LETTERS TO THE EDITOR

MODIFICATIONS TO SCRIBNER MULLINS SHALLOW CUP (SMSC) ELECTRODE FOR RAPID VOLATILISATION

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SCRIBNER Mullins shallow cup (SMSC) electrode, such as L 4030 of National mark from Union Carbide Corporation (Chicago, USA) or 1991 from Ultra Carbon Corporation (Michigan, USA), is useful for rapid volatilisation in emission spectrochemical analysis due to shallowness of its cup. A study of volatilisation characteristics of SMSC electrode in a DC arc by racking plate method showed that even with this electrode the volatilisation of elements Al, Ca, Co, Cu, Fe, Mg, Mn and Si from a graphite matrix prolongs for at least 75 seconds. This necessitates a long exposure time for the determination of these elements by the complete burn method and as such the background becomes very high. Thus the analysis of these elements suffers because of low line to background intensity ratio.

It was found that undercutting of the SMSC electrode restricts the heat to the top undercut portion and in this way the volatilisation of all the elements is achieved in a short time of 30 seconds.

Figure 1 shows the line diagram of the original SMSC electrode and modification made to it by a small cutting tool on a microlathe. By a systematic study of volatilisation of impurities from original SMSC and the undercut (modified), it is seen that the complete burn is achieved in shorter periods in the latter. Table I shows the results of this study for 3 representative elements.

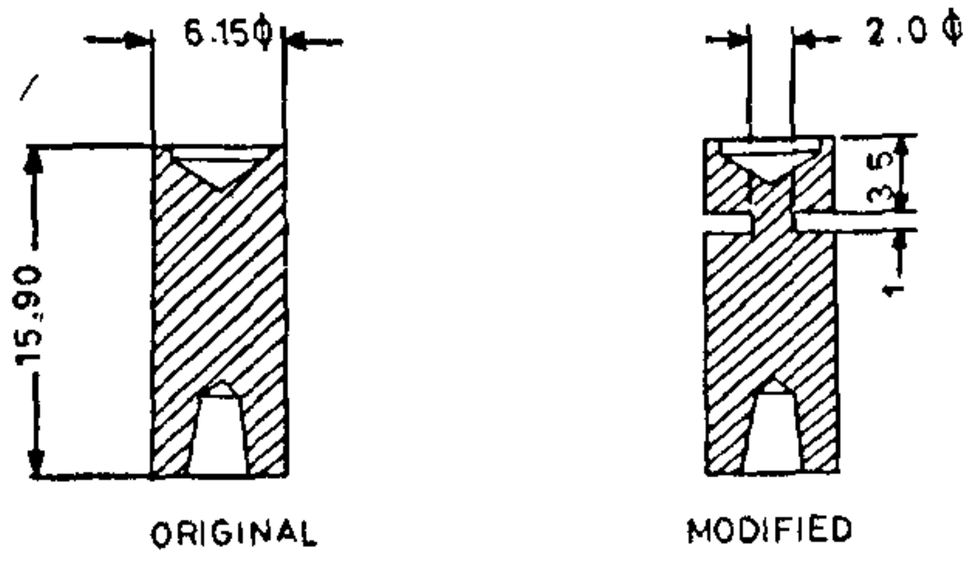

FIG. 1. Line diagram of original and modified Scribner Mullins shallow cup electrode.

TABLE I

Per cent volatilisation of impurities from graphite matrix for different arc periods using original Scribner Mullins shallow cup (SMSC) electrode and the modified one

Element	Arc period Seconds	Per cent volatilisation	
		SMSC	Modified SMSC
Fe	0–15	65	92·2
	15–30	18·3	7·8
	30–45	7·8	0
	45–60	4·9	0
	60–75	3·8	0
Mg	0–15	85·6	92
	15–30	7·6	8
	30–45	3·6	0
	45–60	3·1	0
	60–75	0	0
Si	0–15	66	81
	15–30	11	11
	30–45	13·3	8
	45–60	7·5	0
	60–75	7·1	0

Further experiments were done by undercutting the electrodes at 8 mm (half length) and 4 mm (quarter length) from the cup. A 10 mg weight of the graphite, containing known amounts of impurities, was put in the cup of each one of them and also in the original SMSC electrode. The charge was excited for 30 seconds in a D.C. arc at 10 amperes current and spectra recorded on Kodak SA-1 plate on a Jarrell Ash 3·4 m spectrograph. The densities of selected lines were measured, converted to intensities and compared. Table II shows some results of these experiments. It is seen that additional integrated intensity is obtained in 30 seconds by undercutting the electrode at half length and the highest intensity is obtained when the electrode is cut at quarter length.

A precision study for the elements Al, B, Ca, Co, Fe, Mg, Pb, Si, Ti and V in graphite was undertaken with the original and the modified (undercut at 4 mm from the cup) electrodes. This study showed that for elements B, Pb, Ti, Si and V, the precision was the

TABLE II

Relative intensities obtained for 30 seconds exposure from a graphite sample with Scribner Mullin's electrodes of different shapes

Element	SMSC	SMSC UC at 8 mm	SMSC UC at 4 mm
Ca	5·7	6·8	6·9
Cu	2·8	2·8	5·4
Al	10·2	13·8	15·0
Fe	6·3	7·8	8·0
Mg	1·5	1·9	1·9
Mn	3·6	4·1	4·9
Si	2·9	3·3	3·4

UC = Undercut.

same or better with the modified electrode. For Al, Ca, Co, Fe and Mg, the original electrode gave better precision. The chief advantage of the modified electrode is that the complete burn is obtained in a short time of 30 seconds for all the elements, while it retains the advantage of original electrode, *viz.*, easy loading and unloading on the electrode stand since SMSC sits on a pedestal electrode.

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FISSION TRACK DATING OF MUSCOVITES FROM PEGMATITES OF THE SHILLONG PLATEAU

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THE geochronological data of the crystalline rocks of the Shillong plateau are very scarce. So far, dating of the three samples of the region by Sarkar[1] and Crawford[2] was done by radiometric (K–Ar; Sr–Rb) method. The present work is an attempt to date some minerals of important litho-units of this region by applying fission track technique[3–5]. This study deals with the measurement of fission track ages and uranium concentration of some muscovites from two pegmatites of the Shillong plateau.

Collection of Samples

Coarse books of muscovites were collected from two pegmatites the one of which outcrops near Nengkhra in the Garo Hills district and the other near Nongkhlow in the Khasi Hills district of Meghalaya. Both the pegmatites are within a Precmabrian complex.

Experimental Procedure

Compact and neat muscovite specimens are selected from the coarse books for the study. Each sample is cut into a number of small pieces ($\geqslant 1$ cm^2) which are then cleaved along the basal plane into three slices. The outer slices are etched in 48% HF acid for 2·5 hr at 26° C and then are scanned for the fossil tracks by a polarising microscope under a moderately higher magnification. The inner pieces are irradiated with a known dose ($\sim 10^{16}$ *nvt*) of thermal neutrons in the CIRUS Reactor at Trombay. The neutron dose (ϕ) is accurately determined by measuring the track density on a standard glass of known U-concentration irradiated simultaneously along with the muscovite samples. The irradiated samples are then etched by the similar process as done above and scanned for the induced tracks.

The fission track ages and the uranium concentration of the mineral are determined by using the following equation (Price *et al.*[6]).

$$T = \frac{1}{\lambda_D} \cdot \ln\left[1 + \frac{\rho_s \sigma \phi \lambda I_D}{\rho_i \lambda_f}\right] \qquad (1)$$

where λ_D, λ_f, σ, ϕ and I are constants,

and U-concentration $C = K \frac{\rho_i}{\phi}$ (2)

ρ_s, ρ_i and ϕ are fossil track density, induced track density and neutron dose respectively.

The fission track data and the inferred ages of the muscovites from the two pegmatites are given in Table I [the estimated neutron dose $(3{\cdot}6 \pm 0{\cdot}5) \times 10^{16}$ nvt (4851)].

Conclusion

It is seen from the present fission track study that the mean ages obtained for the MPG and MPK (Table I) are 531 ± 15 m.y. and 501 ± 25 m.y. respectively. The observed data for the muscovites of the two pegmatites of separate occurrences show very close ages and suggest that both the pegmatites of the Shillong plateau may belong to a common geological event which corresponds to the period of Indian Ocean cycle (Sarkar[1]).