CONF-760209--14

Reduction of Surface Erosion Caused by Helium Blistering in Sintered Beryllium and Sintered Aluminum Powder

S. K. Das and M. Kaminsky

Prepared for Presentation

2nd International Conference
on Surface Effects in Controlled Fusion Devices
Sandia Laboratories - ERDA
San Francisco, Ca.
February 16-20, 1976

ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

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

SAN FRANCISCO,CA 2/16-20/76
2nd International Conference on Surface Effects in Controlled Fusion Devices

To be published in Journal of Nuclear Materials - "As Proceedings"

Reduction of Surface Erosion Caused by Helium Blistering in Sintered Beryllium and Sintered Aluminum Powder†

S. K. Das and M. Kaminsky
Argonne National Laboratory, Argonne, Illinois 60439

Abstract

Studies have been conducted to find materials with microstructures which minimize the formation of blisters. A promising class of materials appears to be sintered metal powder with small average grain sizes and low atomic number Z. Studies of the surface erosion of sintered aluminum powder (SAP 895) and of aluminum held at 400°C due to blistering by 100 keV helium ions have been conducted and the results are compared to those obtained earlier for room temperature irradiation. A significant reduction of the erosion rate in SAP 895 in comparison to annealed aluminum and SAP 930 is observed. In addition results on the blistering of sintered beryllium powder (type I) irradiated at room temperature and 600°C by 100 keV helium ions are given. These results will be compared with those reported recently for vacuum cast beryllium foil and a foil of sintered beryllium powder (type II) which was fabricated differently, than type I. For room temperature irradiation only a few blisters could be observed in sintered beryllium powder type I and type II and they are smaller in size and in number than in vacuum cast beryllium. For irradiation at 600°C large scale exfoliation of blisters was observed for vacuum cast beryllium but much less exfoliation was seen for sintered beryllium powder, type I, and type II. The results show a reduction in erosion rate in both types of sintered beryllium as compared to the erosion rate in vacuum cast beryllium, for both room temperature and 600°C.

Introduction

During the operation of thermonuclear reactors the surface of components exposed to bombardment by energetic helium ions from the plasma region can be seriously eroded by radiation blistering [1-4]. Recently, attempts have been made [5] to reduce surface erosion due to helium blistering. One possible way to reduce surface erosion due to helium blistering in fusion reactor components is to maintain the surfaces at a high enough temperature (e.g. > 900°C for Nb and V) so that some of the implanted helium is released without forming large bubbles [3, 6 7]. However, the operating temperatures of various components may be limited by other design criteria. A more desirable solution would be to choose a material with a microstructure which minimizes the formation of blisters. A promising class of materials appear to be sintered metal powders with small average grain sizes and of low atomic number Z. Initial studies of the surface erosion of sintered aluminum powder (SAP 895) and of annealed aluminum held at room temperature due to blistering by helium ions (100 keV, dose 1.0 C cm^{-2}) reveal a high reduction in the erosion rate in SAP by more than three orders of magnitude as compared to the erosion rate of annealed aluminum [5]. These studies have been extended to 100 keV helium ion irradiation of SAP 930 held at room temperature, and of SAP 895 and annealed aluminum held at 400°C.

Beryllium, a material with a substantially lower atomic number than aluminum, has been suggested as a material for coatings or structural components in the conceptual design of fusion reactors [8]. Verbeek and Eckstein [9] observed blister formation in beryllium irradiated at room temperature with low energy (15 keV) D^+ and He^+ ions, however, no detailed study of erosion rates or the effect of temperature were done.

In this paper recent studies [10] of 100 keV helium ion irradiation at room temperature and 600°C of vacuum cast Be and sintered beryllium powder which had been hot rolled after sintering (in the following referred to as type II) have been extended to a differently fabricated sintered beryllium powder (in the following referral to as type I).

Experimental Techniques

The SAP 895 used in this study was prepared at the Holifield National Laboratory and contained a nominal 10.5 wt% Al_2O_3 [11]. Thin discs of SAP of ∿ 60 μm thickness were cut from the billet by spark cutting. The surfaces of SAP were optically polished and then cleaned in ultrasonic baths of trichloroelthylene, acetone, distilled water and methanol, in that order. For Rutherford backscattering measurements the discs were thinned by argon-ion sputtering to a thickness of ∿ 5 μm. The polycrystalline aluminum foils used in this study were of high purity (∿ 99.99%). The samples were mechanically polished, degreased in the same four ultrasonic baths used for SAP, annealed for 2 hours at 300°C in a vacuum of 1 - 3 x 10^{-8} Torr, and finally electropolished in a solution containing 617-ml H_3PO_4, 134-ml H_2SO_4, 240-ml H_2O and 156-g CrO_3 at 70°C. For Rutherford backscattering measurements, thin foils of aluminum were prepared first by electropolishing to a thickness of ∿ 20 μm and then, thinning by argon-ion sputtering to a final thickness of ∿ 3 - 4 μm. The SAP 930 used in the present studies contained a nominal 7.0 wt% Al_2O_3 and was obtained from Dr. W. Bauer, Sandia Laboratories, Livermore, California. The surfaces of the sample received had been polished at Sandia Laboratories and received no further treatment except a cleaning in ultrasonic baths of trichlorethylene, acetone, distilled water and methanol.

Examination of polished SAP surfaces at high magnifications with a scanning electron microscope (resolution ~ 100 Å) did not reveal any porosity of the surface. The SAP samples had nearly the theoretical density of aluminum.

Three types of beryllium were used in these experiments. One was a beryllium foil, obtained from Kawecki Berylco Industries, Inc., Hazelton, Pennsylvania, hot-rolled from an ingot that was vacuum-cast. The other sample was a disc cut from an ingot that was prepared by sintering beryllium powder (Type I). A third beryllium sample was a disc cut from an ingot that was prepared by sintering beryllium powder and subsequent hot rolling (Type II). Both type I and type II sintered beryllium powders were obtained from Brush Wellman, Cleveland, Ohio. The targets were first mechanically polished and then electropolished in an electrolyte containing 100 ml phosphoric acid, 30 ml glycerol, 30 ml ethanol, and 30 ml sulphuric acid at an applied voltage of 35 volts. The targets were irradiated with 100 keV $^4He^+$ ions from a 2-MeV Van de Graaff accelerator. During the irradiation the vacuum in the target chamber was maintained at ~ 5×10^{-8} Torr by ion pumping. The ion flux was 1×10^{14} ions cm^{-2} sec^{-1}. The targets irradiated at 400°C and 600°C were heated directly by ohmic heating, and the temperature was measured with an infrared pyrometer. Other details of irradiation can be found elsewhere [2]. After irradiation the targets were examined in a Cambridge Stereoscan S4-10 scanning electron microscope. The three types of beryllium foils and the SAP 895 used in the present experiments were characterized by transmission electron microscopy for grain size and dislocation density. The average grain size was ~ 3 μm in sintered beryllium type I and II, ~ 5 μm in vacuum-cast beryllium, ~ 300 μm in annealed Al, and ~ 0.5 μm in SAP 895 (See Figure 1). The average grain size for SAP 930 has been determined [12] to be ~ 1 - 2 μm.

Results

The significant reduction of helium blistering in SAP 895 as compared to annealed aluminum for identical irradiation conditions (100-keV He^+ ions to a dose of 1.0 C cm^{-2} at room temperature and 400°C) is illustrated in Figure 2. Figure 2 (a) shows an enlarged view of a portion of an aluminum surface irradiated at room temperature. One can see that four exfoliated layers have been removed. Figure 2 (b) shows a portion of an aluminum surface which had been irradiated at 400°C under otherwise identical conditions. One notices the exfoliation of one layer in several areas. Figures 2 (c) and (d) show typical examples of blisters formed on SAP 895 irradiated at room temperature and at 400°C, respectively. In contrast to the aluminum, where large exfoliation is observed, only a few blisters were ruptured in the case of SAP 895. The erosion rates estimated for the room temperature case from the ruptured and lost skins for aluminum and SAP are 1.75 $\pm$ 0.25 and 0.001 atoms per helium ion, respectively, as reported earlier [5]. The results for annealed aluminum held at 400°C give a value of 0.12 $\pm$ 0.05 atoms per helium ion, whereas for the SAP 895 sample no exfoliation of the blisters could be observed.

Figure 3 allows a comparison of the surface erosion of SAP 895 and SAP 930 held at room temperature during irradiation with 100 keV helium ions to a dose of 1 C cm^{-2}. The figure reveals a rather striking difference in the surface erosion of the two types of SAP. The erosion rate of SAP 930 is

as compared to the erosion rate of 0.001 atoms per helium ion for SAP 895.

Figure 4 (a) shows a scanning electron micrograph of vacuum-cast beryllium irradiated at room temperature with 100 keV He^+ -ions to a dose of 1.0 C cm^{-2}. Blisters with diameters ranging from about 5 μm to 35 μm can be

observed. In a few areas the blisters have exfoliated and a second skin has begun to rupture. Figure 4 (b) shows an unirradiated area. Figure 4 (c) shows a micrograph of a sintered beryllium foil, type I, irradiated under the same conditions mentioned above. Only a few blisters can be observed, and the average blister size is smaller than those seen in Figure 4 (a). No exfoliation of blisters could be observed. The pits seen in Figure 4 (c) are not due to blister exfoliation but were present in the unirradiated samples as can be seen in Figure 4 (d). Figure 4 (e) shows a sintered beryllium foil type II irradiated under identical conditions as the other Be foils. Again, only a few blisters without exfoliation can be seen. The blister diameters for both type I and II Be foil range from about 5 to 15 μm. For comparison an unirradiated area of the type II Be foil is shown in Figure 4 (f). One notices that pits observed in the irradiated area [Figure 4 (e)] are also present in the unirradiated area.

Figure 5 (a) shows a micrograph of a vacuum-cast beryllium foil irradiated at 600°C with 100 keV He^+ -ions to a dose of 0.5 C cm^{-2}, which is half the dose used for the room temperature irradiation [see Figure 4 (a)]. Very serious blister exfoliation can be observed. In some areas as many as seven skin layers have exfoliated and have been lost. In contrast the sintered beryllium foils of type I and II show greatly reduced blister exfoliation [see Figure 5 (b) and (c)]. Only in a few areas blister skins have been lost. The average blister size is smaller in the sintered beryllium foils as compared to the vacuum-cast beryllium. In comparing Figures 5 (b) and (c) one should note that the dose used for the Be type II is twice the dose used for Be type I.

From measurements of the blister skin thickness, and the area from which the skins were lost an estimate of the erosion yields has been made. Table 1 summarizes the results of such yields for the three types of beryllium

foils, for the two types of SAP samples, and for annealed aluminum for different temperatures. For both types of sintered beryllium foils one notices that the erosion yields are lower than for the vacuum cast beryllium at room temperature (yields not measurable for Be type I and II) and at 600°C. For the beryllium foils one notices an increase in the erosion rates as the temperature is increased from room temperature to 600°C. This is the same trend observed earlier [3, 6, 13] for such metals as V, Nb, and stainless steel. In contrast aluminum shows a decrease in the erosion rate at 400°C as compared to room temperature. We have observed earlier that the blister exfoliation and the erosion rates are at a maximum at a temperature which is high enough so that the surface can be deformed easily (reduced yield strength, and increased gas kinetic pressure in the bubble) but low enough so that helium release from the surface is still very small. For aluminum, which has a much lower melting point than metals such as beryllium, vanadium and niobium, this maximum lies at temperatures below those for vanadium for example ($\sim$ 500°C) [3].

Another striking result is that both types of sintered beryllium show significantly less erosion than the vacuum-cast beryllium for both temperatures. At 600°C the erosion yields for sintered beryllium foils type I and II are reduced by more than one order of magnitude in comparison with the vacuum-cast beryllium. It is of interest to note that the erosion yields at room temperature for all three types of beryllium are lower by more than three orders of magnitude than for annealed aluminum. At room temperature the erosion yield for sintered beryllium type I and II is even lower (nondetectable) than for sintered aluminum powder.

Discussion

The drastic reduction in erosion yield in sintered aluminum powder as compared to annealed aluminum was attributed [5, 14] to the dispersion of trapped helium at the large Al-Al_2O_3 interfaces at the large grain boundaries in SAP as determined by Rutherford backscattering. (The average grain size in SAP 895 was $\sim$ 0.5 μm as compared to $\sim$ 300 μm for annealed aluminum.) Furthermore, the fact that the yield strength of SAP 895, for example, is much higher ($\sim$ 35,6000 psi) than for annealed aluminum ($\sim$ 1,700 psi) will help to reduce the blister rupture and exfoliation in SAP from that observed in aluminum. The higher erosion yield in SAP 930 as compared to SAP 895 may in part be attributed to the larger average grain size ($\sim$ 1 - 2 μm) and a lower yield strength ($\sim$ 16,000 - 25,000 psi). The observed reduction in erosion yields in sintered beryllium in comparison to the vacuum-cast beryllium is not clearly understood at the present, since the difference in the average grain size between these two types of beryllium is not very large as mentioned earlier.

Acknowledgements

We are grateful to Dr. Victor Maroni, ANL, for providing us with some of the vacuum cast beryllium. We also like to thank Mr. Marvin Kral for target preparation and Mr. Peter Dusza for his help in the target irradiations.

References

†Work performed under the auspices of the Energy Research and Development Administration.

[1] M. Kaminsky, IEEE Trans. Nucl. Sci. NS18, (1971) 208.

[2] S. K. Das and M. Kaminsky, J. Appl. Phys. 44, (1973) 25.

[3] S. K. Das and M. Kaminsky, J. Nucl. Mat. 53, (1974) 115.

[4] M. Kaminsky, in"Plasma Physics and Controlled Nuclear Fusion Research", Vol. II (International Atomic Energy Agency, Vienna 1975) p. 287.

[5] S. K. Das, M. Kaminsky and T. D. Rossing, Appl. Phys. Lett. 27, (1975) 197.

[6] M. Kaminsky and S. K. Das, Nucl. Tech. 22, (1974) 373.

[7] W. Bauer and G. Thomas, J. Nucl. Mat. 53, (1974) 127 and also 53, (1974) 134.

[8] W. Stacey, Jr. et. al, Tokamak Experimental Power Reactor Studies, ANL/CTR-75-2, Argonne National Laboratory (June, 1975).

[9] H. Verbeek and W. Eckstein, in Application of Ion Beams to Materials, edited by S. T. Picraux, E. P. EerNisse, and F. L. Vook (Plenum Press, New York, 1974) p. 597.

[10] S. K. Das and M. Kaminsky, to appear in the proceedings of the Sixth Symposium on Engineering Problems of Fusion Research, November 18-21, 1975, San Diego, California (IEEE, New York).

[11] G. L. Copeland, M. M. Martin, D. G. Harman, and W. R. Martin, Oak Ridge National Laboratory Report No. ORNL-TM-2215 (1968).

[12] W. Bauer, (private communication).

[13] S. K. Das and M. Kaminsky, Proceedings of Symposium on Engineering Problems of Fusion Research, IEEE Publication No. 73CH0843-3-NPS (Institute of Electrical and Electronics Engineers, New York, New York 1974) p. 31.

[14] S. K. Das and M. Kaminsky, paper presented at II International Conference on Ion Beam Surface Layer Analysis, Karlsruhe, West Germany, September 15-19, 1975 (to appear in the Proceedings, Plenum Press, New York).

TABLE 1. EROSION RATES FOR BE AND AL IRRADIATED WITH 100 KEV $^4He^+$ IONS.

TYPE OF MATERIAL	EROSION YIELDS (SR) IN ATOMS/INCIDENT ION	
	ROOM TEMP. (DOSE 1.0 C/CM^2)	400°C (DOSE 1.0 C/CM^2)
ANNEALED AL	1.75 ± 0.25	0.12 ± 0.05
SINTERED ALUMINUM POWDER (SAP 895)	0.001	BLISTERS NO EXFOLIATION
SINTERED ALUMINUM POWDER (SAP 930)	~ 0.01	-
		600° (DOSE 0.5 C/CM^2)
VACUUM CAST BE	~ 0.001	0.3 ± 0.1
SINTERED BE TYPE I	BLISTERS NO EXFOLIATION	0.02 ± 0.01 (DOSE 1.0 C/CM^2)
SINTERED BE TYPE II	BLISTERS NO EXFOLIATION	0.02 ± 0.01

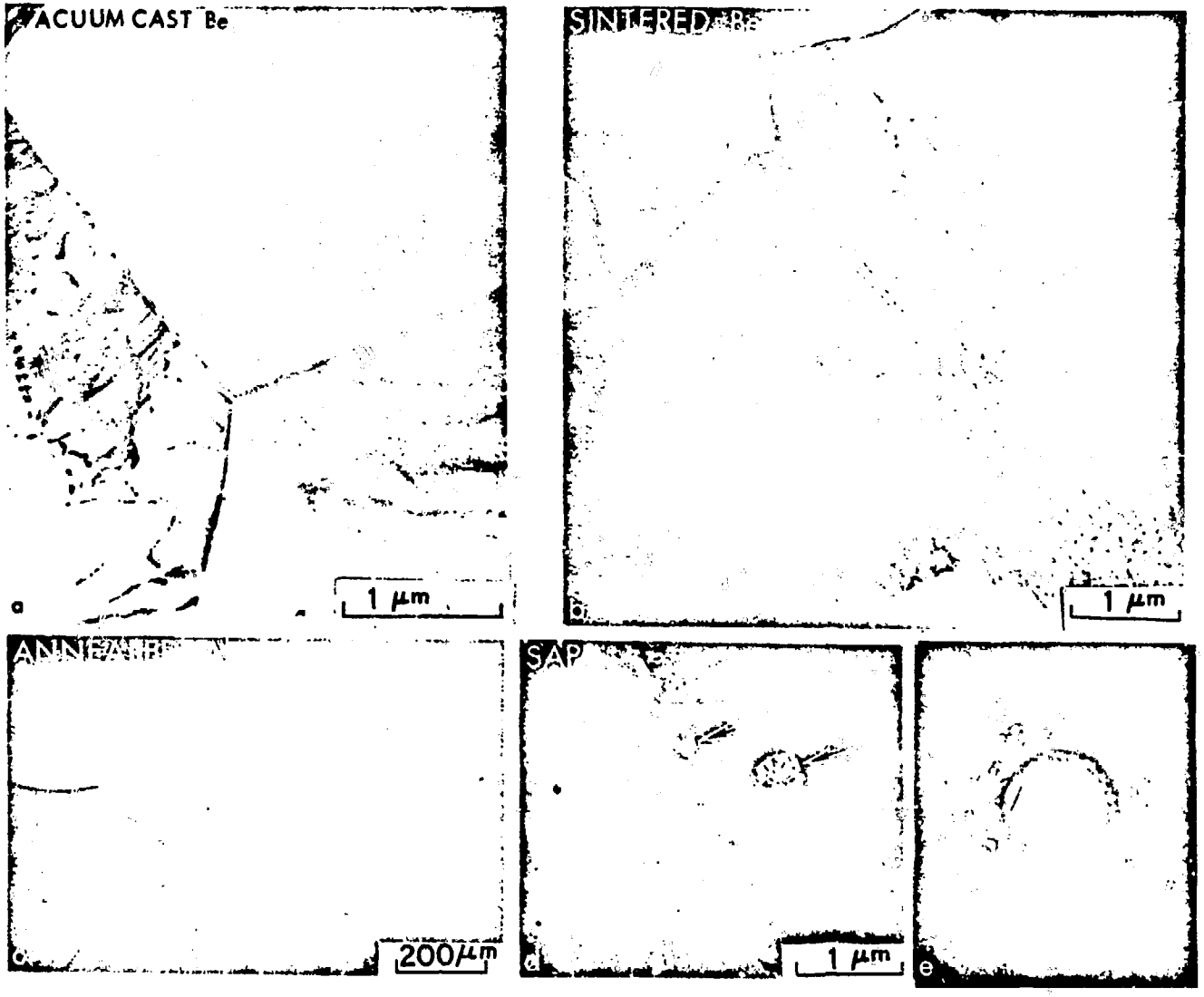

VACUUM CAST Be
1 μm
a
SINTERED Be
1 μm
b
ANNEALED
200μm
c
SAP
1 μm
d
e

Fig. 1

POLYCRYSTALLINE Al and SAP IRRADIATED WITH 100keV $^4He^+$ TO A DOSE OF 1.0 C/cm^2

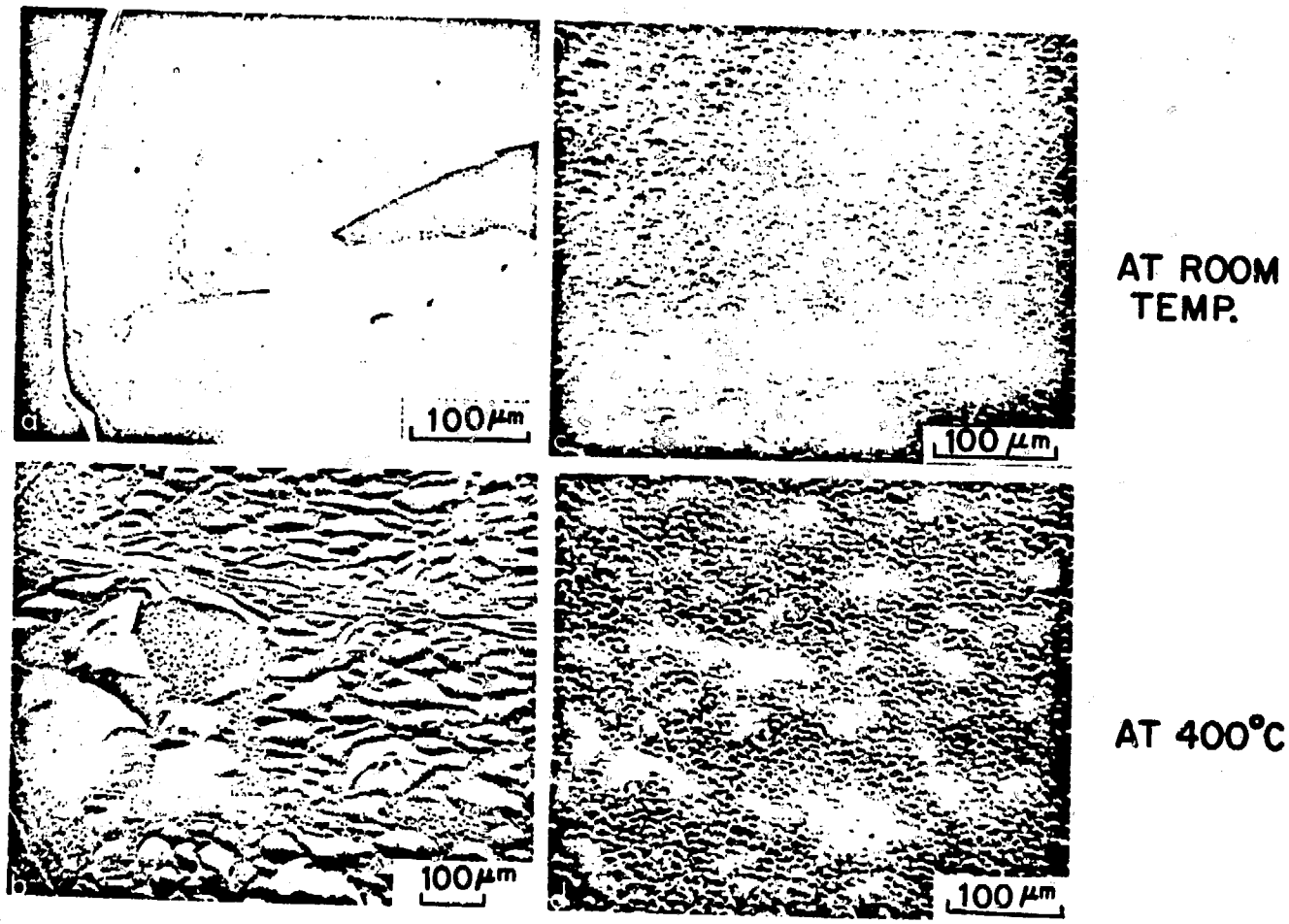
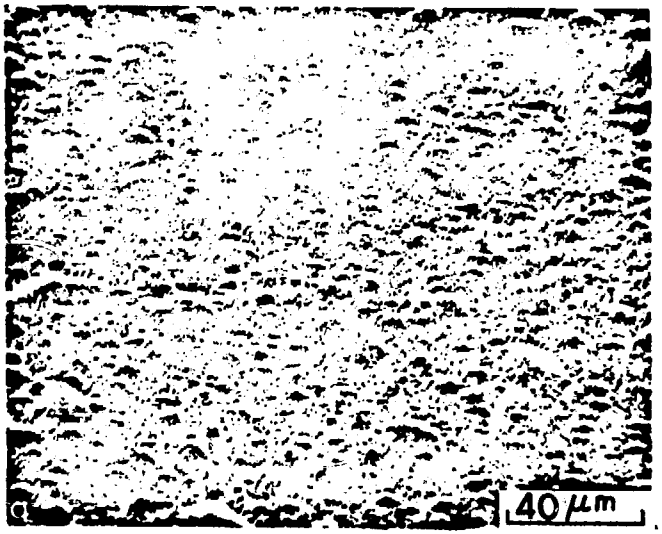


Fig. 2
Das and Kaminsky

SINTERED ALUMINUM POWDER (SAP) IRRADIATED WITH 100keV He^{+} TO A DOSE OF 1.0C/cm^2 AT ROOM TEMPERATURE

SAP 895



SAP 930

Fig. 3
Dan and Kaminsky

BERYLLIUM IRRADIATED AT ROOM TEMPERATURE WITH 100keV $^4He^+$ IONS TO A DOSE OF 1.0 C/cm^2

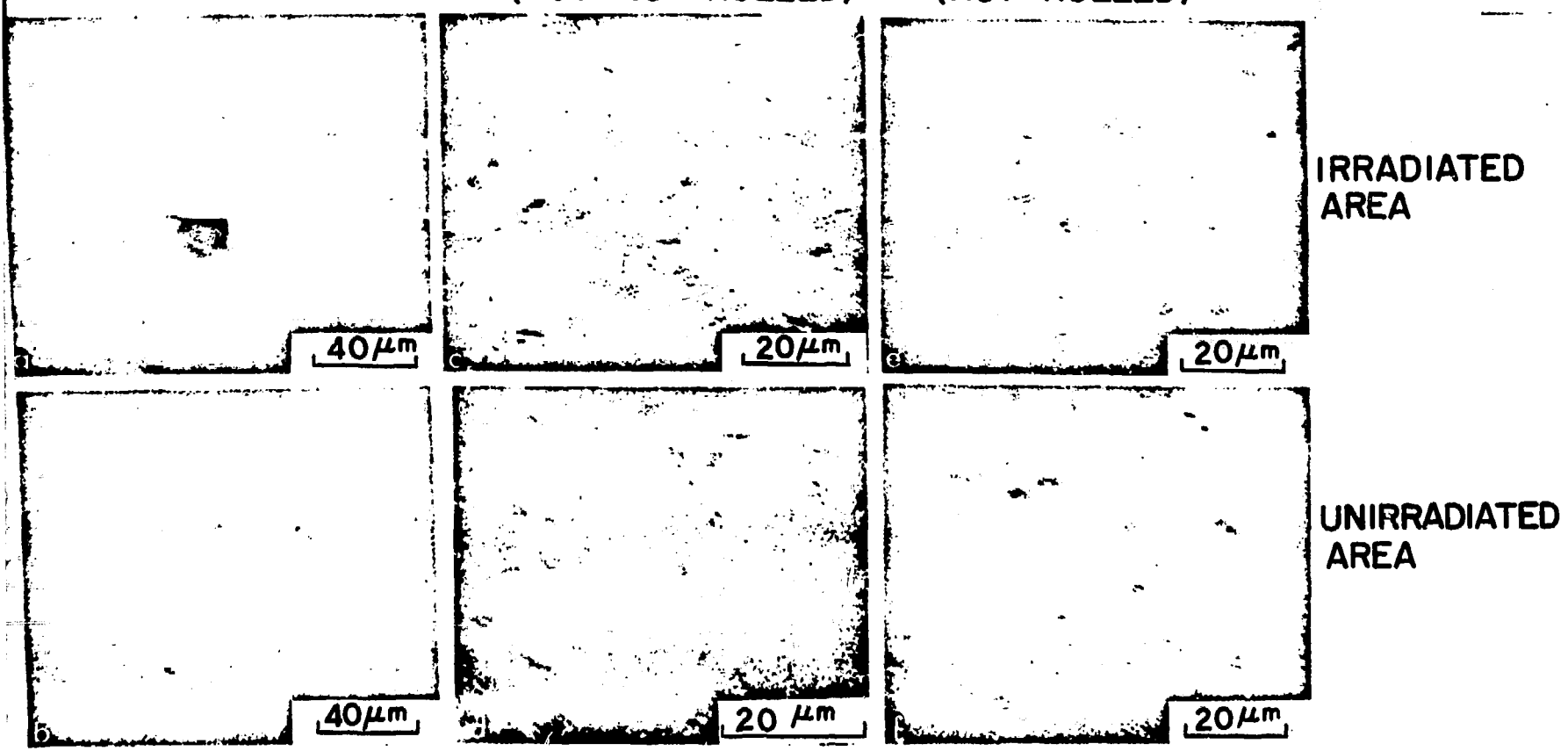


Fig. 4
Dau and Kaminsky
2-19-16-68

BERYLLIUM IRRADIATED AT 600°C WITH 100 keV $^4He^+$ TO DOSES INDICATED

/ACUUM CAST Be

0.5 C/cm^2)

SINTERED Be TYPE I
(NOT HOT ROLLED)
(0.5 C/cm^2)

SINTERED Be TYPE II
(HOT ROLLED)
(1.0 C/cm^2)

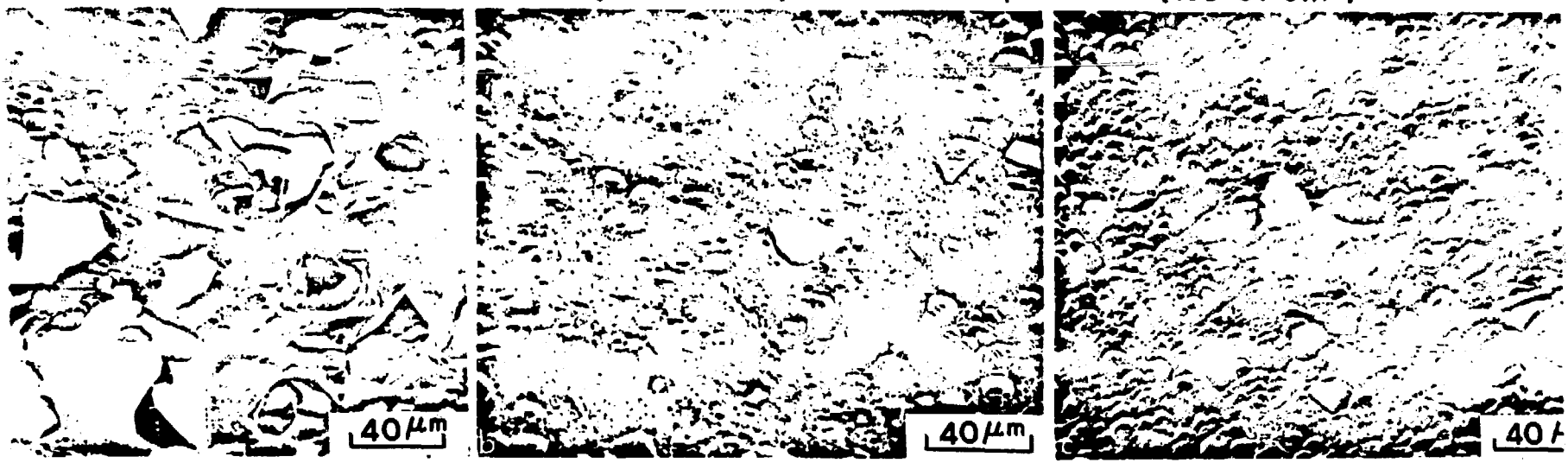


Fig. 5.
Das and Kaminsky
209-76-72