Role of Water in Cracking of Opal

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INTRODUCTION

Much Australian opal won of recent years has been subject to crazing and cracking thereby reducing buyer confidence in its desirability as a gem. Material from certain localities has acquired a reputation for being particularly unreliable, solely in the light of experience, and that from other areas of being remarkably stable. Crazing and cracking may occur when the rough has been won, or just cut and polished, but in some cases may be delayed for months or longer after the polished goods have passed through several hands.

To date there seems to have been no objective criterion by which the stability of a particular sample of opal could be predicted, although experienced dealers and cutters frequently have subjective and often unexplainable techniques for assessing stability. This writer too, during experience with cutting material from various locations, has developed a "feel" for judging cracking possibilities, which, upon reflection, is a synthesis of various features including the nature and quality of the fracture surface of rough, the presence or absence of "ginger whisker" and gypsum, the nature of the surface lustre, and other not specifically identified factors which are unconciously integrated during assessment. However, this is far removed from a quantifiable physical or chemical parameter that can be used as an index of stability and could be routinely measured by prospective dealers or clients to ensure satisfaction in a transaction.

The intention of this investigation was to attempt to identify some physical property which could be shown to correlate with crazing possibility, and samples of opal from known sources were examined by various techniques, including measurement of total volatiles content, thermal analysis, refractive index and relative reflectivity.

CONSTITUTION OF OPAL

Opal has been shown to be largely amorphous hydrous silica gel with a unique ordered microstructure that can cause diffraction of light in the visible band[1,2]. The silica is relatively pure but nevertheless contains varying amounts of iron, titanium and uranium which may be responsible for the body color and its fluorescence and phosphorescence, as well as traces of other elements such as calcium and magnesium.

Florke and Langer[3] have classified opal from various worldwide localities into several categories including non-crystalline and crystalline (disordered cristobalite — tridymite) by X-ray diffraction, which were then subdivided into gel-like and network glass-like varieties. Commercial Australian opal of the Desert Sandstone Series all falls into the gel-like non-crystalline variety according to Florke and Langer's classification, in which the water content comprises two discrete forms, molecular absorbed or structurally unallocated water, and that present as structural silanol (-SiOH) groups. The range of total water contents quoted for opal from various sources is quite wide, 3.13 to 8.85% by Florke et.al[3], 3.4 to 9.73% by Dana[4], and even 1 to 35% by Todor[5] who suggests that most opal varieties lose the bulk of their

water between 100 and 150°C but that some opals may lose water even as high as 300°C.

The extent of variability of water content in Australian opal is not known, as it is particularly difficult to obtain specimens from confirmed locations within a specific mining field, and even more difficult to obtain confirmed samples from the one location but at different depths or levels. Such systematic information would be very relevant to the possible distribution of crazing-susceptible material as it is conceivable that parcels from different depths, even from the one claim or mine could have appreciably different water contents. It is tacitly assumed by miners and cutters that cracking is associated in some way with water content and that rough from greater depths is of higher water content and consequently more prone to cracking; although this has not been actually demonstrated.

It is also generally assumed but not experimentally confirmed that the water content of almost all pieces of rough within a particular lens or seam or parcel is relatively uniform. This seems reasonable as the water vapor partial pressure in equilibrium with the lens would be uniform within the limited confines of an opaliferous zone of a specific sedimentary stratum. However, it has been reported[6] that green "crystal" opal on grey potch from Coober Pedy Eleven Mile field invariably cracks a short time after mining whereas the grey potch substrate is quite resistant to crazing, suggesting that there may be other contributing factors to cracking than simply total water content. A similar circumstance has been reported with crystalline wood opal from Lake Eyre in South Australia in which the glassy portion with closed pore structure cracked abundantly, but the milky portion with an open pore structure was free from cracks[8].

The function of the pigmenting agents in facilitating or inhibiting cracking is unknown. Various opal varieties such as the dark grey material from Coober Pedy Black Flag, or similar coloured material from Grawin and Glengarry are reputedly unstable, unlike the grey to jet black opal

found at Lightning Ridge and Mintabie. White Cliffs, renowned for its white and crystal opal produces a sporadic output of translucent stable grey precious opal especially from Sullivans Hill, and a variety of material called magpie potch from Potch Gully in the "Blocks" area comprising white opal, usually potch, on a jet black potch core. Andamooka too has produced a small quantity of black opal and it is the writer's observation that some of the greyish varieties can be notoriously crack prone but that the very dark to black types are generally crack resistant.

It is not known whether the coloring agent is the same in all of these varieties, and despite the very sizeable capital investment in the opal industry and the revenue that has been reaped, virtually no systematic investigation of this unique product has been undertaken and technically it still remains poorly characterised.

FRACTURE TYPES

Fracturing in opal can be loosely categorised into several types.

(a) Surface crazing is a fine network of fracturing restricted to a surface layer which generally extends with time both in abundance of fractures and in depth.

(b) A more advanced stage of crazing may be observed in which the fractures are much deeper and prejudice the integrity of the stone.

(c) Propagation of pre-existing surface cracks by mechanical, thermal or other means is a more severe extension of stage (b).

(d) The fracturing may also be historical when the opal has suffered a high stress in its past, perhaps from ground movement, or from an excessively dry period that resulted in non-uniform shrinkage followed by ground water infiltrating fractures and depositing water-borne iron oxide as an extremely thin rusty film on the fracture surfaces. This feature is known as "ginger whisker" and immediately sounds a note of caution to the cutter since fracture rehealing

has been prevented by the oxide and the cracks are very readily propagated by the mechanical stresses of cutting, polishing and handling. "Ginger whisker" is particularly common from various areas at Coober Pedy but has been observed in opal from all fields.

Instances of type (c) fracturing have been afforded by ground movement, or mismatching of thermal expansive strains frequently seen on Andamooka "painted lady", or by over enthusiastic use of high brisance (i.e. shattering) explosives during the extraction of the opal. This occurrence is unfortunately seen in opal excavated from harder ground where transmission of such seismic stresses is more pronounced.

Thermal induction of opal fracture may be crazing, in which it is assumed that partial surficial loss of water causes differential shrinkage that can only be accommodated by fracture when the localised stresses exceed the fundamental rupture stress. Localised heating can cause cracking through differential expansive strains and stresses such as has been observed when stones are dopped by the hot sealing wax method, fortunately now largely outdated by cold-dop techniques.

The historical cracking in material from the adjacent Coober Pedy areas of German Gully and Black Flag may be difficult to detect because it is masked by the presence of an adhesive-like glassy transparent film of gypsum, a common opal-associated mineral, sealing the contiguous fracture faces and so preventing total internal reflections off an air film in the fracture. Infiltration of both iron oxide and gypsum into different fractures even in the one piece of opal has been repeatedly observed in Black Flag rough and probably occurs in parcels from other areas. Historical fracturing has also been observed with essentially no alteration or infiltration.

Historically fractured material may now be stable, and once potential crack nuclei have been removed, e.g. excision of ginger whiskered zones, the resulting goods are quite satisfactory for commercial dispersal.

Conversely, historically failed rough may have a tendency to progressive failure irrespective of the care and skill of the cutter, this situation being sadly not uncommon; but modern fracturing can be induced by various mechanisms such as thermal and mechanical shock and explosives during mining. This material may be intrinsically stable under ordinary ambient conditions, and when appropriately subdivided into fracture fragments and cut and polished is satisfactory for commercial dispersal. This applies particularly to explosively fractured material extracted from hard dry ground.

Ancient episodes of partial dehydration and rehydration could account for some internal fracturing of rough pieces of seam which externally appear intact, generating fluctuating stress fields that could locally exceed the rupture stress. The mechanical constraint of gel type opal forming in contact with a sandstone or quartzitic matrix of different thermal and expansive properties may well explain the presence of severe internal "ginger whisker" at the junction, for instance in German Gully rough; the matrix being a convenient source of ferruginous compounds giving "ginger whisker".

More insidious however is apparently sound material that progressively fractures during preparation and handling or upon exposure to normal ambient conditions of temperature and humidity, and to which the following investigation was largely addressed.

STRESS INDUCTION

During its millennia of formation opal is thought to have consolidated from an aqueous sol through a plastic gel to an amorphous solid[7] with such a slow rate of water extraction that the structure has not been disrupted and water concentration gradients have been minimised. The relatively uniform slow drying was attributed to the presence of the tertiary siliceous duricrust, colloquially called greybilly or shin cracker, whose permeability to water was very low in comparison to the opal bearing sediments beneath.

Synthetic silica gel is commonly used as a dessicant due to its ready ability to absorb or release water depending upon temperature and relative humidity. Deliberate immersion of such gel into water causes rapid violent decrepitation and disintegration due to the differential stress generated between particle core and the rind which actively expands as it takes up water.

It is considered that a converse but similar stress field is induced in natural opal as it loses water to the atmospheric environment at higher temperatures and lower relative humidities than the subterranean environment in which it was found and with which it was in equilibrium. Loss of water from the surface with consequent localised shrinkage would subject the opal surface to a tensile stress and the core to a compressive stress. The following experimental work implicitly assumes that the bulk of volatiles comprises water and the results are interpreted in that regard except where otherwise noted.

SPECIMENS INVESTIGATED

The set of specimens of confirmed origin tested comprised three samples from Coober Pedy, two from White Cliffs and one each from Mintabie and Lightning Ridge, described below.

Coober Pedy Black Flag (CPBF). Selected specimens from a parcel of medium grey cutting rough purchased through an intermediary direct from the miner and estimated to have been found at about nine metres depth.

Coober Pedy German Gully (CPGG). Selected representative white specimens from a parcel purchased direct from the miner for cutting, it being the bottom grade remnants of a much larger parcel found four years ago at about thirteen metres.

Coober Pedy Eleven Mile (CP11M) An approximately 30ct cut white, bright colour stone donated for investigation[6], it having developed severe crazing since it was cut.

Mintabie (MINT). Several selected polyhedral seam sections of dark translucent charcoal-grey with somewhat paler grey rind and watery colour, all pieces having originated in the same parcel and reportedly having been found in a shallow level between six and nine metres, exact location unknown.

Lightning Ridge Black Nobby (LRBN). A single large opaque jet-black potch nobby with a small amount of translucent glassy outer rind which was dressed off before investigation.

White Cliffs Golden Gully (WCGG). Portions of a parcel found in the western-most end of Golden Gully at a depth of eleven metres, it being sections of a damaged pseudomorph, found on the third level five years ago and comprising weak colour white opal.

White Cliffs Western Rush (WCWR). Samples from a parcel purchased three years ago and bulldozed from an open cut on the new area close to the western edge of the field on the Wanaaring road. The extraction depth was unknown and it was white potch with flow lines of weak colour, purchased shortly after discovery, directly from the miner.

SPECIMEN PREPARATION

Extraneous material, clay and gypsum, was removed by nippers and opal portions were powdered manually in a ceramic mortar with a pestle to minus two hundred mesh, just before use, to minimise adsorbtive loss or gain of water.

Thin fracture flakes were prepared with carbide nippers to give a clean relatively microcrack-free surface for investigation of fracture induction.

Fragments for refractive index and reflectivity determinations were metallurgically lapped and all polished on a scored perspex faceting lap with cerium oxide and water coolant, a recognized procedure for silica based minerals.

TEST PROCEDURES

1. Total volatiles content was determined by ignition in pre-ignited, lidded silica crucibles in an electric muffle furnace at 900°C for thirty minutes. To confirm that this was an adequate time one sample was re-ignited for a further thirty minutes but no additional weight change was detected. Determinations were conducted in

duplicate on samples of 0.5 to 1.0 gm each.

2. Refractive indices were determined on a G.I.A. Duplex II gem refractometer and reflectivities with a Hanneman relative reflectometer calibrated on synthetic corundum reference.

3. Thermal analysis was accomplished with two instruments, the differential scanning calorimetry with a Perkin Elmer D.S.C. and digital data processing station. Thermogravimetry and differential thermal analysis was conducted on a Rigaku thermal analyser from which chart output data was manually reprocessed.

4. Specific gravities were determined by the hydrostatic technique.

5. Crazing induction was investigated by immersing test-tubes containing dry fracture fragments into simmering water for a prolonged period with regular inspection to determine the time to onset of fracture.

More severe conditions for observing crazing induction were devised by inserting fracture flakes on a low thermal inertia aluminium support into a preheated laboratory oven at 200°C with the intention of periodic inspection for fracture onset under a stereo microscope.

6. In view of the unsatisfactory results obtained in (5) an additional procedure was devised with an intermediate degree of dehydration severity. Samples were exposed to various refluxing liquids with boiling points in the range 100-200°C, (toluene 110°C; xylene 138-142°C; kerosene 188°C) so that any water which was removed from the opal was trapped in the refluxing vapor in the condenser. The sample is thereby also exposed to the constant boiling temperature of the solvent removing the need for thermostatic control, and rapid heat transfer is accomplished. Periodic sample removal and inspection under the stereomicroscope revealed the presence of any crazing or cracking by reflection from the air film in the fracture of the dried specimen.

7. Untreated fracture flakes and those exposed to xylene refluxing where strain may have been induced by partial water removal were examined for anomalous birefringence in a polariscope.

THERMAL ANALYSIS

Thermogravimetry (TGA). Weight changes are continuously determined as a function of temperature in a known atmosphere (in this case air) at a programmed linear temperature elevation rate.

Differential scanning calorimetry (DSC) involves linear ramping of temperature of a specimen whilst measuring the heat input rate as a function of temperature compared to an inert standard. Endotherms or exotherms which represent chemical reactions or phase changes appear as peaks away from the horizontal baseline in the output graphs. Water having a very high latent heat of vaporisation (540 calories per gram) should be eminently detectable as it is progressively driven from the sample. Superimposed on the water vaporisation endotherm may be peaks corresponding to any other reactions such as silica phase changes and alterations in the nature of any coloration agents.

Differential thermal analysis (DTA) consists of programmed linear heating of the sample and a thermally inert reference material under identical conditions whilst continually recording the temperature and the temperature difference between the sample and reference. Departures from the horizontal baseline of temperature difference versus temperature indicate heat absorption or evolution and the thermal peak data complements DSC data in locating the onset of various chemical reactions frequently undetectable by other means.

Thermal analysis samples are typically about 10 to 20 mg.

RESULTS

The DSC and TGA results are presented in the accompanying plots, and the other

numerical data are in the following table. Several DSC and TGA curves have been presented on each plot for purposes of comparison. The initial discontinuity at low temperature of each DSC curve is a machine artefact and not to be interpreted as a discrete endotherm.

Fracture induction was not observed in any of the specimens held at 100°C for 4 hours but all specimens were crazed to different extents of severity after 15 minutes at 200°C. The crazing severity was not readily quantifiable by measuring the time to cracking onset, but was ranked under the stereomicroscope by crack abundance and a description follows the results table.

The resolution of the reflectometer results (each being the value of five consistent consecutive readings as the specimen is slightly moved to expose different zones of the polished face) was insufficient, and unquantifiable slight differences are indicated by a subscript plus or minus. Test faces displaying no diffraction color were selected to avoid interference.

The DTA curves were all of the same general form and displayed no significant features, the diffuse endothermic peaks all occurring at approximately the same temperature as shown in the generalised diagram, figure 1. The temperatures of maximum endotherms are listed in table 1. The volatiles content, assumed to be water, obtained from the weight loss on ignition at 900°C are listed in table 2, together with measured values of R.I., reflectance, S.G. and cracking susceptibility at 200°C in air.
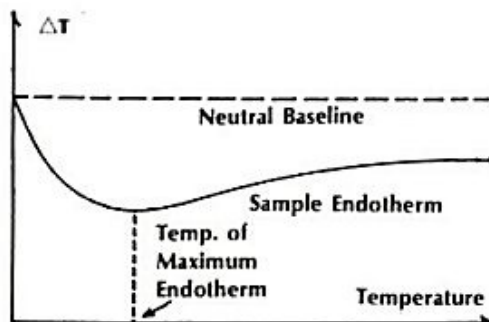

Figure 1. Generalised DTA curve.

ANOMALOUS BIREFRINGENCE

The specimens after refluxing in xylene were examined with a polariscope and compared with unheated fracture fragments to attempt to observe anomalous birefringence attributable to strain. Some apparent anisotropy was observed but it was only noticeable in zones showing diffraction color, and no difference between the xylene-refluxed and unheated specimens was detected. The reason for the apparent anisotropy is not known.

Specimen	Temperature at which Maximum $\triangle T$ occurs (°C)
MINT	187
LRBN	173
WCWR	250
WCGG	237
CPBF	328
CP11M	166
CPGG	292

Crack initiation in refluxing solvent

(a) Toluene (B.Pt. 110°C). Cracking was not observed in any specimen until after 1.75 hours when crazing was just observed on CP11M. Further refluxing was not conducted.

(b) Xylene (B.Pt. 138-142°C). Crazing of CP11M was extensive at 0.25 hours with specimen fragmentation. Crazing was just perceptible on CPGG at 0.75 hours but was not observed on any other specimen when the test was terminated at 5.75 hours.

(c) Kerosene (B.Pt 188°C). Crazing was progressively observed as below:—

Cumulative refluxing time (min).	Crazing onset & severity at time of examination
10	CP11M severely, CPGG mildly.
25	CPBF just perceptible.
40	MINT substantial.
55	WCGG substantial, WCWR just perceptible. LRBN no crazing.

The crazing ranking is thus CP11M, CPGG, CPBF, MINT, WCGG, WCWR, LRBN.
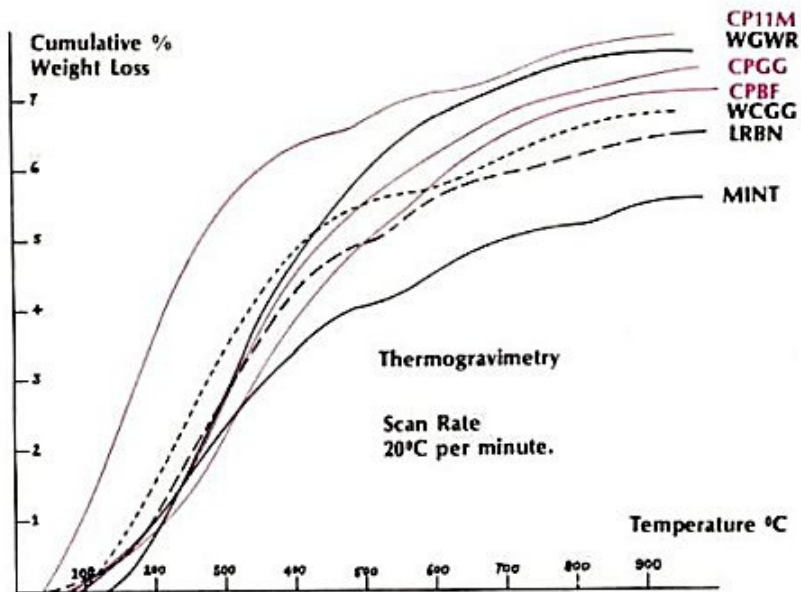
Table 1. DTA Endotherm Maxima

Fig. 2. Thermogravimetric analysis of the opal samples examined.

DISCUSSION

The materials investigated have acquired reputations with cutters and dealers for varied susceptibilities to cracking, the ranking from most to least likely to crack probably being in the sequence CP11M, CPBF, CPGG, MINT, WCWR, WCGG, LRBN. This is very similar to the order obtained in the kerosene refluxing test, but is not exactly the order of total volatiles content (water) shown in table 1.

The total volatiles contents, assumed to be water, closely conform with the ranking of the specimens in cracking severity at 200°C for 15 minutes but the very crazing-
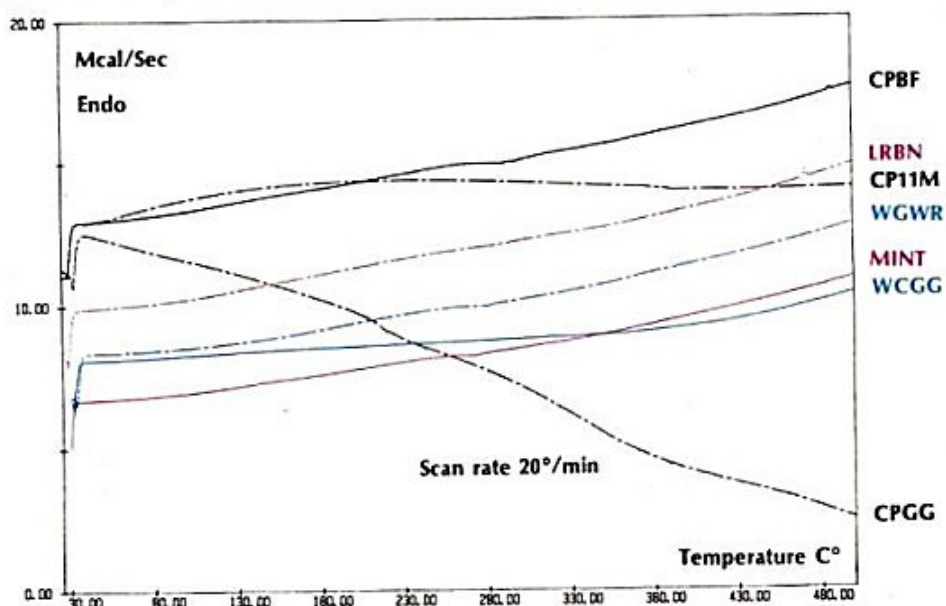

Fig. 3. Differential scanning calorimetry results of the opal samples examined.

susceptible CP11M is not quite of the same water content as the other more stable Coober Pedy varieties, suggesting that other factors may be involved. It has been surmised that voidage between the spheres comprising the opal microstructure may not be entirely discontinuous in some instances so that diffusion channels for facile water elimination from deeper in the stone may exist, thereby minimising concentration gradients and reducing stresses by minimising differential shrinkage. If the voidage was of discontinuous cusp shaped cavities due to deposition of additional cementing silica gel, water elimination through this more glassy non-porous material would necessarily be by slower diffusion through bulk silica, developing greater concentration gradients and shrinkage stresses and leading to higher crazing susceptibility. Such behaviour has already been implicated in Lake Eyre wood opal[8] and possibly Eleven Mile potch and color[6].

No correlation with crazing susceptibility was found for specific gravity or relative reflectance. However a dependence of refractive index on volatiles content was observed, the greater the water content the lower the refractive index, conforming with the principle of Kokta (1931) and of Taliaferro (1935) as described by Langer and Florke[3] and agreeing with the latter's results. An apparent anomaly to this rule was discovered in data given by Dana[4] in which it is claimed that prolonged water immersion increased the R.I. of each of four opal samples of diverse origin (one being from White Cliffs), and that prolonged drying at 74°C reduced the R.I. It is considered possible that these two data columns have been inadvertently interposed.

Nevertheless the variation in R.I. is too low to be a useful indicator of total water content or of cracking susceptibility, the range from highest to lowest values obtained being only 0.003.

Materials	Volatiles Content (%)	R.I.	R. Reflectance	200° Cracking Severity Ranking	Specific Gravity (20°/4°)
CPGG	7.95 ± 0.05	1.457	0.52_+	5=6=7	2.112
CP11M	7.88 ± 0.02	1.458	0.51_5	5=6=7	2.100
CPBF	$7.607.88 \pm 0.02$	1.458	0.52_-	5=6=7	2.114
WCGG	$6.967.96 \pm 0.1$	1.459	0.50_5	4	2.110
WCWR	6.60 ± 0.1	1.459	0.51_5	3	2.120
LRBN	6.49 ± 0.04	1.459	0.50_0	1=2	2.116
MINT	5.52 ± 0.02	1.460	0.51_0	1=2	2.128

Cracking Severity at 200°C for 15 minutes

CPGG — intensely crazed and shattered with appreciable explosive spalling.

CP11M — almost identical to CPGG and CPBF with perhaps marginally less severe crazing.

CPBF — exactly as CPGG.

WCGG — seriously crazed with some minor spalling but much less severe than the CP in that uncracked domain size is much larger by a factor of 10 to 20 times.

WCWR — almost identical to WCGG, perhaps marginally less severe with slightly larger uncracked domain size.

LRBN — very minor surface crazing with discontinuous cracks reminiscent of fishscales, no spalling.

MINT — almost identical to LRBN, but fishscale cracks more conspicuous in the grey transparent matrix in comparison to the black translucent to opaque LRBN, and thus appear somewhat more severe. Close inspection reveals approximately equal crazing.

The tolerances noted in the volatiles content column represents the spread about the quoted mean of the duplicate determinations.

Table 2. Comparative properties of opal.

THERMAL ANALYSIS

DSC. The DSC scans of the White Cliffs, Lightning Ridge and Mintabie samples WCWR, WCGG, LRBN and MINT respectively are all very similar with a distinct diffuse endotherm throughout the experimental temperature range of ambient to 500°C indicative of progressive water loss. No discrete or diagnostic peaks or characteristic features are evident other than a few minor inconsequential glitches possibly due to decrepitation of larger particles in the powder. The Coober Pedy Black Flag sample also displays a very similar constant featureless endotherm, but the highest water content Coober Pedy samples from Eleven Mile and German Gully (CP11M and CPGG) exhibit apparently anomalous behaviour in that the former material, CP11M, appears to be thermally inactive despite the demonstrated progressive water loss in the determination of total volatiles content and in the TGA scan. It seems clear that a competing progressive exothermic reaction approximately compensates for the highly endothermic water loss. The exothermic effect is far more pronounced for CPGG throughout the experimental temperature range. However, this sample loses the largest volatiles content, the major proportion being driven off between ambient and 500°C which can be seen by inspection of the TGA graph. The nature of this over-compensating exothermic reaction is quite unknown, but clearly is unconnected with chemical exidative changes in the dark pigmenting compounds of LRBN, MINT. or CPBF, which are visibly absent in CP11M and CPGG.

It was observed however that the pigmentation was almost entirely bleached from those three dark coloured samples during thermal treatment, but the nature of the chemical change is unclear and it is not apparent on the respective DSC scans.

TGA. The thermogravimetry scans of all but CP11M are quite similar, particularly in the lower temperature range. However CP11M loses volatiles at least at twice the rate of that of the other samples from ambient to 400°C and has the greatest susceptibility to cracking and almost the highest water content.

It is not known whether the rapid water loss from CP11M is a reflection of its proven crazing susceptibility by rapid generation of water concentration gradients and consequently shrinkage stresses, or of more facile water elimination through open pores of inter-sphere voidage. It is speculated that the difference in TGA behaviour of the various specimens may be related to the distributions of the different water categories described by Langer and Florke[3] including the structurally unallocated cage and film types of adsorbed water and structural silanol water.

DTA. The differential thermal analysis results do not faithfully reflect all of the DSC results in that only DTA endotherms were observed but the DSC data indicate that CP11M was approximately thermally inactive and CPGG was actually inactive despite having the highest exothermic despite having the highest water content. Little correlation of ΔT temperature location with crazing susceptibility was observed, except perhaps that the very crazing-prone CP11M exhibited the lowest temperature at which ΔT was at maximum. As with the TGA results the reasons for the different behaviours of the specimens tested can only be speculated upon at this stage, but they do not appear to be relevant to crazing susceptibility.

SOLVENT REFLUXING

There is no specific reason to equate ranking with respect to time of crazing initiation with the severity of crazing but it would seem reasonable that the materials most prone to early failure, would suffer worst if crazing is due primarily to progressive loss of water. In practical commercial terms however, once a stone has crazed the subsequent severity of crazing is of little consequence, so the prime criterion of stability is considered to be the crazing onset time. In this regard, the periodic inspection of test material refluxed in an appropriate inert solvent is a better assessment than comparison of uncracked domain size after exposure for a specific time to a much elevated

temperature such as 200ºC.

The conditions of refluxing toluene or xylene are insufficiently agressive to obtain useful results in a convenient time. Boiling kerosene at 188ºC causes crazing at progressive stages of all of the specimens tested within one hour except for the Lightning Ridge black nobby, which was also observed to be the most resistant to crazing at 200ºC in air.

The test specimens were readily ranked in susceptibility.

If the writer's cutting experience of Black Flag opal is considered representative and that it is on the borderline of satisfactory commercial stability, refluxing kerosene offers a convenient test for accepting a parcel of stone or penalising or rejecting it depending upon whether crazing is observed after twenty five minutes of boiling it gently in kerosene of confirmed boiling point 188ºC; such a test is very easily performed with a minimum of apparatus. Examination can be conducted with a 10x handlens although a stereomicroscope is preferable.

One slight disadvantage of kerosene is that its reduced volatility increases the difficulty of drying the stone before inspection and removing the liquid from any crazing fractures, although this is only a minor inconvenience that can readily be bypassed, even by a rinse in a more volatile solvent such as acetone, or by a little more patience before inspection. **Appropriate fire hazard precautions should be observed.**

CONCLUSIONS

There appear to be unresolved anomalies in the thermal behaviour of some opaline materials, but thermal analysis behaviour and other properties including specific gravity, refractive index and reflectance appear not to correlate well with crazing susceptibility. However, total volatiles content was almost in the same sequence as crazing susceptibility in refluxing kerosene, and with practical experience.

Exposure of samples to elevated temperature induces crazing, depending upon exposure temperature and duration, and kerosene refluxing was considered to be by far the simplest and most convenient assessment technique, and requiring only simple equipment. The test should preferably be performed concurrently with appropriate reference material of predetermined susceptibility. The specific reasons for opal crazing were not clearly elucidated but certainly total volatiles content has a major bearing on crazing propensity. There remains considerable opportunity for more detailed investigation of thermal and chemical properties of this unique gemstone.

SUGGESTED TEST PROCEDURE

In the absence of more sophisticated chemical apparatus it is recommended that a pyrex test tube of about 20 mm diameter and not less than 150 mm long be used. A sample of about a carat or two of the material to be tested with fresh fracture faces, together with a similar sample of reference material is immersed in about 20 mm of kerosene which is gently boiled for twenty-five minutes over a flame, such as a small alcohol burner or even a candle, whilst being supported in an appropriate stand. The ring of refluxing vapor should not be allowed to extend more than about 50 mm above the liquid surface for safety, but to ensure active refluxing it should be not less than 10 mm above the liquid. It is suggested that the tube be plugged with cotton wool to prevent escape of flammable vapor.

The samples are removed from the kerosene when it has cooled somewhat and are allowed to dry prior to careful inspection for the presence of crazing under the stereomicroscope, or less preferably with a handlens. If crazing is detected the opal fails the test.

In view of the flammability of hot kerosene, appropriate fire precautions must be observed.

ACKNOWLEDGEMENTS

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ERRATA — Role of Water in Cracking of Opal. *Aust. Gemmol.* **15**, 12 p442.

A number of errors occurred in the printing of this article, and this is regreted. The most important concerns two figures in column 2, Volatiles content, in Table 2.

The figure for CPBF should read 7.60 + 0.02, and that for WCGG 6.96 + 0.1; these alterations are important to the conclusions drawn.

Ed.