Reprinted from NUCLEAR SCIENCE AND ENGINEERING, Vol. 2, No. 4, July 1957
Academic Press Inc. *Printed in U.S.A.*

NUCLEAR SCIENCE AND ENGINEERING: **2,** 513–515 (1957)

Radiation-Induced Hardness Changes in Graphite[1]

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Received August 28, 1956

Changes in Knoop Hardness of artificial graphite from the range 9 to 12 to the range 30 to 75 are found to accompany fast neutron irradiation at 30°C.

The irradiation of graphite is accompanied by large changes in its electrical, thermal, and mechanical properties (*1*). The magnitude of most of these has been measured quantitatively, and in many cases has been at least qualitatively correlated with a microscopic physical model of radiation damage (*2*, *3*). To the writers' knowledge, no quantitative reports of the large changes in hardness which accompany irradiation have been made, although these are well known to laboratory mechanics who have prepared experimental samples of irradiated graphite. Quantitative measurements of graphite hardness have been made on a microscopic scale with a Tukon Microhardness Tester. This machine was chosen in order to permit hardness readings to be taken over an area comprised of a small number of grains.

Initially, microhardness measurements were made upon large crystallite graphite obtained by the cooling of a supersaturated solution of carbon in molten nickel to room temperature. The nickel was etched away leaving graphite particles of large size. Samples were mounted by "floating" the platelets on a redissolved surface of acrylic mounting material.[2] Microhardness readings obtained upon these samples were erratic due to the fact that graphite platelets within the grains were not parallel to the mount surface.

Better reproducibility was obtained with finer grained graphite. Extruded artificial graphite (AGOT-KC) was sectioned, mounted, and polished. Similar samples of neutron-irradiated AGOT-KC graphite were also prepared. As certain property changes induced by irradiation in graphite are known to an-

[1] This work was performed under the auspices of the U. S. Atomic Energy Commission.

[2] Material used was Acrylic Denture Powder mixed with #7 Fast Repair Liquid manufactured by Wm. Getz Corporation, Chicago, Illinois.

neal out at temperatures near 100°C, the curing temperature of the mounting material was kept below 100°C. The Knoop Hardness Numbers of the specimens measured are shown in Table I. There were 16 to 30 hardness readings taken on each specimen. One-half of the number of readings on each specimen were taken with the indentor moving in a direction parallel to that on which the graphite had been extruded, the other half, in a direction perpendicular to the extrusion axis.

From the table it may be seen that the irradiated specimens have a larger spread between high and low values than the unirradiated specimens at a low (50 g) indentor load. This is a result of the exponential curve relating Knoop Hardness to the length of the indentations on each of the various specimens. The latter varied only by approximately 1 Filar unit under a 10.25 lens. To simulate the reproducibility of macrohardness readings the Tukon Tester was also used with 100 and 300 g loads. As seen by the Knoop Hardness Numbers under the appropriate headings in the table the spread between high and low values is less than with a 50 g load.

The data in the table show a definite increase in hardness in irradiated graphite specimens, due presumably to irradiation. However, the small difference in hardness between specimens irradiated to 5×10^{19} *nvt* and to 1.5×10^{20} *nvt* (146 Mwd/*T* and 460 Mwd/*T*, respectively, in units of reference *1*) suggests strongly that radiation effects on graphite hardness have saturated at some lower irradiation level. During earlier work, in which thermal setting mounting plastics were used, it was observed that most of the hardness change had an-

TABLE I

KNOOP HARDNESS NUMBERS OF GRAPHITE SAMPLES

		Measured parallel to extrusion axis			Measured perpendicular to extrusion axis		
		50 g	100 g	300 g	50 g	100 g	300 g
Unirradiated (KC)	Average	11	12	12	11	10	12
	High	13	15	14	13	14	15
	Low	9	10	11	9	8	9
Irradiated 5×10^{19} nvt (fast) at 30°C	Average	40	41	43	37	34	38
(Kc Sample 207)	High	66	72	58	53	45	40
	Low	31	19	38	29	23	25
Irradiated 1.5×10^{20} nvt (fast) at 30°C	Average	50	47	58	71	68	44
(KC Sample 202)	High	81	65	67	81	81	58
	Low	37	33	49	37	48	35
Irradiated 1.5×10^{20} nvt (fast) at 30°C	Average	44	42	47	40	43	49
(KC Sample 293)	High	77	59	74	71	72	70
	Low	29	35	37	29	29	40

nealed out during a one-hour 100°C oven treatment. While the detailed mechanism relating radiation induced hardness increases to microscopic changes in the graphite remains to be propounded, the observed trends are similar to those reported for other properties of graphite after neutron irradiation.

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