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Thermal properties of Australian sedimentary opals and Czech moldavites

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Abstract The thermal properties are presented for a precious opal sourced from Coober Pedy, South Australia and a moldavite from Bohemia, Czech Republic whose origins differ significantly as opal is derived from the slow isothermal diagenesis of silica, while the tektites are specimens of vitreous silica formed from the terrestrial impact of asteroids. The differences between the two glassy silicates are presented through measurement of the TG–DSC, TMA and high-vacuum-hot-extraction DEGAS analysis.

Keywords Amorphous Silica · DEGAS · Moldavite · Opal · TG–DSC · TMA

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Introduction

Natural vitreous and diagenetic silicate glasses are found widely distributed on the earth and lunar surfaces. The vitreous glasses are formed from the melting and quenching of silicate rocks, while the diagenetic process is through the dissolution and precipitation of silica either through chemical, hydrothermal, or biotic means. Although both routes produce amorphous glassy substances, the mechanism of formation results in glasses with differing properties and in a range of quantities which can vary from micrograms through to the kilo tonne quantities of the "glassy mountains" [1]. A range of thermal histories is also observed. In the extremes, tektites like moldavites are formed by extremely fast heating and melting at very high temperatures (>3,000 K) followed by quenching at extreme cooling rates ($\geq 10^6$ K/s) [2]. In contrast, the formation of glasses through mineral diagenesis or biotic processes occurs at ambient temperatures; the formation processes are essentially isothermal and take place over long periods of time of the order of months to years or at extremely low cooling rates (K/year) [3]. The formation history of the natural glasses also results in a variety of microstructures. All of these glasses are amorphous and as such have no short range order, but under specific conditions, long range order has been observed in the diagenetic opaline glasses.

Although a variety of natural silicate glasses is available for characterisation and discussion, this article limits the discussion to two natural glasses, Moldavites from Bohemia in the Czech Republic and sedimentary opal from Coober Pedy in South Australia, as these glasses are found on the microgram to gram scale, are desired for their aesthetic qualities and are formed at the extremes of the temperature and time profiles.

Australian sedimentary opal

Precious or noble opal is prized for its play-of-colour (POC), the origins of which are based on the diffraction of visible light off ordered arrays of monodispersed silica spheres [4, 5]. Precious opal is an unusual material as it is composed of amorphous hydrous silica with the general formula $SiO_2 \cdot nH_2O$, but is precipitated as monodispersed colloidal spherical particles with diameters in the size range 150 to 400 nm. The monodispersed colloid is concentrated by sedimentation, evaporation or by filtration under pressure into ordered arrays that diffract visible light to produce the characteristic, prized POC.

The monodispersed silica colloid is formed through the dissolution and precipitation of silica. A likely source of the silica is from the weathering of silicates [2, 3]. In the weathering model, the source of the silica is the sedimentary rocks associated with the GAB where the chemical weathering of relatively soluble silicates such as the feldspars contained in these sediments results in the formation of an alkaline silica solution. An example of such a mineral is potassium feldspar which weathers through the idealised stoichiometry:

$$2KAlSi_3O_8 + 3H_2O \rightarrow Al_2Si_2O_5(OH)_4 + 2KOH + 4SiO_2$$

by the permeation of ground water through the sediments resulting in kaolinite, dissolved silica and an increase in pH through the release of potassium hydroxide. The reported trace element distributions in opal from a wide variety of sources are consistent with such a weathering model [6–8]. Once the silica is in solution, the enrichment of the solution by evaporation or filtration can occur. Increasing the concentration of the silica solution coupled with a lowering of the pH through alkali ion exchange with the surrounding clays allows the nucleation of primary silica spheres and subsequent sphere growth as more silica is supplied to the system. The supply of silica in the solution also appears to be a cyclic process as generations of growth rings are observed in the silica spheres. Once the monodispersed silica spheres have reached a suitable size, concentration of the colloid is then required and in certain cases, ‘crystallisation’ of the monodispersed colloid occurs to form the ordered arrays which results in the prized POC. The interstices are subsequently in-filled with a silica cement completing the formation of the opal producing a hard material which, despite the gel-like structure, has a Berkovich hardness of 5.7 to 6.2 GPa which is similar to that of soda glass (6.4 GPa), but, as might be expected for a gel like material, is significantly less than that of fused silica (10.8 GPa) [9].

Although the weathering model is important in aiding the understanding of the formation of opal, a number of other models have been proposed to account for specific observations in specimens acquired by the authors of these models. Examples of such models are the “microbial” model where microbes have been observed in Lightning Ridge matrix specimens suggesting that microbial action is responsible for the source of the silica [10], the syntechtonic fluid model where high pressure, warm hydraulic silica rich fluids are the silica source [11] and the mound spring model where the silica rich alkaline waters derived from the artesian basin well up through mound springs supplying the silica rich solutions required for opal formation [12]. Although these models source the silica from different origins, the formation of the monodispersed colloid must, however, in each case occur by homogenous precipitation followed by a concentration mechanism such as evaporation, filtration or sedimentation.

A sedimentation mechanism for the formation of opal does have some support [8, 13]. Elemental distributions in banded opal have suggested that although the bands must be formed from the same solution (e.g., constant aluminium concentrations across bands), the variation in ion concentration between bands of highly charged metal ions suggests that the bands form by flocculation of the silica colloid. Leisegang type phase separation (repeating colour rings) on precipitation of the silica may also explain the concentration variations of trace elements between bands [14].

The ‘sol–gel’ nature of the formation of opal is responsible for the hydrous gel-like structure of the silica and as a consequence opal contains a significant proportion of water much of which is reported to be molecular [15, 16]. Four types of water have been postulated: molecular water surface adsorbed (e.g. pore), bulk cage molecular water, surface silanol water and bulk silanol water [3]. Quantification of the amount of each species has proven to be difficult; however, Langer and Flörke [16] from their infrared spectroscopic studies suggested that approximately 90% of the water contained in sedimentary opal was present as molecular water which confirmed Segnet et al’s [15] postulation based on the calculation of surface area of the spheres that comprised POC opal. Further confirmation has been supplied by ^{29}Si NMR measurements where approximately 10 and 26% of the water was estimated to be in the form of silanol groups in Coober Pedy white POC opal and Tintenbar crystal opal, respectively [14, 17]. Thermal analysis has also demonstrated the presence of a significant proportion of molecular water in both volcanic and sedimentary opal [18].

Tektites and moldavites

Tektites are centimetre to decimetre-sized bottle green to blackish glassy “bodies”. *Tektite* is derived from the

Greek, *tektos* meaning molten and *tekein* to melt. They are found in gravels ranging in age from upper tertiary to alluvial in the Radomilice area, Chlum near the Moldau river in Moravia, Bohemia, Czech Republic (Moldavites), in Lusatia, Saxonia Germany, in Indochina, Vietnam, Thailand, Malayan Peninsula, Java, Flores, Billiton, Borneo, Philippines, Australia and Tasmania (Australites), in Zhamnanshin (Irghizites) Kazakhstan, Ouellée on the Ivory Coast in Africa, Texas and Georgia in the United States (Bediasites). Microtektites (vitreous/crystalline spherules <500 μm) have been found in deep-sea deposits in the Gulf of Mexico, Caribbean Sea, north-western Atlantic Ocean and on Barbados. The chemical composition of microtektites is closely associated with the North American tektites. Transparent colourless to pale brown microtektites are also found associated with clinopyroxene spherules in the western Pacific and Indian Ocean in the upper Eocene marine deposits [19].

The origin of tektites was intensively studied and discussed in the 20th century, particularly in the latter decades, with respect to the mass extinction during the short time event known as the Cretaceous–Tertiary boundary. The discussion was dominated by two alternative origins of tektite material: the Terrestrial Impact Theory (TIT) and the Lunar Volcanic Theory (LVT) [20, 21]). As Izokh [20] noted the TIT "won a complete victory over O'Keefe's [21] Lunar Volcanic Theory" and, currently, the majority of scientific community discusses the origin of tektites and mass extinction event on the basis of TIT as, in particular, the chemical evidence supports the formation of tektites from terrestrial rocks and soils [22].

The terrestrial impact theory is based on the significant transfer of energy from impacting asteroids with the earth's surface. As asteroids are massive, impact with the earth surface produces high-energy collisions and high pressures and temperatures in the impact zone which results in the fusion and vaporisation of the material around the impact zone. The energy of these impacts was such that fused and vaporised material was catapulted out to near space. As this material returned to earth, it rapidly cooled and solidified with an absence of gaseous inclusions (although vacuous inclusions are observed). During the quenching process the solidifying phase was generally shaped aerodynamically and as the molten droplets were spinning, a range of bizarre shapes; plate, spheroid, rod, dumbbell, teardrop, star, curly, winkled and bubble, were produced. As these particles were returned to earth, the particles were distributed by aerodynamic transport over large areas on the Earth-surface in strewn fields. The tektites found in particular strewn fields are postulated to have been derived from the same impact event due to the high degree of compositional homogeneity within an individual strewn field [23].

The impact-formed or metamorphic glasses have a significant durability. The age of impact-formed glasses at the earth's surface has been determined to be between 700,000 years (australites) and 34 million years (bediasites). In a more inert environment such as at the lunar surface, glass spherules have been aged at more than 2 billion years. In general, recovered specimens of tektite have been observed to be resistant to hydration or devitrification of the bulk [24]. Only a characteristic corrosion of the surface is observed at the Bohemian tektites—the moldavites [25].

Experimental

The opal samples investigated were sourced from the Shell Patch field in Coober Pedy, South Australia. The moldavite samples were sourced from Bohemia in the Czech Republic.

Thermomechanical analysis (TMA) was carried out on rectangular logs of opal (5 × 5 × 10 mm) and moldavite (2 × 3 × 5 mm) using a TA Instruments 2940 Thermomechanical Analyser by heating the specimens to 960 °C at a heating rate of 1 °C/min in a static air atmosphere followed by cooling at 5 °C/min to room temperature. The cycle was then repeated for each sample.

Density measurements were carried out on the TMA specimens before and after the heating cycles using the Archimedes' principle; $\rho_{\text{sample}} = (\rho_{\text{liquid}} \times \text{dry weight})/(\text{dry weight} - \text{immersed weight})$. The immersed weight was determined by immersion in analytical grade hexane. The density of the hexane was measured using a pycnometer of known volume.

A direct-coupled-evolved-gas-analysis-system (DEGAS) was used to carry out a high-vacuum-hot-extraction of the evolved gases with gas analysis carried out using a quadrupol mass spectrometer [26, 27]. Pieces of opal and moldavite of the order of 100 mg were heated at a rate of 10 °C/min under vacuum (10^{-4} Pa) to 1,400 °C, while the release of water (m/z = 18 amu), hydrogen (m/z = 2 amu) and methane (m/z = 13 and 15 amu) were monitored.

Simultaneous thermogravimetric analysis (TG)–differential scanning calorimetry (DSC) was carried out on a Netzsch STA 449 C Jupiter by heating 40–60 mg of sample in a platinum crucible at 20 °C/min in a synthetic air atmosphere (N_2:O_2 80:20) at a flow rate of 70 mL/min to 1,640 °C.

Results

Thermogravimetric analysis–differential scanning calorimetry (TG–DSC)

Simultaneous TG–DSC data for the opal and the moldavite specimens are shown in Fig. 1. The measurements are

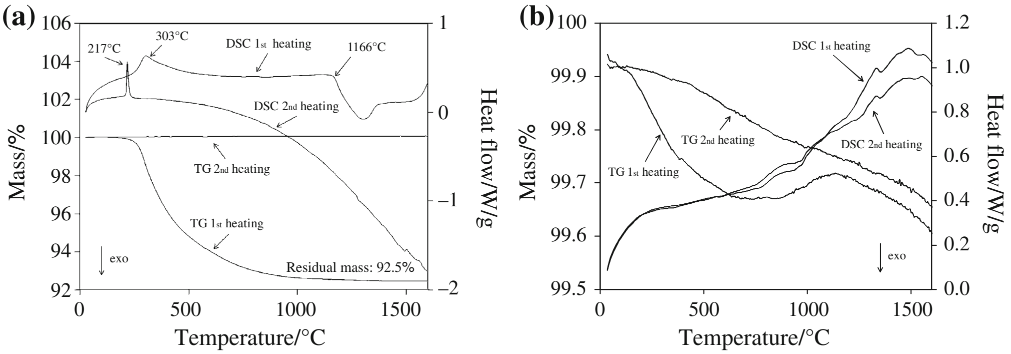

Fig. 1 TG and DSC data of the CP white play-of colour opal (**a**) and Bohemian moldavite (**b**) heated to 1640 °C at 20 K/min in an air atmosphere

characteristic of the origins of these specimens. As the opal is formed though a solution-precipitation process, a significant amount of water (7.5% by weight) is contained within the specimen which is removed between 200 and 1,000 °C, with peak rate of loss from the DTG curve (not shown) at 297 °C. The second heating curve shows, as expected, no mass loss. As the Moldavite was formed through a process of rapid melting to high temperature followed by rapid cooling, no mass loss was expected. A small mass loss of 0.39 and 0.34% in the 1st and 2nd heating runs, respectively, was, however, observed (Fig. 1b). As this small mass loss was reproduced in both heating runs, the mass loss was ascribed to the removal of gases adsorbed at low temperature which diffuse into the pore structure and, hence, are removed slowly over the period of the heating.

The 1st heating run DSC curve for the opal (Fig. 1a) shows an endotherm at 303 °C which corresponds to the maximum water loss. At elevated temperature, an exothermic peak around 1,287 °C with an onset at 1,166 °C is observed. Reheating the specimen in the 2nd heating run reveals an endothermic peak with an onset temperature of 211 °C and a peak temperature of 217 °C that corresponds to the α- to β-cristobalite transition. The exotherm above 1,166 °C in the 1st heating curve can, therefore, be ascribed to crystallisation of the opal. The DSC curves for the moldavite are essentially featureless showing that even

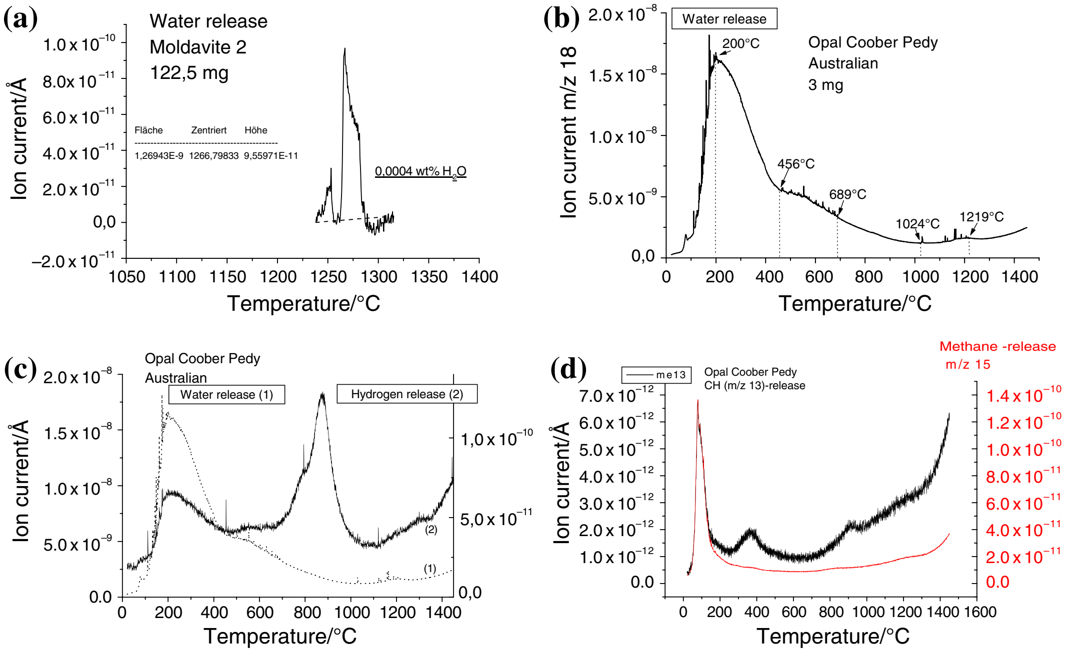

Fig. 2 DEGAS data for **a** the water evolution from a moldavite specimen and **b** the water, **c** the hydrogen and **d** the methane evolution from a Coober Pedy white POC opal

heating to 1,640 °C had no effect on the morphology of the specimen which is likely to be due to the relatively high alumina content of moldavites (10.3% [23]).

High-vacuum-hot-extraction DEGAS

The DEGAS data is displayed in Fig. 2. Negligible gas evolution was observed from the moldavite (Fig. 2a) with only 0.0004% mass loss due to water evolved above 1,200 °C. The mass loss ascribed to the adsorbed gases under atmospheric conditions observed in the TG data are not present as this technique is carried out under vacuum thus removing any adsorbed gasses prior to elevation of the temperature.

The thermal extraction of gases from the opal, however, is much more eventful. Figure 2b shows the DEGAS curve for water which corresponds to 99.6% of the total mass loss. The evolution of water is observed as a main peak around 200 °C which is lower than that observed in the TG data. The DEGAS is carried out under the reduced pressure which shifts the peak to lower temperature. A shoulder to the main evolution peak is also observed between 400 and 700 °C accounting for 0.3% of the water loss and continued evolution of water is observed up to and beyond 1,000 °C accounting for 0.1% of the water loss and is consistent with the TG data. It is also notable that the spikes in the data are observed. These spikes correspond to sudden losses which may be ascribed to sudden release from fluid inclusions or entrapped micropores in which the pressure escalates to beyond the breaking stress of the opal.

Figure 2c and d show the DEGAS curves for hydrogen and methane, respectively. It should be noted that the combined evolution of these species corresponds to less than 0.4% of the total mass loss. The hydrogen released around 880 °C is likely to be derived from micellar decomposition of silanols [28], but the methane evolved around 100 °C is a little more difficult to explain as the temperatures are too low for the micellar reactions inferred for the evolution of hydrogen. It is possible that very small proportions of molecular methane are trapped in the gel structure as the opal network is formed.

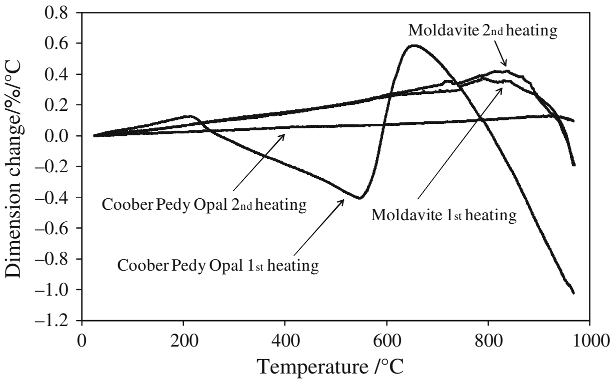

Fig. 3 TMA data for 1st and 2nd heating runs on a moldavite specimen and a Coober Pedy

Thermomechanical analysis (TMA)

The TMA data for the opal and moldavite is shown in Fig. 3. The data for opal is characteristic of a hydrated silica where, in the 1st heating, significant expansion occurs up to 210 °C. Above 210 °C, contraction occurs which is associated with the densification of the silica network on removal of the water as well as sintering of the structure [18, 29]. At 540 °C, the onset of a second expansion is observed for this sample of opal and is ascribed to a ‘blowing’ of the structure due to the increased pressure of water contained within the structure. It is also possible that this second expansion can be ascribed to slow cracking of the opal which can occur and was observed in the specimen after removal from the furnace. The expansion is, however, reproducible for samples which undergo this second expansion although it should be noted that not all samples of opal undergo this type of expansion; many samples sourced from different fields in Coober Pedy or in other regions do not undergo this type of expansion [18]. Further heating results in contraction as further sintering and densification of the structure occurs [29]. The second heating of the opal produces a linear expansion curve that might be expected of a dehydrated specimen. The moldavite specimens produced reproducible expansion curves each time a specimen was heated suggesting that heating the moldavite to 960 °C did not significantly affect its structure which is in keeping with the TG data and reported expansion measurements of tektites [2].

The expansion data for the opal and moldavite specimens are listed in Table 1. Data is listed so that comparison can be made between the samples in the linear regions of the expansion curves. Not surprisingly, the greatest

Table 1 Expansion Coefficients determined for heating and cooling curves using TMA for Coober Pedy POC opal and Czech Moldavite

Sample	Heating 100–200 °C ($\times10^{-6}$/°C)	Heating 300–500 °C ($\times10^{-6}$/°C)
Coober Pedy 1st heating	+7.4	−13.6
Coober Pedy 2nd heating	+1.4	+1.3
Moldavite 1st heating	+3.7	+4.6
Moldavite 2nd heating	+4.3	+4.1

Table 2 Density measurements for Czech moldavite and Coober Pedy POC opal before and after heating to 960 °C in the TMA

Sample	Density (g/cm^3)
Coober Pedy Opal before heating	2.109
Coober Pedy Opal after heating to 960 °C	2.115
Moldavite before heating	2.375
Moldavite after heating to 960 °C	2.354

expansion coefficient is observed for the opal on 1st heating and is consistent with the water exerting hydrostatic pressure on the opal causing significant expansion. However, once dehydrated, the opal specimen has the lowest expansion coefficient. Although opal contains approximately 99% SiO_2 on an anhydrous basis, the expansion coefficient for dehydrated opal remains significantly greater than that of fused silica ($0.5 \times 10^{-6}/^{o}C$) [2, 24]. The expansion coefficient for moldavite is greater than that for dehydrated opal, but is less than that for obsidian ($6.3 \times 10^{-6} K^{-1}$) and soda lime glass ($7.5 \times 10^{-6} K^{-1}$) [2, 24]. The thermal expansion coefficient for moldavite is most aptly compared to that of obsidian as both minerals have a similar rhyolitic composition. The difference in the expansion coefficient is most likely due to the relative proportions of sodium and potassium with respect to calcium and magnesium within the silica network. Opal has approximately 0.5% alkali and alkali earth metals as part of its composition thus raising the expansion coefficient to above that for fused silica. Moldavite contains significantly greater concentrations of the alkali and alkali earths and thus the expansion coefficient is greater than that of the dehydrated opal. Although moldavites and obsidian have a similar rhyolitic composition, moldavites have a greater concentration of CaO and MgO and lower Na_2O content than obsidian which results in a more tightly bound structure and, hence, a lower expansion coefficient [2].

The densities for the TMA specimens before and after the cycling are listed in Table 2. The density of the opal increases, as might be expected, as sintering of the specimen occurs at elevated temperature [18, 29, 30]. This change in density is, however, small and indicates that the structure has not significantly changed during heating to 960 °C. The small change in density is also indicative of the nature of the water in opal; that is, molecular water trapped in silica cages. The change in density of the moldavites is, however, somewhat surprising as a reduction in the density after heating in the TMA is observed. This is in contravention to published data [2]. It is likely that this reduction in density is due to the thermal history as the samples investigated were twice heated to 960 °C at 1 °C/min and cooled under controlled conditions at a cooling rate of 5 °C/min allowing relaxation of the glass to a marginally more open structure.

Discussion

Opal and moldavite were selected for comparison in this study as their formation histories are at the extremes. The extreme conditions under which the moldavites are produced results in a vitreous glass which is completely degassed and, therefore, shows no mass loss either in the TG or in the DEGAS experiments where little evolution of volatiles was observed. The opal, by contrast, is a solution precipitated glassy substance and this sample has a water content of 7.5% (some variability in the water contents in opal are observed and depends on the field within a region as well as the level (depth) within a mine). The predominant mass loss for this sample is associated with the water loss (99.6%). Hydrogen was observed to be evolved at elevated temperature but this was ascribed to cage decomposition of the silanol groups. Methane was also observed and was observed to evolve at low temperature suggesting that molecules of methane are trapped in silica cages. For the methane to be trapped in the silica cages, the methane must have been present in the silica solution as the opal was formed. The origin of the methane is unclear, but it may have originated from the artesian waters of the Great Artesian Basin or from meteoric waters.

Expansion coefficients for the as received samples are consistent with the relative contents of volatiles (water) present in the opal and the moldavite. The moldavite is unaffected by the heating to 960 °C except for a small decrease in density. The opal is dehydrated and although cracking occurs during the first heating, subsequent heating cycles produces linear and reproducible expansion curves with coefficients of expansion less than that of moldavite, but greater than that of fused silica which is consistent with the relative elemental compositions. The small increase in density after the heating cycle indicates that the silica structure in the opal is also resistant to rearrangement below 1,000 °C. Above 1,166 °C, however, crystallisation to cristobalite is observed whereas the moldavite is apparently unaffected by heating to 1,640 °C. These differences may be attributed to the differences in the alumina content which is of the order of 1% for the opal and of the order of 10% for the moldavites.

Final remarks

Opals and moldavites are rare natural non-crystalline solids, whose primary value is aesthetic. Both are important both in their as found shapes: in the case of moldavite the aerodynamic forms and colour and in the case of opal the infilling of voids, seams, cracks and in fossil replacement and both are valued as mineral gems for jewellery.

Both of these natural minerals have an important part to play in the understanding of geological processes as their formation is a record of change in the natural environment. The tektites, based on their models of formation, have given an insight into the impact processes that have remodelled the Earth's fauna and flora. The opal is the first step to understanding the diagenesis of silica and its mobility. Even though both of these non-crystalline solids are formed at the extremes of forming processes, both tektites and opals help to enrich our lives.

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