35

technical note

International Journal of Applied Radiation & Isotopes, Vol. 31, pp. 649 to 651

0020-708X/80/1001-0649$02.00/0

Intercalibration of Glass Dosimeters for Neutron Fluence Determination

HARDEV SINGH VIRK

Department of Physics, Guru Nanak Dev University, Amritsar-143005, India

(*Received* 22 *January* 1980; *in revised form* 26 *February* 1980)

Fission track technique can be used for neutron fluence measurement from a reactor using a glass dosimeter. Calibration constants of various glass dosimeters are determined using Fleischer's 0.4 ppm glass as reference standard. The calibration constant for our laboratory glass slide is $\sim 1.1 \times 10^{11}$ n/track.

1. Introduction

GLASS dosimeters doped with known concentrations of uranium are being widely used in various laboratories all over the world for neutron fluence measurement in nuclear reactors. The glass standards being used are mostly supplied by Corning Museum of Glass, Corning, New York; General Electric Company, Schenectady, New York and National Bureau of Standards, Washington, DC, U.S.A.

FLEISCHER *et al.*[1] were the first to standardize a microscope glass slide for neutron flux density determination of a reactor by using both fission track counts and radiochemical techniques. The U content of this glass is 0.4 ppm and the thermal neutron fluence is calculated by using the relation.[1]

$$\phi = k\rho \tag{1}$$

where ρ is the number of tracks cm^{-2} counted over the internal surface of glass dosimeter and k, the calibration constant, which is estimated to be 2.26×10^{11} neutron/track.

SCHREURS *et al.*[2] made use of Corning glass with 41 ppm of U content as the reference standard (U_1) and prepared a number of sub-reference standards (U_2–U_7) by using fission track calibration. The value of k, the calibration constant for U_1 glass is $\sim 4 \times 10^9$ n/track.

CARPENTER and REIMER[3] of National Bureau of Standards have done an excellent work in preparing fission track glass dosimeter SRM's 961, 962, 963 and 964. These are available in the form of wafers at four different U concentrations of 461.5 ppm, 37.88 ppm, 0.823 ppm and 0.0721 ppm, respectively. They are prepared from the same lot of material as SRM's 610 through 616. In preparation of these dosimeters, the uranium used for the dopant is depleted uranium but they are certified for more than 60 trace elements and at present considered to be the best standards available.

The purpose of this study is twofold—to calibrate a glass dosimeter for our laboratory and to prepare an intercalibration chart for various glass dosimeters being used in neutron fluence measurements taking Fleischer's glass (0.4 ppm) as a reference standard.

2. Experimental

Sample preparation and irradiation

Glass samples were prepared from a series of glasses, i.e. Fleischer's GEC glass standards with 0.4 ppm and 20 ppm U content, NBS standards SRM's 612, 614 and 962, and a laboratory soda lime microscope glass slide to be calibrated. All the samples were washed with alcohol and wrapped in polythene jackets before packing into Al irradiation capsules. The irradiations were carried out in the thermal column of 40 MW CIRUS reactor at BARC Trombay, Bombay at a conventional neutron fluence of 10^{15} (nvt) using IC-1 self serve position. The neutron flux density in this position is $\sim 5 \times 10^{12}\, n\, cm^{-2}\, s^{-1}$ and is predominantly thermalized.

Dose measurement

Irradiated glass samples were fractured, ground with cerium oxide and polished with alumina powder. Fleischer's glass (0.4 ppm) was etched in 40% HF at 30°C for 30 s. The track counting was done on internal surface for 4π geometry under total magnification of 300 × using Olympus microscope. The neutron fluence calculated by using equation (1) is 0.994×10^{15} (nvt) which is amazingly close to the nominal value of 10^{15} (nvt).

The above procedure was repeated for SRM 962 and other glass specimen. Thermal neutron fluence was calculated for SRM 962 by comparison of its induced track density with that of irradiated wafer supplied by NBS using the mean value of neutron flux density for Cu and Au foil irradiations in RT-3 position.[3] The irradiation fluence is estimated to be 1.046×10^{15} (nvt). Thus the irradiation fluences calculated by using Fleischer's glass standard and NBS-SRM 962 for CIRUS reactor differ by 5% only. The counting statistical errors are nearly the same in two cases.

The data for all the irradiated glass samples is summarized in Table 1.

Etching efficiency

The most troublesome parameter in neutron glass dosimetry is etching efficiency factor E.[4,5] Glasses are easily etched by HF acid. The etching efficiency depends upon concentration of the etchant, its temperature and time of etching. An optimum combination of these parameters has to be found by a hit and miss method for proper revelation of tracks.

In our study Fleischer's reference glass standard was repeatedly etched in 40% HF at 30°C for varying intervals of time starting from 5 s. Induced fission tracks were counted after each etching. It was observed that the track density attains a plateau value for an etching interval of 30 s.

This procedure was repeated for other glass dosimeters and it was observed that optimum etching conditions depend upon the chemical composition of the material. The numerical value of E for optimum etching conditions approaches unity.

Geometry factor

A correction known as geometry factor (G)[6,7] has to be applied in case of track measurements carried over external surfaces of glass dosimeters, i.e. under conditions of 2π geometry. This factor varies with the composition of the material and hence with its etching parameters. Under optimum etching conditions the glasses were etched and the tracks were counted over external and internal surfaces alternatively before and after polishing. The external (2π

TABLE 1. Calibration constants for glass dosimeters

S. No.	Glass dosimeter	Track density $\rho \times 10^{-2}$ cm^{-2}	U conc. (ppm)	Calibration constant k $\times 10^{-10}$ (n/track)	Geometry factor G
1.	Fleischer's Reference Standard	44.00 (780)*	0.40	22.6	0.82
2.	GEC† Glass	2140 (795)	19.50	0.46 ± 0.3	
3.	‡SRM 962	1248 (935)	34.42	0.79 ± 0.4	0.82
4.	SRM 612	1296 (891)	35.74	0.76 ± 0.04	
5.	SRM 614	31.18 (256)	0.74	31.8 ± 2.3	
6.	Glass Slide	90.90 (935)	0.83	10.9 ± 0.5	0.60

Total thermal neutron fluence (nominal value) = 10^{15} (nvt).
Total thermal neutron fluence (monitored value) = 0.99×10^{15} (nvt).
Total thermal neutron fluence (SRM 962 value) = 1.05×10^{15} (nvt).
* Brackets show number of tracks counted.
† General Electric Company glass (20 ppm).
‡ Standard Reference Material supplied by National Bureau of Standards.

geometry)/internal (4π geometry) ratios observed for Fleischer's reference glass, SRM 962 and microscope glass slide are recorded in Table 1.

3. Intercalibration

Calibration constant

It is important to realize that calibration constant k must be determined precisely for each glass standard for calculation of conventional reactor fluence and intercomparison of results between the various laboratories engaged in radiation dosimetry. However, this constant depends upon a large number of factors, e.g. composition of glassy material, etching parameters, annealing conditions, reactor irradiation position and on the subjective criterion used by each individual observer as to what he will call a track. Thus k must be determined by each laboratory and cannot be specified precisely.

Using Fleischer's glass (0.4 ppm, $k = 2.26 \times 10^{11}$ n/track) as reference standard the values of calibration constants for all other glasses are calculated using relation (1) with the conventional value of thermal neutron fluence of $0.99 \pm 0.04 \times 10^{15}$ (nvt). Statistical errors in the values of k (Table 1) are calculated on the basis of 1σ error in track counts.

Uranium contents

Glass dosimeters have proved to be a useful tool for U estimation studies in materials.[8–10] Uranium contents of the glass standards are estimated by comparison with the Fleischer's glass using the following relation:[2]

$$Wux = Wus \times \frac{Is}{Ix} \times \frac{\rho x}{\rho s} \times \frac{Rs}{Rx} \qquad (2)$$

where

Wu = weight fraction of uranium
I = isotopic ratio of $^{235}U/^{238}U$,
ρ = induced density of tracks cm^{-2},
R = average dia. of tracks in μm,

and s and x denote standard and unknown.

The U contents are summarized in Table 1. As the track diameters in glass are very sensitive to etching and annealing effects it is pertinent to carry out all measurements under identical conditions of temperature and etching. The isotopic ratios should also be known accurately.

4. Discussion and Conclusions

The values of calibration constant k are determined from an empirical relation (equation 1). The accuracy of our method depends upon the reliability with which thermal neutron fluence is measured by Fleischer's glass standard. From the present analysis, as well as from past experience, the conventional neutron fluence determined by this dosimeter never differed by more than 5% from the nominal or monitored value of fluence at CIRCUS reactor of BARC, Trombay.

CARPENTER and REIMER[3] used both Au and Cu fo[illegible] monitors in calibrating the neutron fluence received b[illegible] SRM 962 in the NBS reactor. It is observed that the neutron fluence determined by SRM 962 is in good agreement with the nominal value if mean value of neutron flux density (5.61×10^{13} n cm^{-2} s^{-1}) monitored by Au and Cu foils is used in calculations. Thus the irradiation position in a reactor is of great significance in calibration studies.

HURFORD and GLEADOW[11] have recently calibrated U_3 and SRM 612 glasses for interlaboratory use. They determine a mean value of $k = 6.62 \times 10^9$ n/track for SRM 612 which is lower than the value reported in Table 1. However, the values of k for SRM's 962 and 612 are in good agreement with each other as both the glasses are identical in nature.[3]

The values of G for Fleischer's glass standard and NBS–SRM 962 are concordant and agree with the value determined by REIMER *et al.*[6] for microscope slide glass. The value of G for our microscope slide is quite low in comparison.

U contents of glasses are calculated by assuming the U dopant in Fleischer's glass to be of standard isotopic ratio. U dopants for SRM's 962 and 612 are depleted to 0.239% ^{235}U and for SRM 614 to 0.279% ^{235}U. The values obtained in Table 1 are not concordant with the certified values given by NBS.[3]

The laboratory microscope glass slide is found to contain 0.83 ppm of uranium and a uniform distribution of induced fission tracks. It can serve as a cheap and reliable glass dosimeter for routine neutron flux density measurements in a reactor at high neutron fluences with a calibration constant of 1.1×10^{11} n/track.

Acknowledgements—The author is thankful to Drs Robert L. Fleischer and Robert McCorkell for the supply of glass dosimeters. He is particularly indebted to the latter for helpful discussions which made this work possible. Service irradiation by Isotope Division, BARC, Trombay and technical assistance of Mr T. M. Singh in preparation of samples are acknowledged.

References

1. Fleischer R. L., Price P. B. and Walker R. M. *J. nucl. Sci. Engng* **22,** 153 (1965).
2. Schreurs J. W. H., Friedman A. M., Rokop D. J., Hair M. W. and Walker R. M. *Radiat. Effects* **7,** 231 (1971).
3. Carpenter B. S. and Reimer G. M. Calibrated glass standards for fission track use. NBS special Publication 260–49, p. 27 (1974).
4. Kleeman J. D. and Lovering J. F. *Radiat. Effects* **5,** 233 (1970).
5. Khan H. A. and Durrani S. A. *Radiat. Effects* **13,** 257 (1972).
6. Reimer G. M., Storzer D. and Wagner G. A. *Earth planet. Sci. Lett.* **9,** 401 (1970).
7. Gleadow A. J. W. and Lovering J. F. *Nucl. Track Det.* **1,** 99 (1977).
8. Fisher D. E. *Analyt. Chem.* **42,** 414 (1970).
9. Virk H. S. and Koul S. L. *C.r. hebd. Séanc. Acad. Sci. Paris* **284,** 295 (1977).
10. Virk H. S. and Kaur H. *Curr. Sci.* **48,** 293 (1979).
11. Hurford A. J. and Gleadow A. J. W. *Nucl, Track Det.* **1,** 41 (1977).