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## OBSIDIAN HYDRATION DATING OF A PLEISTOCENE AGE SITE FROM THE MANUS ISLANDS, PAPUA NEW GUINEA

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Obsidian hydration dating relies on the precise measurement of the depth of hydration developed over time in the surface of obsidians, but the loss of surface by natural dissolution at some archaeological sites can result in erroneous age determinations. By focussing the hydration measurement on internal crack surfaces protected from external surface erosion, acceptable results have been achieved from a Pleistocene age site in Papua New Guinea. Measurement of the hydration profile in thin sections of the sampled obsidian by computer imaging results in an improved reading error for the hydration depth. By using the system on archaeological obsidians from the Pamwak site, the relative age results are more consistent with the radiocarbon age determinations than conventional obsidian hydration dating.

### INTRODUCTION

Hydration is a measurable part of a weathering process affecting the surface of obsidians exposed to suitable moisture and temperature conditions. By showing experimentally that the hydration progress is proportional to the square root of time, Friedman and Smith (1960) were able to establish a dating system based on the hydration thickness of obsidian artefacts. Subsequent work by others has confirmed the basic hydration rate determining mechanisms that were proposed by Friedman and Smith (Ericson, 1988). Over geological time the obsidian may convert to perlite as a consequence of the approximately ten-fold increase in water content (Friedman *et al.*, 1966), or suffer surface dissolution at a rate mainly conditioned by the chemical nature of the aqueous medium surrounding the obsidian (McGrail *et al.*, 1988). The twin effects of hydration and surface loss occur as complementary weathering mechanisms in reducing glassy obsidian to other products.

Archaeological work aimed at establishing the rate constants of this weathering process for dating purposes has generally ignored the dissolution effect and concentrated on the visible hydration in order to calculate its time dependence (McGrail *et al.*, 1988). When the depth of hydration is used as a measure of time since a pristine obsidian surface was exposed on a newly struck flake, any unaccounted surface loss that may accompany the hydration will result in an indifferent or ambiguous age determination. Even where an independent system, usually radiocarbon dating, provides the time factor for calculating the hydration velocity for selected obsidians within an archaeological deposit, any unaccounted surface dissolution can create errors in the rate constants extrapolated to other obsidians. Hydration can be observed in microscope thin sections but the surface loss may not always be clearly indicated by surface effects, such as irregular surface pitting (Ambrose, 1976).

### SURFACE DISSOLUTION

Bates *et al.* (1988), Stevenson *et al.* (1989a) and Tremaine and Frederickson (1988) review the evidence for experimentally found hydration rates of obsidians at temperatures up to 250°C, in deionised water, and conclude that high temperature hydrothermal conditions produce unreliable hydration rate constants for application to hydration dating in normal terrestrial conditions. This mainly emerges from surface dissolution in silica–water reactions at elevated temperature in aqueous solution with low solute concentration as described by Marshall (1980) and Rimstidt and Barnes (1980). Marshall's results show a distinct reduction of silica solubility below 100°C but approximately straight line behaviour above this temperature when the logarithm of silica solubility is plotted against the reciprocal of absolute temperature (Fig. 1). As silica dissolution decreases faster at lower temperatures, the high temperature derivation of hydration rates for application at a lower temperature could yield younger than expected ages when the constants are applied to archaeological obsidians. This result could be expected from a temperature related surface loss rate, where measurement of the residual hydration would yield a lower apparent ratio of hydration rate to temperature. Such a higher temperature response projected to a temperature below 100°C would be evaluated as an artificially fast hydration velocity, which when applied to archaeological obsidians would produce a lower than 'real' age. At 25°C Marshall's measured solubility for silica in water is about 13% less than predicted from the solubility measurements between 100 and 300°C. While the high temperature studies emphasise the soluble nature of obsidian in extreme conditions, the same effect can occur over time in normal site conditions and needs to be recognised as a potential source of dating error. The dissolution of obsidians in the normally variable and heterogeneous conditions of archaeological sites precludes the

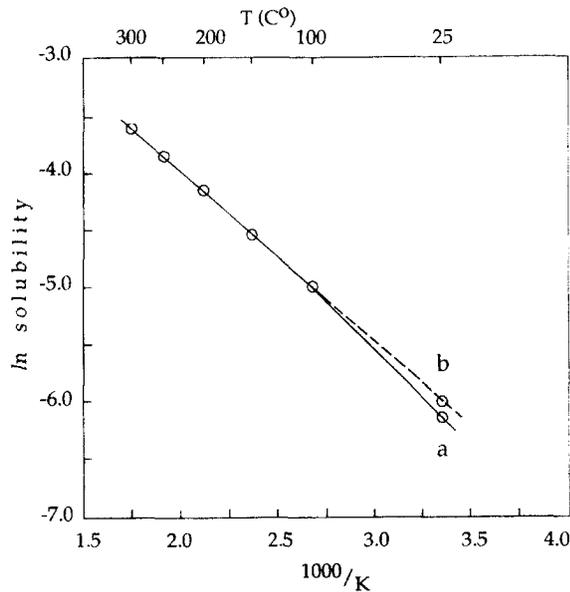


FIG. 1. Measured solubility of amorphous silica in water at 300, 250, 200, 150, 100 and 25°C (a) from published data of Marshall (1980) showing significant change between 25 and 100°C, and (b) predicted solubility from the 100 to 300°C regression slope.

use of any simple laboratory based procedure for determining loss rates.

The suggestion of Stevenson *et al.* (1989a), that the problem of dissolution can be overcome at high temperature by adopting saturated water vapour instead of liquid water for accelerated hydration studies, does not address the difficulty of assessing the nature of hydration at lower temperature within archaeological deposits. In natural sub-soil conditions high local saturation and condensation is probably commonplace, so that surface dissolution from a combination of chemical and biological attack can occur. The real difficulty in the field is that this can be highly variable from one part of a site to another.

The obsidian-water reaction is complex with different models for the dissolution and hydration of natural glasses being proposed (Abrajano *et al.*, 1988; White, 1988). In the case of related crystalline silicates the progress of surface dissolution is dependent on aqueous surface chemistry (Casey *et al.*, 1992), and is preceded by leaching of some cations. Burial in a clay matrix can accelerate surface dissolution in glass by causing chemical exchange reactions with the surrounding clay minerals (Curti *et al.*, 1993). Cryptoendolithic microbial communities, have also been instrumental in reducing basaltic glass to a porous pit-textured surface (Thorseth *et al.*, 1992), and indicate an additional complexity in understanding the development of hydration in archaeological deposits. Dissolution can be occurring at a rate different from, and superimposed upon, a hydration rate developing as a function of the square root of the existing hydration depth, so that the time dependence of the growth of the remnant hydration will be a differential and no longer a simple square root function of time. The ultimate thickness of the hydration layer on exposed surfaces is governed by the dissolution rate and will balance at a static thickness when hydration growth is equalled by dissolution loss. If the depth of hydration is to be used to determine the

age of manufacture of an obsidian flake, then the loss of surface due to dissolution needs to be considered along with the other major determinants of hydration growth; namely the temperature history at the site and the chemistry of the obsidian itself.

An impression left by many efforts to use the rate of hydration as a dating system is the poor correlation between hydration thickness and time. For example the comparison of radiocarbon, tree ring, archaeomagnetic and hydration methods, by McGrail *et al.* (1988, in their Fig. 1), shows consistently younger hydration age values with an overall wider age range. Such unsystematic variations in hydration thickness may often be attributed to site disturbance or artefact re-use, when surface loss may also be implicated.

This paper examines the use of hydration measurement in dating a site where advanced surface erosion of the obsidians is visible on most of the artefacts, thus making the conventional hydration system of direct surface measurement unworkable. By focussing on internal surfaces created when the obsidian flakes were struck, a consistent series of hydration measurements has been made. The reading error common to visual methods for measuring the microscopic hydration image has been reduced by employing computer digital scanning and video analysis software.

#### DATING THE PAMWAK SITE

The Pamwak rock shelter is an erosional feature at the base of a Miocene limestone outlier about 4 km inland on the southern side of Manus Island, in Papua New Guinea. It is subject to active tropical weathering being only 2° south of the equator. The site is archaeologically important because of the richness of the remains both faunally and artefactually, but particularly for the time range of its occupation based on radiocarbon dates extending to around 14,000 BP from about midway through the 3.8 m depth of the site. The site represents probably the oldest evidence for long distance oceanic settlement yet recorded, at about 200 km from the nearest islands of New Ireland. More than 30 radiocarbon dates are completed for Pamwak but some anomalies of age with depth occur, and may be due to site disturbance during the prehistoric use of the site (Fredericksen *et al.*, 1993).

Several thousand flakes of obsidian were recovered in excavations over 4 m<sup>2</sup> and to a depth of about 1.8 m; but below this level obsidian is absent, with chert being the dominant artefact material. The sheltered nature of the site, being shaded from direct sunlight for most of the day, the deep profile with abundant obsidian, a good series of radiocarbon dates, and a stable oceanic equatorial mean temperature regime recorded from the main Manus island at 27.2°C ± 2.8 (McAlpine *et al.*, 1983), and measured at the site over a 3 week period as 25°C, make it a suitable location for applying obsidian hydration dating. The attraction of dating the obsidian is to give a comparison with the series provided by the radiocarbon determinations where datable organic material is sparse. On the other hand the limestone sedimentary conditions, and weathering processes that are very active in the humid tropical environment, have produced aggressive conditions with severe surface erosion of the obsidians. Therefore a major question to be addressed

is the feasibility of employing hydration dating over such a long time span in a site where weathering processes are most rapid. A successful application of hydration dating at the Pamwak site will provide a useful strategy to apply at other archaeological sites with similar weathering problems.

The Pamwak obsidians vary in appearance with a gradient between highly reflective glassy flakes in the upper level of the site to corroded, opaque and dull flakes becoming more common with greater depth. In microscope thin section the dissolution of the obsidians has resulted in different forms of surface appearance ranging from deep etch pitting, and the formation of micro-erosion channels, to a low profile micro-texture or areas where apparently no surface erosion is present, but where the non-uniform thickness of the hydration indicates a differential loss of surface material. Although hydration is present in most of the sampled obsidians as a zone beneath the weathered surface some examples show no visible hydration. Despite this poor prospect for measuring hydration on the obsidian surface, there are other zones within the artefact that are more promising.

#### CONCEALED FISSURE HYDRATION

When obsidian is struck to produce flake artefacts, the point of percussion receives a blow over an area that may suffer inconsequential shattering. A flake may be only partly detached due to incompletely propagated cleavage and remain as a fissure plane or cone that is preserved to some depth in the body of a core or flake. The fissure allows entry of water vapour into the obsidian so that hydration occurs, but is sealed from the erosional influence of the soil surrounding the outer surface of the obsidian. By selectively measuring the hydration within fissures of incompletely propagated flakes, satisfactory readings have been made, even in cases where weathering has produced an external surface devoid of hydration. Because there is often some progressive lateral weathering from the outer surface along the fissure to produce an open conduit, the contrasting unaffected zone is very clear as two parallel hydration bands in contact along their length. In these instances the relative hydration thickness in the weathered and unweathered portions is clearly resolved (Fig. 2). From this observation

TABLE 1. Differences in obsidian hydration thickness between the interior concealed fissures and the external exposed surfaces on the same specimen. The generally higher exposed/concealed hydration ratios in the deposits above 0.7 m may indicate a depth related difference in erosion rate, or a time related change when less aggressive weathering conditions existed in the period after deposition of the 0.7 m level

Index no.	Level m	Concealed $\mu\text{m}$	Exposed $\mu\text{m}$	Ratio exp/conc
4794	-0.20	8.09	6.09	0.75
4782	-0.50	10.02	9.22	0.92
4788	-0.60	10.26	9.33	0.91
4789	-0.60	11.28	9.45	0.84
4791	-0.60	10.38	9.23	0.89
4792	-0.60	9.97	9.16	0.92
4800	-0.65	10.75	8.60	0.80
4801	-0.70	10.15	3.97	0.39
4802	-0.70	10.41	5.92	0.57
4804	-0.75	10.32	5.85	0.57
4775	-1.25	12.69	6.87	0.54

the rate of surface dissolution could be found by using the concealed hydration to provide the time base for the reaction. Not all the obsidians in this study provide clear evidence of the relative rate of hydration to dissolution, but those that do show that the outer surface hydration thicknesses vary widely and would provide no basis for dating the Pamwak site

#### HYDRATION MEASUREMENT

An important factor in obsidian hydration dating where errors are prone to occur is in the act of measurement of the depth of the hydration zone. Commonly this procedure involves the use of visual matching of the hydration zone against a graticule or split image device installed in the microscope eyepiece and calibrated similarly against a stage micrometer. The effect of a 'fuzzy' diffusion front (Scheetz and Stevenson, 1988) makes measurement error a persistent factor whatever device is employed. A survey across several dating laboratories to cross-calibrate results of visual measurements on standard microscope thin sections of hydrated obsidians, has illustrated the error-prone nature of this procedure (Stevenson *et al.*, 1989b; Jackson, 1990) and shows that the inherent limits of optical resolution are compounded by the need to use visual judgement to define

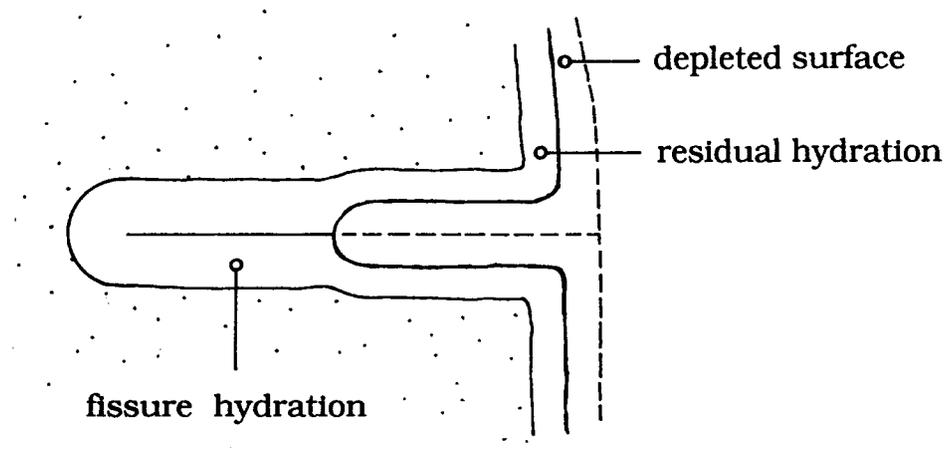


FIG. 2. Schematic view of hydration within an old fissure with dissolution proceeding from the outer surface along the cleavage plane.

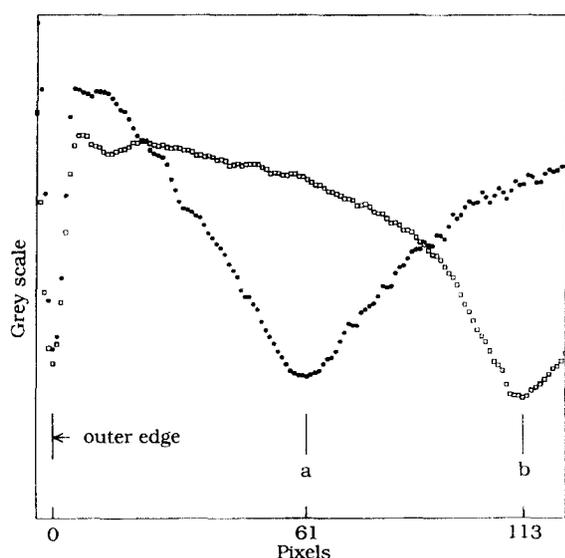


FIG. 3. Typical profiles of obsidian hydration (sample 4775) showing combined readings from the external weathered surface (a) of 61 pixels and (b) the internal concealed surfaces of 113 pixels. The scans are combined from 3 and 18 strips respectively. The combined scans of the hydration zones indicate spans of 6.73  $\mu\text{m}$  and 12.46  $\mu\text{m}$ . The arithmetic derivation of hydration widths from the raw strip scans gives 6.87  $\mu\text{m} \pm 0.75$ , and 12.69  $\mu\text{m} \pm 0.73$ .

the boundaries of the hydration zone. The net effect is poor reproducibility and error margins in calculating the final hydration thickness.

Instrumental methods which avoid the need for visual judgement are available with computer imaging technology and have been applied successfully to obsidians from archaeological sites in Manus (Ambrose, 1993). This approach is related to photodensitometry which was first applied to obsidian hydration measurement by Haller (1963) and Michels and Bebrich (1971). For the Pamwak collection, observation of the hydration bands are made at  $\times 1000$  magnification using a Leitz microscope. The image of selected strips is captured via the JAVA<sup>®</sup> PC video analysis

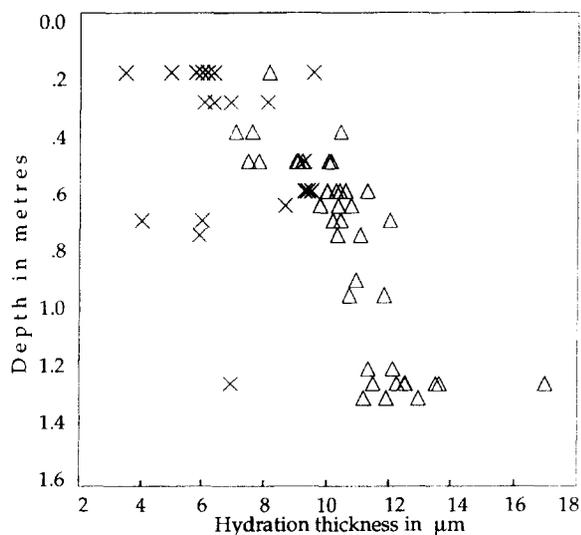


FIG. 4. Relationship of hydration thickness, in micrometres ( $\mu\text{m}$ ), with depth in cm, at the Pamwak site with readings made on internal surfaces shown as triangles and exterior surfaces as crosses.

system (Jandel Scientific, 1990) and then integrated as a single grey scale pixel profile across the hydration zone. The measurement of hydration width as expressed in pixels is calibrated against measurements of a stage micrometre. From this a one pixel dimension is equivalent to  $0.1103 \mu\text{m} \pm 0.0010$  and is the limit of resolution with this equipment. Depending on the size of the obsidian and presence of clear hydration, up to 20 strip scans are made and combined into a single profile which effectively gives an integrated hydration thickness for the specimen. All scans are filed on disc to allow the statistics of hydration width for each obsidian piece to be calculated. Two sets of combined scans from specimen 4775 of the diminished outer surface (a) and the concealed internal surface (b), show the extent of surface dissolution (Fig. 3). In this example the 21 primary scans when combined into two groups produce hydration zone spans for (a) and (b) of 61 and 113 pixels respectively. The calculation of thicknesses from the statistics of all scans gives (a)  $n3 \ 6.87 \mu\text{m} \pm 0.75$ , and (b)  $n18 \ 12.69 \mu\text{m} \pm 0.73$ .

TABLE 2. Hydration thickness readings for concealed surfaces and relative hydration ages based on radiocarbon dates for level -6.0 m

Index	Level	Microns $\mu\text{m}$	Years	
4794	-0.20	$8.09 \pm 0.24$	$4599 \pm 273$	
4779	-0.40	$7.55 \pm 0.11$	$4005 \pm 117$	
4779	-0.40	$7.02 \pm 0.17$	$3463 \pm 168$	
4780	-0.40	$10.42 \pm 0.39$	$7629 \pm 571$	
4782	-0.50	$10.02 \pm 0.25$	$7055 \pm 352$	
4783	-0.50	$8.97 \pm 0.24$	$5654 \pm 303$	
4783	-0.50	$9.00 \pm 0.22$	$5691 \pm 278$	
4783	-0.50	$9.02 \pm 0.21$	$5717 \pm 266$	
4783	-0.50	$10.10 \pm 0.26$	$7168 \pm 369$	
4784	-0.50	$7.76 \pm 0.33$	$4231 \pm 360$	
4785	-0.50	$7.42 \pm 0.30$	$3868 \pm 313$	
4787	-0.50	$9.17 \pm 0.24$	$5908 \pm 309$	
4788	-0.60	$10.26 \pm 0.15$	$7397 \pm 216$	C14 ANU 7122
4789	-0.60	$11.28 \pm 0.23$	$8940 \pm 365$	and ANU 8239
4790	-0.60	$10.57 \pm 0.77$	$7850 \pm 1144$	pooled mean age
4791	-0.60	$10.38 \pm 0.15$	$7571 \pm 219$	of bp 7732 $\pm$ 89
4792	-0.60	$9.97 \pm 0.18$	$6984 \pm 252$	
4798	-0.65	$10.33 \pm 0.06$	$7498 \pm 87$	C14 ANU 7763
4799	-0.65	$9.74 \pm 0.49$	$666 \pm 671$	BP 9893-9500
4800	-0.65	$10.75 \pm 0.25$	$8120 \pm 378$	
4801	-0.70	$10.15 \pm 0.06$	$7239 \pm 86$	
4802	-0.70	$10.41 \pm 0.42$	$7614 \pm 614$	
4803	-0.70	$12.00 \pm 0.29$	$10,118 \pm 489$	
4804	-0.75	$10.32 \pm 0.23$	$7483 \pm 334$	
4805	-0.75	$11.06 \pm 0.42$	$8595 \pm 653$	
4572	-0.90	$10.92 \pm 0.23$	$8379 \pm 353$	
4575	-0.95	$10.70 \pm 0.38$	$8045 \pm 571$	C14 ANU 8240
4576	-0.95	$11.82 \pm 0.20$	$9817 \pm 332$	BP 10883-10365
	-1.15			C14 ANU 8243
				BP 12429-12104
4582	-0.20	$11.32 \pm 0.18$	$9004 \pm 286$	
4583	-1.20	$12.11 \pm 0.34$	$10,304 \pm 579$	
4775	-1.25	$13.60 \pm 0.50$	$12,996 \pm 956$	
4775	-1.25	$13.48 \pm 1.03$	$12,768 \pm 1951$	
4775	-1.25	$12.51 \pm 0.42$	$10,996 \pm 738$	
4775	-1.25	$12.24 \pm 0.19$	$10,527 \pm 327$	
4776	-1.25	$16.99 \pm 0.47$	$20,282 \pm 1122$	
4777	-1.25	$11.47 \pm 0.19$	$9244 \pm 306$	
4777	-1.25	$12.49 \pm 0.47$	$10,961 \pm 825$	
4585	-1.30	$11.17 \pm 0.11$	$8767 \pm 173$	
4586	-1.30	$11.90 \pm 0.26$	$9950 \pm 435$	
4587	-1.30	$12.93 \pm 0.17$	$11,747 \pm 309$	

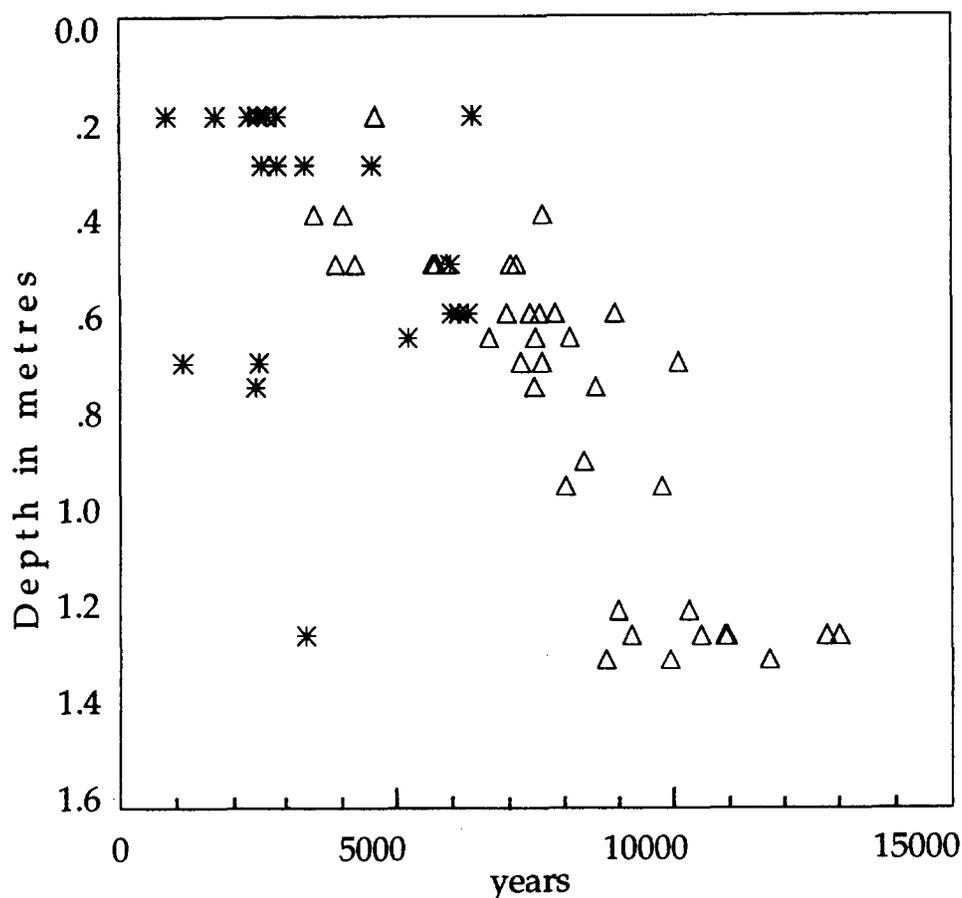


FIG. 5. Depth in metres against age in years, with the hydration rate constant based on the hydration thickness of obsidians, and the corrected pooled mean of two radiocarbon ages ( $7732 \pm 89$  BP), from between 0.60 to 0.65 m depth. Obsidian no 4776 (Table 2) at around 20,000 years is outside the plotted age range of this figure. Triangles = concealed hydration, stars = exposed surface hydration.

### MATERIALS

A single  $0.5 \text{ m}^2$  column of 5 cm spits from the SE quadrant of Square 2 was sampled for obsidians that appeared in hand specimen to have remnant shatter cones or un-propagated flakes from the time of their manufacture. These features were sought and often found around the bulb of percussion. Sections were cut across the target zone using a 0.2 mm wire diamond saw to produce a wafer oriented normal to the plane of hydration. The wafer was then used to prepare a standard microscope thin section. Some small specimens yielded less than  $2 \text{ mm}^2$  surface area or failed to retain the hydration zone after slide preparation. Fifty obsidians were processed to provide the sample described here.

### RESULTS

It can be seen from Fig. 4, and Table 1, that a consistent increase in hydration thickness follows depth in the site for readings made on the concealed fissures. This compares with the lack of correlation between hydration thickness and depth for the hydration readings made on the outer surfaces where they are present.

In order to compare the set of obsidian hydration results with the set of radiocarbon dates, it is necessary to convert the hydration readings into age values. In the absence at this stage of independent evidence for hydration rates for the

Pamwak obsidians, two corrected pooled mean radiocarbon dates for spit 6.0 are used as the rate determining age for the associated obsidians. The radiocarbon dates are calibrated using the program of Stuiver and Reimer (1993) on samples from the same stratigraphic sampling column as the obsidians. These are ANU 7122 at  $6280 \pm 250$  BP and ANU 8239 at  $7949 \pm 90$  BP, which are significantly different, but for the purpose of providing an interim age for the level of 0.60–0.65 m, they are combined to give a pooled mean value of  $7732 \pm 89$  BP. The mean hydration thickness of the 0.60 m level obsidians is ( $n=5$ )  $10.49 \pm 0.49 \mu\text{m}$ . Using the standard obsidian hydration equation of  $X^2 = kt$  where  $X$  is hydration thickness in  $\mu\text{m}$ ,  $k$  is the rate constant and  $t$  is time, values for the complete set of measured obsidians were determined with  $k$  at 0.01423. The results are presented in Table 2 and Fig. 5.

The real age values eventually derived from laboratory based hydration rates of the obsidians will probably differ from those expressed here, but their relative ages should remain. Analysis of the major chemical components of the obsidians shows them to be from three chemically closely related sources. Further development of the dating programme at Pamwak is continuing and will include considerations of site temperature, chemical analysis of all the hydration specimens and experimental determination of hydration velocity using powder samples at normal terrestrial temperatures.

## CONCLUSION

Despite the unpromising aggressive tropical weathering conditions, a useful application of hydration measurement has been achieved at the Pamwak rock shelter site. The picture presented by the radiocarbon dates of some mixing of deposits over time is paralleled by the obsidian results, although a clear age/depth trend is evident. The study is proceeding on a larger set of obsidians in order to test the hypothesis of site disturbance, to identify major periods of occupation, to assess changes in obsidian weathering rates over time, to apply experimentally derived hydration rates, and to incorporate site temperature measurements to allow age assessments to be made independent of radiocarbon dating.

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