

Conf-751125-73

IRRADIATION OF GRAPHITE CLOTH
AT VARIOUS TEMPERATURES
WITH DEUTERONS AND HELIUM IONS

By

R. Ekern, S. K. Das and M. Kaminsky

MASTER

Prepared For Presentation at
6th Symposium on
Engineering Problems of Fusion Research
November 18-21, 1975
San Diego, CA.

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.



U of C-AUA-USERDA

ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

DISTRIBUTION OF THIS REPORT IS UNLIMITED

operated under contract W-31-109-Eng-38 for the
U. S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

IRRADIATION OF GRAPHITE CLOTH AT VARIOUS TEMPERATURES WITH DEUTERONS AND HELIUM IONS[†]

R. Ekern, S. K. Dan, and M. Kaminsky
Argonne National Laboratory, Argonne, Illinois 60439

Summary

Graphite cloth samples were irradiated with 100 keV deuterons and $^4\text{He}^+$ ions at room temperature and at elevated temperatures. Scanning electron microscopy was used to examine the surfaces of irradiated and unirradiated graphite fibers. Irradiation at room temperature with $^4\text{He}^+$ to a total dose of 3.1×10^{18} ions cm^{-2} produces considerable flaking of individual fibers, which is not observed on unirradiated fibers. Identical irradiations at 400° and 800°C with $^4\text{He}^+$ did not produce any detectable flaking or other surface damage. The elevated temperatures apparently prevent an accumulation of helium in localized areas which in turn could cause flaking in near surface regions. Results obtained for deuteron bombardment of graphite cloth at room temperature and at 600°C are also discussed.

Introduction

Charged and uncharged particles from the plasma of a controlled thermonuclear fusion reactor will interact with the surfaces of exposed components and cause (a) surface erosion and (b) plasma contamination. The plasma contamination caused by the release of impurities from the surfaces of irradiated components is not only a potential problem for future fusion reactors but is also a current problem in large size plasma devices. The impurity release can be caused by such surface effects as physical and chemical sputtering, vaporization, blistering, particle impact induced desorption, and other effects discussed in more detail elsewhere^{1,2}. The impurities reaching the plasma can have an important effect on the power balance of fusion devices and reactors. For example, the addition of high-Z impurity atoms (or ions) to a D-T plasma will increase the effective charge Z_{eff} of the plasma, where Z_{eff} is given by $Z_{\text{eff}} = (1 + \sum_i f_i Z_i^2) / (1 + \sum_i f_i Z_i)$. In this equation, f_i is the impurity fraction of species i ($f_i = n_i/n_{D,T}$), n_i is the impurity atom (ion) density, $n_{D,T}$ is the D-T plasma density (assuming $n_D = n_T$), and Z_i is the charge of species i . An increase in the value of Z_{eff} due to an increase in the impurity concentration n_i and/or in the atomic number Z_i results not only in an enhanced plasma resistivity, but also affects the plasma containment time³⁻⁵. For example, for Argonne's preliminary conceptual design of the experimental power reactor⁶ a value of Z_{eff} of about 3

would prevent ignition of the reactor without additional power injection. (If the impurities were molybdenum alone, an impurity fraction of 0.13 per cent is already sufficient to yield $Z_{\text{eff}} = 3.1$, raising the minimum ignition temperature to 30 keV.) Furthermore, heavy impurities tend to diffuse towards the plasma center and collect on the axis of the discharge and thereby seriously affect the steady state operation of a reactor^{7,8}. In addition, the impurities can cause plasma power loss via bremsstrahlung, line, and recombination radiation. If these losses increase above critical

levels they can cool the plasma temperature below fusion reaction temperatures. It can be seen from the definition of Z_{eff} that for the same value of Z_{eff} a higher concentration of an impurity of low Z can be tolerated than for a high Z impurity.

A possible solution to the problems of plasma contamination and surface erosion has been offered by Kulcinski et al.⁹. A flexible two-dimensionally woven graphite cloth will be placed between the plasma and the metallic vacuum wall of a fusion reactor. This cloth intercepts most of the ions and neutral atoms leaking from the plasma as well as a fraction of the photons. It will also collect most of the impurities released from the irradiated surfaces and prevent their reaching the plasma. The graphite curtain, of course, will release carbon impurities into the plasma, but because of the lower Z , concentrations of several per cent can be tolerated without affecting the plasma properties seriously⁹.

In order to assess the possible advantages of a graphite curtain for use in fusion reactors, it is necessary to study the effects of irradiation on this material. This paper will address the specific problem of irradiation of graphite cloth at room temperature and at elevated temperatures with energetic deuterons and helium ions.

Experimental Procedures

The graphite cloth (WCA cloth, manufactured by Union Carbide) used in these experiments was furnished by Professor G. Kulcinski of the University of Wisconsin - Madison. The graphite fibers used for the cloth were produced by pyrolysis of rayon fibers with subsequent heating to graphitization temperature.^{9,10} The graphite fibers have diameters varying from 6 to 10 μm . The graphite yarn used for the cloth consists of bundles of 700 - 1000 continuous fibers and is woven in a square weave pattern (see Fig. 1a). Graphite cloth targets of approximately 3 cm x 1 cm were clamped to an insulating substrate with OFHC copper busbars and mounted in a high vacuum chamber. The targets were irradiated with a mass- and energy-analyzed beam of 100 keV D^+ and $^4\text{He}^+$ ions from a 2 MeV Van de Graaff accelerator. During irradiation a vacuum of $\sim 5 \times 10^{-8}$ Torr was maintained in the target chamber by ion pumping. The dose rate for both D^+ and $^4\text{He}^+$ irradiations was 5×10^{13} ions $\text{cm}^{-2} \text{sec}^{-1}$ and the total dose was 1.9×10^{18} ions cm^{-2} and 3.1×10^{18} ions cm^{-2} for the D^+ and $^4\text{He}^+$ irradiations, respectively. To irradiate the targets at elevated temperatures direct resistive heating was used. The target temperatures were measured with an infrared pyrometer; an emissivity of 0.8 was assumed and corrections for transmission of the glass window of the vacuum system were made. The temperature across the irradiated area (0.3 cm diameter) was found to be uniform within 2-5 per cent. The irradiated targets were examined following irradiation in a Cambridge Stereoscan S4-10 scanning electron microscope.

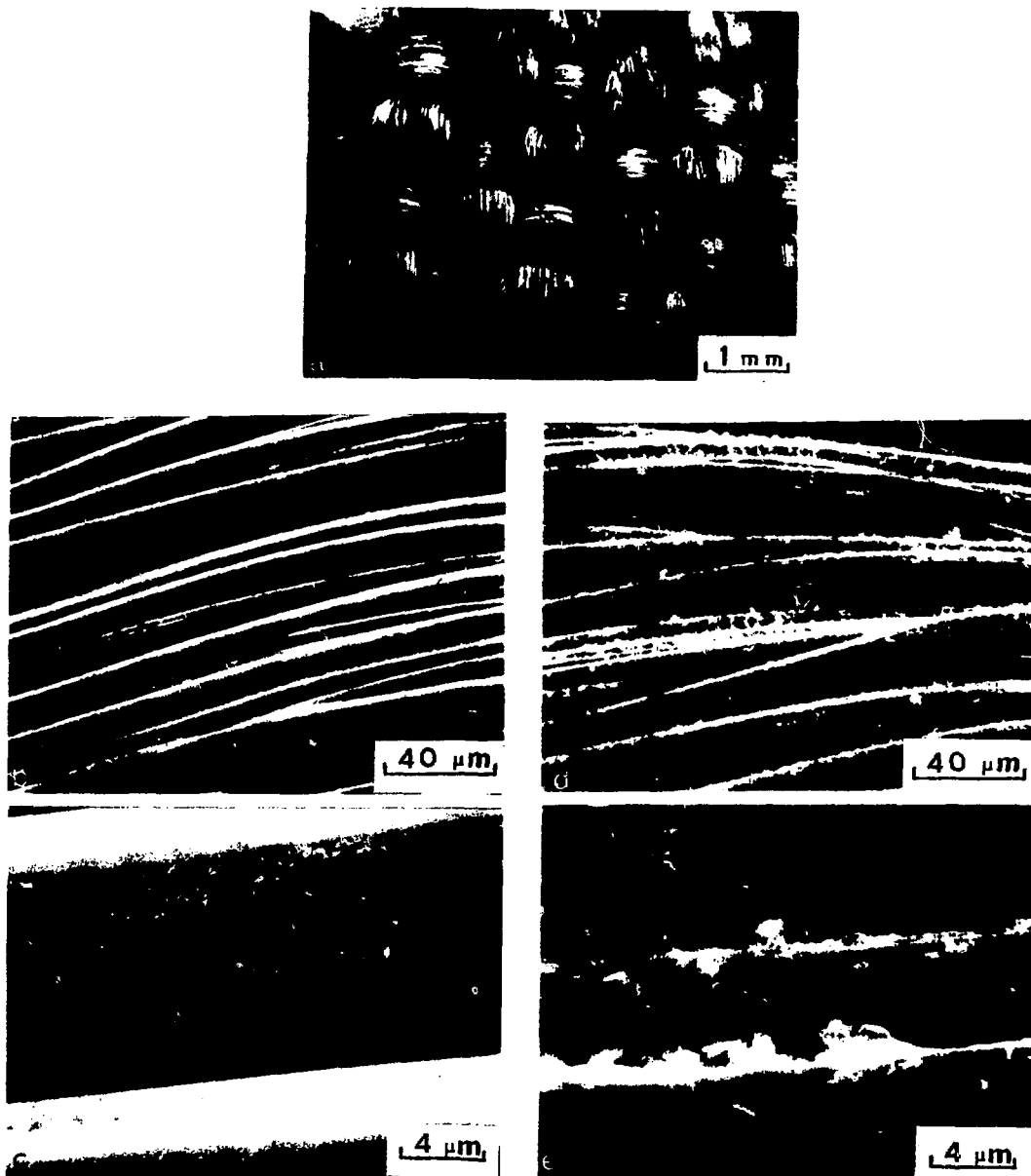


Fig. 1 Scanning electron micrographs of graphite cloth, (a) - (c) unirradiated areas for three different magnifications, (d) and (e) high magnification views of fibers in the area irradiated with 100 keV $^4\text{He}^+$ ions to a total dose of 3.1×10^{18} ions cm^{-2} where flaking and loss of material is observed (room temperature irradiation).

Results

Scanning electron micrographs of unirradiated areas of graphite cloth are shown for three different magnifications in Figure 1 (a)-(c). The nearly cylindrical shape of the fibers can be made out readily in Figures 1(b) and (c). Some small furrows running parallel to the long axis of the fibers can be seen also in Figure 1 (c). The average fiber diameter is found to be $\sim 8 \mu\text{m}$. Figures 1 (d) and (e) show at two different magnifications an irradiated area of the

cloth which had been bombarded with 100 keV $^4\text{He}^+$ ions to a dose of 3.1×10^{18} ions cm^{-2} . If one compares Figures 1 (b) and 1 (d) one notices that the irradiated fibers show significant flaking. Micron size flakes can be seen readily. At higher magnifications, the flakes are observed to protrude randomly from the fiber, shown for a typically damaged fiber in Figure 1 (e). Also, a significant amount of material has been removed from the fibers. The depth of the furrows running parallel to the fiber axis has been increased by the irradiation since such deep furrows

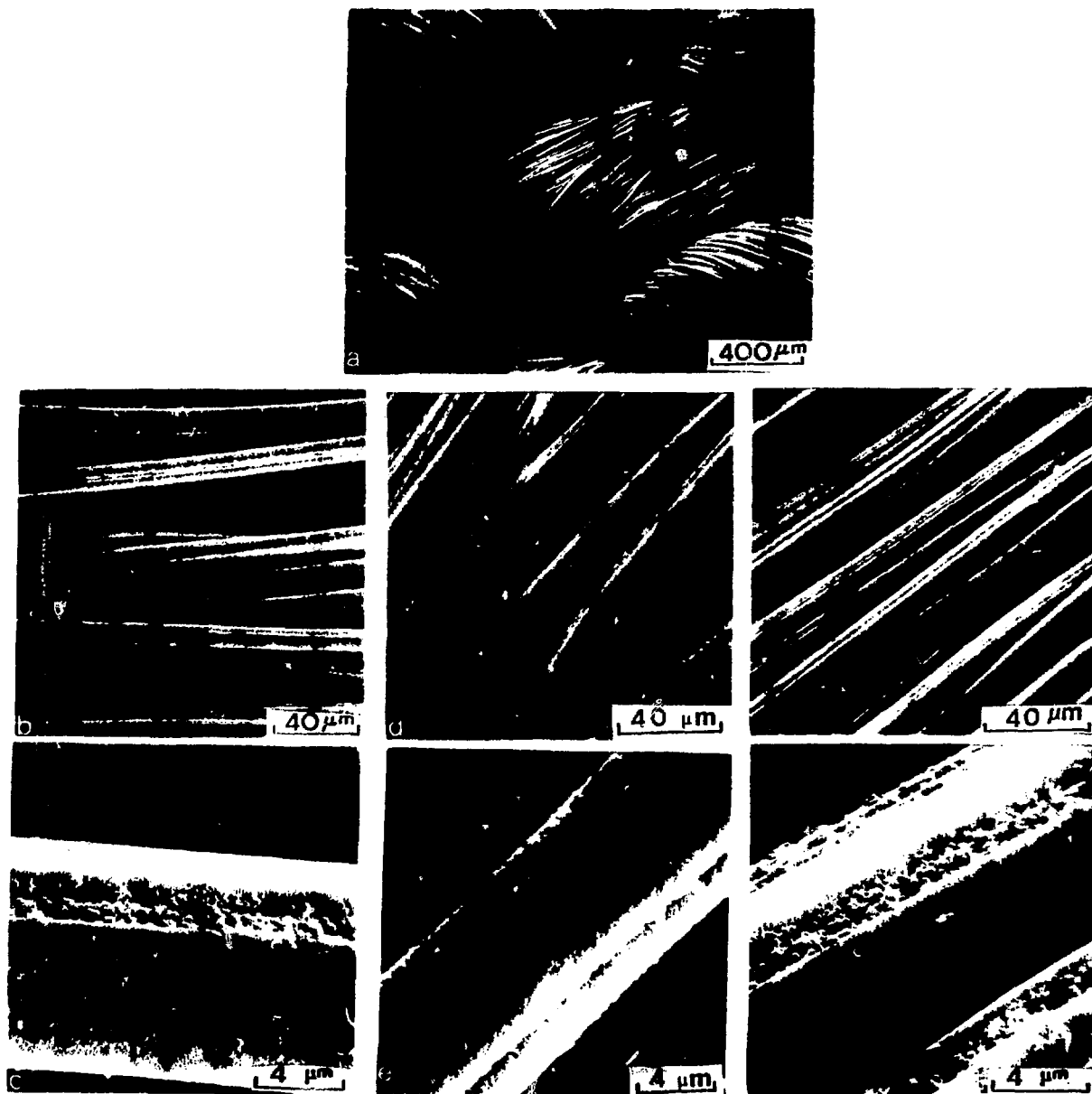


Fig. 2 Scanning electron micrographs of graphite cloth, (a)-(c) unirradiated areas for three different magnifications, (d) and (e) high magnification views of fibers in the area irradiated at 400°C with 100 keV $^4\text{He}^+$ ions to a total dose of 3.1×10^{18} ions cm^{-2} , and (f) and (g) high magnification views of fibers irradiated at 800°C under otherwise identical conditions as in (d) and (e).

are not observed in unirradiated fibers. In this connection, it should be mentioned that the projected range of a 100 keV $^4\text{He}^+$ ion in carbon is approximately 1 μm . It is near the end of this range where most of the lattice damage is expected to occur. (For a related discussion of projected ranges and damage energy distributions in certain metals and some ceramics, see references 11 and 12).

Figures 2 (a)-(c) show scanning electron micrographs at three different magnifications of unirradiated areas of a graphite cloth which was used in

irradiation experiments at an elevated temperature. Again, small furrows parallel to the fiber axis can be made out in Figure 2(c). Figures 2(d) and (e) show at two different magnifications fibers which have been irradiated at 400°C with 100 keV $^4\text{He}^+$ ions to a dose of 3.1×10^{18} ions cm^{-2} . In comparison with the room temperature irradiation shown in Figure 1(e) no significant flaking can be observed, and no noticeable loss of material from the furrows of a fiber seems to occur. Figures 2(f) and (g) show fibers irradiated at an even higher temperature of 800°C under otherwise identical

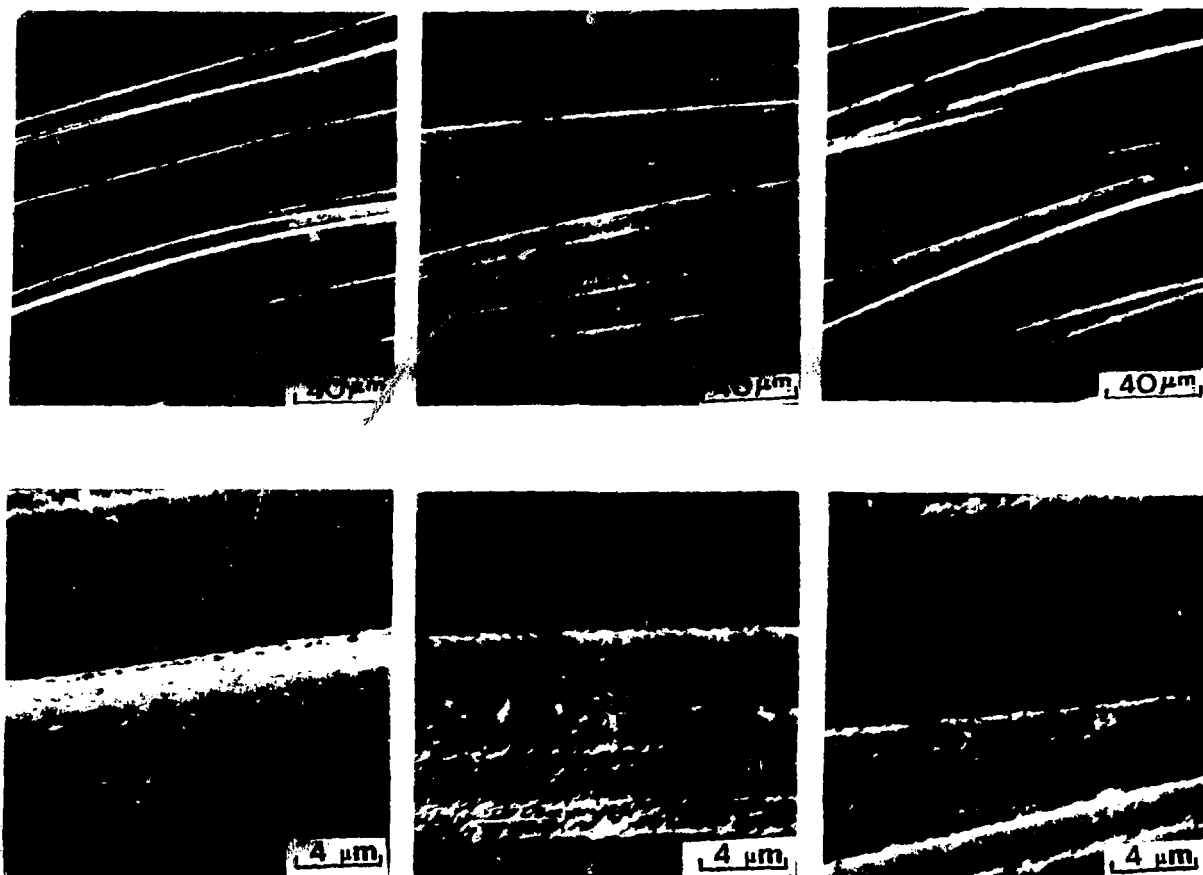


Fig. 3 Scanning electron micrographs of graphite cloth, (a) and (b), unirradiated areas for two different magnifications, (c) and (d) high magnification views of fibers in the area irradiated at room temperature with 100 keV deuterons to a total dose of 1.9×10^{18} ions cm^{-2} , and (e) and (f) high magnification views of fibers irradiated at 600°C under otherwise identical conditions as in (c) and (d).

conditions. Again, no flaking can be observed and the unirradiated and irradiated fibers look nearly identical.

Scanning electron micrographs of a graphite cloth used in irradiation experiments with deuterons are shown in Figure 3. Figures 3 (a) and (b) show unirradiated areas of the graphite cloth at two different magnifications. Figure 3 (c) and (d) show at two different magnifications fibers which have been irradiated at room temperature with 100 keV D^+ ions to a dose of 1.9×10^{18} ions cm^{-2} . One should note that

this is a smaller dose than the one used for the $^4\text{He}^+$ ion irradiations. The surfaces of the irradiated fibers shown in Figure 3 (d) appear more roughened than those shown in Figure 3 (b) (unirradiated fibers). They also appear to have more protrusions and small flakes than those shown in Figure 3 (b). Figures 3 (e) and (f) show at two different magnifications fibers which have been irradiated at 600°C with D^+ ions under identical conditions to those mentioned above. The surfaces of the fibers appear to be rougher than those for unirradiated fibers (See Figure 3 (b)), but not as rough as the fibers irradiated at room temper-

ature (See Figure 3 (d)). The surfaces also show a smaller number of protrusions and flakes than those shown in Figure 3 (d).

Discussion

The surface damage of graphite fibers irradiated at room temperature by 100 keV ^4He ions to a dose of 3.1×10^{18} ions cm^{-2} is found to be considerable. However, identical irradiations at 400°C and at 800°C show no significant surface damage. Similarly, the surface damage caused by 100 keV D^+ ions to a dose of 1.9×10^{18} ions cm^{-2} is more significant at room temperature than at 600°C. The elevated temperatures apparently prevent an accumulation of helium and deuterium in localized areas which in turn helps to prevent flaking of the surface region. Since the operating temperature of the graphite curtain can be 800°C or higher⁹, the observed results indicate that the erosion due to helium and deuterium impact may not be a serious problem during reactor operations. In this connection it is also of interest to note that recent results on sputtering of graphite with H^+ ions¹³ also show a dramatic reduction in the sputtering coefficient at temperatures above ~ 800°C.

Acknowledgements

We are grateful to Professor G. L. Kulcinski from the University of Wisconsin-Madison, and to Dr. D. L. Kummer, McDonnell Douglas Astronautics Co. - Kent, for supplying us with samples of graphite curtain. We also like to thank Mr. P. Dumza for his help in irradiating the samples.

References

- [†]Work performed under the auspices of the Energy Research and Development Administration.
- ^{††}The degree of graphitization of the cloth fibers is not known.
- ¹M. Kaminsky, "Plasma Contamination and Wall Erosion in Controlled Thermonuclear Fusion Devices and Reactors", in Plasma Physics and Controlled Nuclear Fusion Research, Vol. II, IAEA (Vienna, 1975).
- ²M. Kaminsky, Int. Summer School in Surf. Science, University of Wisconsin-Milwaukee (Aug. 1975).
- ³J. T. Hogan and J. F. Clarke, "Fluxes of Charged and Neutral Particles from Tokamaks", in Surface Effects in Controlled Fusion, H. Wiedersich, M. Kaminsky, and K. Zwilsky, editors (North-Holland, Amsterdam, 1974), pg. 1.
- ⁴D. Dimock, et al., Report No. MATT-906, Princeton Plasma Physics Laboratory, (1972).
- ⁵L. Artsimovich, Nucl. Fusion 12, 215 (1972).
- ⁶W. Stacey, Jr. et al., Tokamak Experimental Power Reactor Studies, ANL/CTR-75-2, Argonne National Laboratory (June, 1975).
- ⁷D. Duchs, G. Haas, D. Pfirsch, and H. Vernickel, "On the Impurity Problem in Quasi Steady-state Toroidal Plasma Experiments and Fusion Reactors", see reference 3, pg. 102.
- ⁸J. Taylor, Phys. Fluids 4, 1142 (1961).

- ⁹G. Kulcinski and R. Conn, Nucl. Fusion 15, 327 (1975).
- ¹⁰G. Kulcinski, et al., UWMDM-108, University of Wisconsin-Madison (Aug. 1974).
- ¹¹M. Kaminsky, S. Das, G. Fenske, Appl. Phys. Lett. 27, 521 (1975).
- ¹²S. K. Das, M. Kaminsky and G. Fenske, Proc. Int. Conf. Appl. on Beams to Materials (Sept. 1975).
- ¹³N. P. Busharov, E. A. Gorbakov, V. M. Gusev, M. I. Guseva, and Yu. V. Martinenko, I. V. Kurchatov Order of Lenin Atomic Energy Institute, Moscow, 1975 (ANL-TRANS-996, Argonne National Laboratory, Oct. 1975).