and 14d. It can be seen that many ridges have formed after irradiation (Figure 14a and 14b) and were not present before irradiation (Figures 14c and 14d). The unirradiated surface had many pores, and many areas had large holes. Figure 15a shows a micrograph of reactor grade graphite (UT6FT) irradiated in continuous mode with 60-keV D^+ to a dose of 3.2×10^{18} ions/cm² (equivalent of 1000 pulses). In this case also ridges of $\sim 0.6 \mu\text{m}$ width have formed on the surface after irradiation, ridges which were not present in the unirradiated area (Figure 15b). These results are very similar to the results obtained for ATJ graphite (e.g., see Figure 14b).

In order to see the effect of total dose on the formation of these ridges and grooves, ATJ graphite samples were irradiated with 60-keV D^+ to different doses similar to those used for molybdenum. Figures 16a, 16b and 16c show scanning electron micrographs of ATJ graphite irradiated with 60-keV D^+ for total doses of 1.6×10^{18} (equivalent of 500 pulses), 3.2×10^{18} (equivalent of 1000 pulses) and 8.1×10^{18} ions/cm², (equivalent of 2500 pulses), respectively. Even for a dose of 1.6×10^{18} ions/cm², one can clearly see the ridges formed in the irradiated area (Figure 16a). The ridges and grooves become more pronounced as the dose is increased to 3.2×10^{18} ions/cm² (Figure 16b) and then to 8.1×10^{18} (Figure 16c) ions/cm². The origin of these surface structures observed in these two types of graphite is not understood at present. Since the surface temperature during irradiation rises to $\sim 260^\circ\text{C}$, there may be some chemical attack of graphite by D^+ ions. In addition, the compaction of graphite under ion impact may induce stresses which, in turn, may cause the formation of ridges.

In Figure 17 we compare the surface structure of ATJ graphite samples which have been irradiated in a pulsed mode with 40-keV and 60-keV D^+ ions for total doses of 1000 and 2500 pulses, respectively. For both energies the formation of ridges is more pronounced at 2500 pulses than at 1000 pulses. The estimated average width of the ridges increases from about $1.3 \mu\text{m}$ to $1.7 \mu\text{m}$ as the D^+ ion energy is increased from 40-keV to 60-keV (for

the case of 2500 pulses). In this connection it is of interest to note that the calculated projected range R_p increases from $\sim 0.42 \mu\text{m}$ to $0.58 \mu\text{m}$ as the D^+ ion energy increases from 40-keV to 60-keV. A comparison of the surface structure of ATJ graphite samples which had been irradiated to the same dose in a continuous mode (2500 pulses equivalent - see Figure 16c) and in a pulsed mode (2500 pulses - see Figure 17d) reveals no major significant difference in the surface structure formed.

These results indicate that for identical ion doses and energies the surface damage in ATJ graphite is significantly more severe than in either Mo or T2M. At this time the formation of the ridges is not understood. Future work should be initiated to irradiate ATJ and reactor grade graphite with 120-keV D^+ ions in both d.c. and pulsed mode operations since the neutral beam injectors in TFTR will have a more intense 120-keV D^0 component than the 40-keV and 60-keV components. Furthermore, in future work the mechanisms leading to the severe surface damage of D^+ ion irradiated ATJ and reactor grade graphite need to be explored in order to make better choices in the methods of reducing such surface damage. Finally, it should be pointed out that the use of large graphite surface areas ("armor plates") in a fusion device which uses hydrogenic plasmas and beams may not be advisable in view of the significant trapping of hydrogenic gases (e.g. tritium trapping) in graphite.

B. Sputtering of polycrystalline Mo

In these studies we determined the total sputtering yields, S for 40-, 60- and 120-keV D^+ ion bombardment of polycrystalline Mo surfaces at normal incidence. In the order of the energy values listed above we obtained $S = (1.10 \pm 0.11) \times 10^{-3}$ atom/ion, $S = (0.75 \pm 0.07) \times 10^{-3}$ atoms/ion, and $S = (0.4 \pm 0.05) \times 10^{-3}$ atoms/ion. In Figure 18 we have plotted these yield values as a function of deuteron energy together with S -values obtained previously by H. L. Bay, J. Roth and J. Bohdansky, J. Appl. Phys. **48**, 4722 (1977) for the same ion-target system but at lower deuteron energies. The dotted curve has been calculated according to Sigmund's theory [P. Sigmund, Phys. Rev. **184**, 383 (1969)], using appropriate corrections for backscattering electronic stopping, and the surface reflection as suggested by Weissman et al. [Rad. Effects **19**, 7, 69 (1973)], and by Anderson et al. [J. Appl. Phys. **46**, 2416 (1975)], and by fitting a calculated value with the experimental value at 2 keV. In general, the calculated curve seems to represent the energy dependence of the yield value quite well for the energy range shown here, although relatively small deviations at lower and higher energies can be observed. However, without fitting the calculated curve to an experimental value near S_{max} , the calculated values would be a factor 6.5 to 8 larger, as we have shown earlier (M. Kaminsky, Proceed. VII International Conf. Atomic Collisions in Solids, Moscow, USSR, September 1977).

At this time we have only some very tentative indications that S -values for light ion bombardment (e.g. H^+ , D^+ , He^+) depend on the total dose. If we can substantiate this in future studies, then the yield values reported here should be considered as being typical only for the dose range studied. In the summary, we have already pointed out that based on the yield values reported here, the thickness loss of Mo "armored plates" due to sputtering by

40-, 60-, and 120-keV D^+ ions would be negligibly small ($\approx 150\text{\AA}$ for even a long period of 10,000 deuterium pulses and for the deuterium fluxes estimated by Dr. J. Cecchi. However, during a 1 sec deuterium pulse an average Mo impurity concentration $n_i \approx 1.5 \times 10^{10}$ atom cm^{-3} could be built up in a TFTR plasma, resulting in a $Z_{\text{eff}} \approx 1.31$. This in turn will cause an increase in the plasma radiation losses (e.g., a 32% increase in the bremsstrahlung losses of a hydrogenic plasma, with plasma density of 1×10^{14} atoms/ cm^3 , if the Mo impurity atoms are fully stripped).

CONCLUSIONS

For the same deuteron energy and total dose, the average blister diameter in Mo is smaller for continuous irradiation than for pulsed irradiation for both 40- and 60-keV D^+ irradiation. For Mo target temperatures of 300°C or above, no blisters are observed in either pulsed or continuous irradiation. On the basis of the results obtained for Mo and TZM, it appears that although both show blistering, no severe surface erosion will be expected in a beam dump made out of Mo or TZM, particularly if the surfaces can be maintained at elevated temperatures (300°C or higher). Based on limited results, TZM appears to be a better choice than Mo as a beam dump material, since, for ambient target temperatures and similar irradiation conditions, it showed less blistering than Mo. For both ATJ and reactor grade (UT6FT) graphite the surface damage induced by 40-, or 60-keV D^+ ion irradiation is significantly more severe than for Mo and TZM surfaces under comparable irradiation conditions. The mechanisms (chemical and physical) contributing to the surface damage of the irradiated graphite samples is not understood at this time. If graphite continues to be considered as a candidate material for protective plates in TFTR it appears necessary to conduct deuteron irradiations at 120-keV for the appropriate fluxes, and to study the mechanisms contributing to surface damage.

The total sputtering yields S for 40-, 60- and 120-keV D^+ ion irradiation of polycrystalline Mo surfaces at normal incidence were determined to be $S = (1.10 \pm 0.11) \times 10^{-3}$ atoms/ion, $S = (0.75 \pm 0.07) \times 10^{-3}$ atoms/ion, and $S = (0.4 \pm 0.05) \times 10^{-3}$ atoms/ion, respectively. Based on these S -values and an estimated total dose of 10^4 pulses of D^+ ions striking the protective plates, it has been concluded that the thickness loss of Mo protective plates due to sputtering will be negligible. The buildup of Mo impurities in the TFTR plasma could reach concentrations of $n_i = 1.5 \times 10^{10}$ atoms cm^{-3} during one pulse, corresponding to an impurity fraction of $n_i/n_p = 1.8 \times 10^{-4}$ for a plasma density $n_p = 1 \times 10^{14}$ atoms cm^{-3} . Such a relatively small value for the impurity fraction will cause only a moderate increase in the plasma radiation losses (e.g., a 32% increase in the bremsstrahlung losses of a hydrogenic plasma if the Mo impurity atoms are fully stripped).

FIGURE CAPTIONS

- FIGURE 1 Photograph of the beam line after the analyzing magnet, showing magnetic quadrupole, liquid nitrogen traps and the target chamber.
- FIGURE 2 Photograph of the target chamber at the end of the beam line showing the position of infrared pyrometer for measuring target surface temperature.
- FIGURE 3 Photograph of the stepping motor - target micromanipulator assembly.
- FIGURE 4 A closeup view of the target assembly showing the arrangement of the thermocouple and the shielded biased aperture.
- FIGURE 5 Auger spectrum of (a) as electropolished molybdenum surface (b) the same area as in (a) but after 210 minutes of irradiation with 2-keV Argon ions (sputter-cleaning process).
- FIGURE 6 Rutherford backscattering spectrum of a ~ 4.5 monolayer thick Mo deposit on a Si(III) substrate (resulting from a 60-keV D^+ Mo sputtering run) obtained with a 750-keV primary $^4He^+$ ion beam and for a scattering angle of 154° .
- FIGURE 7 Variation of the Mo deposit thickness t as a function of distance R on the substrate as measured from the center of the deposit spot ($R=0$) along radial directions. The solid curve is based on calculated values using equation (1). The experimentally determined values are indicated by $\dagger$.
- FIGURE 8 Scanning electron micrographs (SEMs) of polycrystalline Mo irradiated surfaces (a-d) with 40-keV D^+ for 100 pulses in (a), 500 pulses in (b), 1000 pulses in (c), 2500 pulses in (d); (e-h) with 60-keV D^+ for 100 pulses in (e), 500 pulses in (f), 1000 pulses in (g), 2500 pulses in (h); (i-l) with 120-keV D^+ for total doses equivalent to 500 pulses in (g), 1000 pulses in (h) and 2500 pulses in (i).
- FIGURE 9 SEMs of polycrystalline Mo surfaces irradiated in the continuous mode (a-c) with 40-keV D^+ for total doses equivalent to 500 pulses in (a), 1000 pulses in (b), 2500 pulses in (c); (d-f) with 60-keV D^+ for total doses equivalent to 500 pulses in (d), 1000 pulses in (e), 2500 pulses in (f); (g-i) with 120-keV D^+ for total doses equivalent to 500 pulses in (g), 1000 pulses in (h) and 2500 pulses in (i).

- FIGURE 10 SEMs of polycrystalline Mo surfaces irradiated to a dose of 2500 pulses or its equivalent (a) with 40-keV D^+ in the pulsed mode, (b) with 40-keV D^+ in continuous mode, (c) an unirradiated area, (d) with 60-keV D^+ in pulsed mode, (e) with 60-keV D^+ in continuous mode, (f) with 120-keV D^+ in pulsed mode, (g) with 120-keV D^+ in continuous mode.
- FIGURE 11 Histograms of the percentage of blisters in a class for polycrystalline Mo irradiated (a) with 40-keV D^+ in pulsed mode, (b) with 60-keV D^+ in pulsed mode, (c) with 40-keV D^+ in continuous mode and (d) with 60-keV D^+ in continuous mode.
- FIGURE 12 SEMs of polycrystalline Mo irradiated at 300°C for a dose of 2500 pulses or its equivalent (a) with 40-keV D^+ in a continuous mode, (b) with 40-keV D^+ in pulsed mode, (c) an unirradiated area of the target surface, (d) with 60-keV D^+ in continuous mode, and (e) with 60-keV D^+ in pulsed mode.
- FIGURE 13 SEMs of polycrystalline TZM alloy irradiated to a dose of 2500 pulses or its equivalent with (a) 40-keV D^+ in continuous mode, (b) 60-keV D^+ in continuous mode, (c) 40-keV D^+ in pulsed mode and (d) 60-keV D^+ in pulsed mode.
- FIGURE 14 (a-b) ATJ graphite irradiated with 60-keV D^+ to a total dose equivalent to 1000 pulses, (c-d) unirradiated area of the ATJ graphite surface shown at two different magnifications.
- FIGURE 15 (a) Reactor grade (UT6FT) graphite irradiated with 60-keV D^+ to a total dose equivalent to 1000 pulses, (b) an unirradiated area of the same graphite.
- FIGURE 16 ATJ graphite irradiated with 60-keV D^+ in continuous mode for total doses equivalent to (a) 500 pulses, (b) 1000 pulses and (c) 2500 pulses.
- FIGURE 17 SEMs of ATJ graphite irradiated in a pulsed mode with (a,b) 40-keV D^+ for 1000 and 2500 pulses, respectively, and (c,d) 60-keV D^+ for 1000 and 2500 pulses, respectively.
- FIGURE 18 The dependence of the sputtering yield S on the incident ion energy for polycrystalline molybdenum targets bombarded by deuterons at normal incidence. Experimental values marked (Δ) obtained by Bay et al [JAP 48, 4722 (1977)], and those marked ($\circ$) see this report. The solid line has been interpolated between experimental values. The dotted curve has been calculated according to Sigmund's theory using appropriate corrections for backscattering, electronic stopping, and surface reflection.

ARGONNE NATIONAL LABORATORY

May 26, 1977

Dr. Joseph L. Cecchi
Plasma Physics Laboratory
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PO Box 451
Princeton, New Jersey 08540

Dear Joe,

We have used the weekends over the last four weeks to modify and test a low-energy accelerator system for the D^+ ion irradiation experiments you had suggested in your letter of April 20, 1977. We needed to set up a beam handling system (quadrupoles, beam steerers), a target chamber and a high vacuum system (differential pumping). After completion of the assembly we have tested the performance of this accelerator system. I am able to guarantee the following performance: At 40, 60 and 120 keV a D^+ flux of 1.5×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$ on a 2 mm diam. target spot, held at 2×10^{-8} Torr (unbaked) during irradiation. Under optimum conditions we seem to be able to reach at 60 keV, 2.5×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$, and at 120 keV, 3×10^{15} ions $\text{cm}^{-2} \text{sec}^{-1}$, however, I feel I cannot guarantee these fluxes on a continuous basis. The target spot size has been proven to be large enough in previous experiments to obtain good statistics on blister density and surface erosion (flaking, sputtering).

We could set up an irradiation program for the weekends (Saturday and Sunday, 24 hours a day, 48 hours per weekend) for the months of June, July, August and September 1977, with the exception of the Fourth of July weekend and the Labor Day weekend. Since we have to operate at accelerators with two men per shift (safety regulation), we would operate a total of 90 shifts (eight hours/shift or 180 man-days). In addition, we need scientific effort (the equivalent of one man-month) in target analysis prior to and after irradiation and in the data analysis. We also need help by an assistant (equivalent of two man-months) for target preparation (e.g., optical, polishing, etching, electropolishing, cutting of target slices, target procurement). In addition, funds for materials and services (e.g., repair of source filaments, construction of beam pulsing device, SEM-emitters, photographic films, portable vacuum system, cold fingers) are needed. While I am not authorized to give you a final cost estimate I would guess the total costs to reach \$60K.

Dr. Joseph L. Cecchi

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Following your suggested outline of TFTR-surface experiments (your letter of April 20, 1977) I propose the following experiments:

- (1) Studies of blistering of beam dump armor plates("beam dumps") made out of Mo and TZM.
 - (a) In one set of experiments we would expose Mo targets (commercial grade or Marz grade, cold rolled, polished) to D^+ ion pulses (~ 1 min repetition rate) for a total of 100, 500, 1000 and 2500 pulses. These irradiations will be conducted for the following conditions:

Species	Energy (keV)	Flux (ion $cm^{-2} sec^{-1}$)
D^+	120	1.5×10^{15} (3×10^{15}) $_{max}^*$
D^+	60	1.5×10^{15} (2.5×10^{15}) $_{max}^*$
D^+	40	1.5×10^{15} (1.8×10^{15}) $_{max}^*$

* the figures in parenthesis are not guaranteed

We plan to monitor near surface temperatures with a thermocouple, and for surface temperatures above $\sim 2800^\circ C$, we will use infrared pyrometry. In this set of experiments no target temperature control will be applied in order to determine the beam heating effect (as you suggested in our phone conversation). If you want target temperature control for some runs, we could do so with the aid of special feed throughs. Please indicate to us if you want to use 3/4 inch thick Mo-armor plate sections (which you would supply) or if we could use Mo-foils (~ 0.005 inch thick) mounted on a $\sim 3/4$ inch metal block (e.g., Cu, stainless steel). During the irradiation the targets will be held at $\sim 2 \times 10^{-8}$ Torr background pressure. The targets will be examined by SEM after the irradiations and measurements of blister density, blister diameter distributions, and surface erosion due to blister rupture and exfoliation will be conducted. Since the SEM has a resolution of 80-100 $\text{\AA}$ any serious cratering due to vaporization (beam heating) could be detected. The data would be evaluated and prepared for an interim report to be issued in August 1977.

- (b) In a second set of experiments the Mo-targets would be exposed to a continuous D^+ ion bombardment for total fluences of 3×10^{18} ions $cm^{-2} sec^{-1}$ and 6×10^{18} ions $cm^{-2} sec^{-1}$ which would show the integrated effect of about 1.5-3 thousand pulses. We would compare the surface damage caused by the pulsed-mode irradiation [see (a)] with the one obtained by continuous irradiation. These experiments would be conducted for the same D^+ ion energies and fluxes listed under (a).

Dr. Joseph L. Cecchi

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- (c) The experiments listed under (a) and (b) will be extended to another material, TZM, a molybdenum alloy, which supposedly shows a small "swelling" effect. These experiments will not follow the entire schedule listed under (a) and (b) but only a selection of parameters to insure that the general trends observed for Mo also hold for TZM with the hope, of course, that TZM will display in general a reduced blistering effect in comparison to Mo. An interim report of some of the pertinent results of these studies will be issued during September 1977.

(2) Measurement of sputtering yields for Mo under D^+ ion bombardment.

I propose to conduct D^+ irradiation experiments of Mo for the same ion energies and fluxes shown in the table under (1a), and to collect the sputtered material on monocrystalline Si (111) collector surfaces. The amount of the sputter deposit will be determined by (a) Rutherford back-scattering and (b) by sputter profiling using our scanning Auger spectrometer. We may, as a third independent check, use the ion microprobe in a few cases. While these irradiations are expected to be finished by September 1977, it is anticipated that we will carry out some of the sputter deposit determinations into the months of October and November 1977. It appears feasible that we will be able to provide some data for 1 to 2 keV D^+ ion sputtering of Mo before the end of 1977.

I anticipate that we would prepare a final report of the measurements listed under (1) and (2) at the end of calendar year 1977 and issue it at the beginning of 1978.

(3) Low-Z surface coatings for SS (304 LN) plate sections.

We consider this development program as very interesting and very important for TFTR first-wall applications. In view of the few months available in FY 77, however, we feel we cannot obtain significant information which would be useful to you. We would be most interested in FY 1978 to start such a program (e.g., BN, B_4C , BeO on stainless steel) if needed funding could be made available. This would be a cooperation program between ANL's Physics Division, Materials Science Division and outside vendors. The work would include selection of suitable Low-Z coating materials, specification of coating processes (e.g., plasma spraying vs. chem. vapor deposition) and process parameters (e.g., grain size, coating temperature, substrate pretreatment), qualification tests of specimens with respect to procurement specifications, structural characterization (e.g., phases present, chem. composition, grain size, pore morphology), bond strengths, chemical stability, and resistance to various surface radiation effects. A crude estimate of the minimum costs involved would be about \$220K during FY 1978. Again, I am not authorized to give you a binding estimate. I am convinced, however, that this program would have to be extended for at least one additional year (FY 1979) at the same funding level (or a higher one) to obtain meaningful data.

Dr. Joseph L. Cecchi

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I very much hope that you will find the general outline of this proposed work acceptable. I am looking forward to your comments and suggested changes in the scope of the work. Since we would like to start with the work on June 4, 1977, (in order not to lose more time) I hope we can settle on the contractual agreement very soon. I will submit this letter to Dr. Gerald T. Garvey, the director of the Physics Division, for further processing.

Sincerely yours,

Manfred Kaminsky

MK:LN

FIGURE 1

FIGURE 2

FIGURE 3

FIGURE 4

FIGURE 5a

FIGURE 5b

FIGURE 6

FIGURE 7

POLYCRYSTALLINE Mo IRRADIATED WITH D^+ IN PULSED MODE

FIGURE 8

POLYCRYSTALLINE MOLYBDENUM IRRADIATED WITH D^+ IN CONTINUOUS MODE

500 PULSES EQ.

1000 PULSES EQ.

2500 PULSES EQ.

FIGURE 9

POLYCRYSTALLINE MOLYBDENUM IRRADIATED WITH D^+ TO A DOSE OF 2500 PULSES OR ITS EQUIVALENT

FIGURE 10

FIGURE 11

POLYCRYSTALLINE MOLYBDENUM IRRADIATED AT 300°C WITH D^+

FIGURE 12

POLYCRYSTALLINE TZM ALLOY IRRADIATED WITH D^+ TO A DOSE OF
2500 PULSES OR ITS EQUIVALENT

CONTINUOUS

PULSED

40-keV

60-keV

FIGURE 13

ATJ GRAPHITE IRRADIATED WITH 60- KEV D^+ TO A DOSE OF 0.52 C/CM²

IRRADIATED AREA

UNIRRADIATED AREA

FIGURE 14

IRRADIATED AREA

UNIRRADIATED AREA

FIGURE 15

ATJ GRAPHITE IRRADIATED WITH 60-KEV D^+ IN CONTINUOUS MODE

500 PULSES EQ.

1000 PULSES EQ.

2500 PULSES EQ.

FIGURE 16

1000

2500

40

60

FIGURE 17