



## THE THERMAL STABILITY OF FISSION TRACKS IN THE 612 NIST GLASS STANDARD AT ROOM TEMPERATURE

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(Received 26 September 1994)

**Abstract**—Two wafers of the NIST (formerly NBS) glass standard SRM 612 recently irradiated have been compared to the pre-irradiated wafers RT3 and RT4 of glass SRM 962a, stored for 9 years at 5°C, and SRM 962, stored for 20 years at room temperature. Track area densities on internal surfaces of the glass as well as track size measurements suggest that (1) the old SRM 962 and the more recent SRM 962a calibrations are consistent and (2) annealing of the fission tracks in the pre-irradiated wafers is negligible. This last experimental result enables a direct comparison of contemporary and previous fission track age calibrations.

### INTRODUCTION

Twenty years ago Reimer *et al.* (1972) studied the thermal stability of fission tracks in the National Institute of Science and Technology (NIST—formerly NBS) glass standards for fission track use. They concluded that the fission tracks in these glasses had a relatively low thermal stability, and recommended storage of irradiated wafers at temperatures <20°C to prevent partial track annealing. Moreover, they suggested checking possible annealing effects by track size measurements. These standards, especially SRM 962 (Carpenter and Reimer, 1974) and, more recently, SRM 962a (Carpenter, 1984) have been used for several fission track groups as neutron flux standards for fission track dating.

SRM 962 and SRM 962a are made from the same glass, SRM 612. Each packet includes two reference glasses irradiated in two positions, RT3 and RT4, at the NIST reactor. Following the NBS certificate, the neutron flux in a given irradiation facility can be obtained by comparison of track densities in internal polished surfaces of a glass irradiated in that facility and of the pre-irradiated wafers. (The more recent SRM 962a is supplied with a muscovite and a polycarbonate external detector attached to the glass during irradiation; thus, the neutron flux can be obtained by means of the external detectors.) The NIST certificate recommends polishing and etching (at the same time) for the glasses to be compared, for reproducibility of experimental conditions. Obviously, if the tracks in the pre-irradiated glasses are affected by a certain annealing amount,

the NIST standards will over-estimate the neutron flux.

To compare age determination made in the past using the previous SRM 962 standard with more recent measurements, as well as to check whether some annealing effect was detectable in the reference glasses stored for a long time, track densities and track sizes were measured in two recently irradiated wafers and in four reference wafers irradiated in the RT3 and RT4 facilities about twenty years ago (SRM 962) and about nine years ago (SRM 962a), respectively.

### EXPERIMENTAL

Two SRM 612 wafers were irradiated in the Lazy Susan (cadmium ratio 6.5 for Au and 48 for cobalt) facility of the LENA Triga Mark II reactor (University of Pavia, Italy). After irradiation, they were mounted, polished and etched for 30 min in 20% HF at 40°C together with the SRM 962 and SRM 962a, RT3 and RT4 pre-irradiated wafers. The track densities were determined under a Jena Jenaval microscope. Track size (the major axis of the etch pits) measurements were performed with a micrometric eyepiece and with Microvid equipment coupled to a Leitz Orthoplan microscope. The experimental results are shown in Table 1. The mean track sizes measured using the Microvid equipment are systematically lower (by 1–2%) than those determined using the eyepiece. This is due to calibration problems, as different microscopes and magnifications (1000× and 750×, respectively) were used.

Table 1. Track densities and track sizes in the SRM 962 and SRM 962a glass standards

Wafer (certified fluence. Cu, Au reference) $\times 10^{14}$	Track density $\text{cm}^{-2} (\pm 1\sigma)^*$	Tracks counted	$s'$	Mean track size $\mu\text{m}$	$(\pm 1\sigma)$
Cu 4.232	(24,500)†				
962-RT3	$107,400 \pm 2100$	2488	2.1	$7.63 \pm 0.06\ddagger$ (120§)	$7.54 \pm 0.04\parallel$ (304)
Au 4.744	(22,660)				
Cu 1.548	(26,400)				
962-RT4	$40,900 \pm 1000$	1579	2.5	$7.68 \pm 0.07\ddagger$ (120)	$7.58 \pm 0.05\parallel$ (314)
Au 1.716	(23,800)				
Cu 4.37	(25,400)				
962a-RT3	$111,000 \pm 2400$	2115	1.9	$7.71 \pm 0.08\ddagger$ (100)	$7.55 \pm 0.04\parallel$ (300)
Au 4.75	(23,700)				
Cu 3.87	(28,300)				
962a-RT4	$109,600 \pm 2400$	2143	1.8	$7.71 \pm 0.08\ddagger$ (100)	$7.56 \pm 0.05\parallel$ (329)
Au 4.17	(26,300)				
P 26	$792,900 \pm 13,700$	2449	2.0	$7.56 \pm 0.06\ddagger$ (100)	$7.53 \pm 0.04\parallel$ (336)
P 30	$700,700 \pm 16,000$	2597	2.3	$7.69 \pm 0.04\ddagger$ (100)	$7.49 \pm 0.04\parallel$ (335)

\*Track area density for a fluence of  $10^{14} \text{ cm}^{-2}$ ; †computed using Poisson statistics (by a chi-square test, the resultant track distributions were consistent with Poisson distributions);  $s'$ : standard error of the mean of the track counts:  $\pm 0.06$ : standard error of the mean; §number of tracks measured for mean size determination; ‡, ||measured using a Leitz  $16 \times$  micrometric eyepiece and using Leitz Microvid equipment, respectively.

## DISCUSSION

Comparison of track densities shown in Table 1 with certified fluence values suggest that the ratio track density/neutron fluence is systematically lower for the SRM 962 wafers, especially for the RT4 irradiation facility ( $\sim 7\%$  for Cu calibration and  $\sim 10\%$  for Au calibration). Obviously, the neutron fluences computed for irradiations P26 and P30 by the track densities of Table 1 and by the various certified fluences, show the same systematic differences mentioned above, when the SRM 962 or the SRM 962a standards are used as the reference. Consequently, the ages that may be computed in addition to the well-known differences due to inconsistent Cu and Au calibrations ( $\sim 10\%$  for SRM 962 and  $\sim 8\%$  for SRM 962a), show systematic variations correlated with the different standards. Systematic differences of fluence values (and ages) could be interpreted as a consequence of a certain annealing rate of tracks in the reference wafers, but this is not true in the present case, for two reasons: (1) the fluence variations depend upon the irradiation facility and the metal foil used for calibration (negligible for the RT3 position, Cu calibration, it reaches  $\sim 10\%$  for the RT4 position, Au calibration); and (2) the mean track sizes (Table 1) do not show significant variations.

However, when considering the experimental errors of track densities and the certified uncertainties of neutron fluences, only the  $\sim 10\%$  difference of the RT4 wafers (Au calibration) seems to be significant.

## CONCLUSION

Track density and mean track size measurements, on the SRM 962 and SRM 962a NIST glass

standards, irradiated 20 and 9 years ago, respectively, support the following conclusions: (1) the old and the more recent standards are substantially coherent: the results shown in Table 1 suggest a significant systematic difference for the Au calibration, RT4 facility only; and (2) the fission tracks are unaffected by partial annealing (note that, whereas the SRM 962a reference wafers were stored at  $\sim 5^\circ\text{C}$ , those of SRM 962 were conserved at room temperature, normally  $> 20^\circ\text{C}$ , up to  $30^\circ\text{C}$  during summer time).

The absence of annealing in the reference wafers enables direct comparison of old fluence and age determinations with more recent measurements. Whatever the calibration at present adopted by a fission track group, by using the new calibration and the previous standard in the same irradiation the possible systematic differences can be estimated.

Although we do not confirm with our results the indication of possible annealing effects that was deduced by laboratory experiments made 20 years ago, we do not state that the recommendation by Reimer *et al.* (1972) should be ignored: storing the reference glasses at low temperatures remains prudent good practice that we will continue.

## REFERENCES

- Carpenter B. S. (1984) *Standard Reference Material: Calibrated Glass Standards for Fission Track Use*. NBS Spec. Publ. 260-92.
- Carpenter B. S. and Reimer G. M. (1974) *Standard Reference Material: Calibrated Glass Standards for Fission Track Use*. NBS Spec. Publ. 260-49.
- Reimer G. M., Wagner G. A. and Carpenter B. S. (1972) The thermal stability of fission tracks in the standard reference material glass standard (National Bureau of Standards). *Radiat. Effects* **15**, 273-274.