

RAMAN SPECTROSCOPY ON GEM-QUALITY MICROCRYSTALLINE AND AMORPHOUS SILICA VARIETIES FROM ROMANIA

DANA POP¹, CIPRIAN CONSTANTINA¹, DAN TĂTAR² & WOLFGANG KIEFER²

ABSTRACT. Previous micro-Raman investigations on a wide range of types of microcrystalline silica samples have evidenced the presence of a new silica polymorph, *i.e.* moganite, in virtually every sample. In order to test the ubiquity of moganite in such geological materials in Romania, micro-Raman measurements were performed on gem-quality microcrystalline quartz (chalcedony, including agate and jasper) and additionally on quasi-amorphous silica (opal, silicified wood) samples from different occurrences.

All the studied materials, including the quasi-amorphous varieties contain moganite in variable amounts (from traces up to local concentrations of 75 wt. %). Establishing correlations between colour and texture of the chemically-“pure” microcrystalline silica samples and their mineralogical composition - as traced by micro-Raman measurements, requires further research.

Key words: Raman spectroscopy, microcrystalline quartz, opal, moganite, Romania

INTRODUCTION

Raman spectroscopy is used as a powerful tool for mineral phase identification in geological samples, and for characterising the crystal chemistry of heterogeneous materials. A special case is represented by mineral polymorphs, such as the various SiO₂ phases. Previous micro-Raman investigations on a wide range of types of microcrystalline silica samples (Kingma & Hemley, 1994) have evidenced the presence of a tetrahedrally coordinated silica polymorph - moganite, in virtually every sample.

The main mineral component of microcrystalline SiO₂ („chalcedony“, „jasper“, „chert“, or „flint“) is low-, or *a*-quartz. Based on X-ray diffraction, Flörke et al. (1976) have evidenced a new silica polymorph, “moganite” (type locality: Mogan, Gran Canaria, Spain) that often forms an intimately intergrowth with *a*- quartz in many microcrystalline SiO₂ varieties. Moganite was approved as a new mineral by IMA in 1999 (Grice & Ferraris, 2000).

Moganite has an AB₂ structure type which is closely related to quartz. It typically contains up to 3 wt. % of water which is not a constituent of the structure. Moganite is monoclinic with the cell parameters $a = 8.75$, $b = 4.87$, $c = 10.71$, $\beta = 90.09^\circ$, space group $I2/a$, $Z(\text{SiO}_2) = 12$, $D = 2.55 \text{ g/cm}^3$. The structural principle is a periodic twinning according to the quartz Brazil-law on a cell dimension scale (Miehe & Graetsch, 1992).

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Conventional X-ray diffraction (XRD) (long-range order) is constrained in identifying moganite, due to the nano-range size of moganite crystallites and its close structural relationship - thus strong similarities in the XRD pattern - to α -quartz, to which it is usually associated. However, micro-Raman spectroscopy (short-range order) proved to be the perfect tool for detecting the presence of moganite (Kingma & Hemley, 1994) and for evaluating the relative moganite-to-quartz ratio (Götze et al., 1998).

Kingma & Hemley (1994) have identified 19 distinct bands in the moganite Raman spectrum. The corresponding wavenumbers are (in cm^{-1}) 129, 141, 220, 265, 317, 370, 377, 398, 432, 449, 463, 501, 693, 792, 833, 1058, 1084, 1171, 1177. The strongest band is located around 501 cm^{-1} and, according to Götze et al. (1998), it represents the main symmetric stretching-bending mode of moganite.

MATERIALS AND EXPERIMENTAL TECHNIQUE

In order to test the ubiquity of moganite in Romanian gem-quality micro-crystalline and amorphous SiO_2 varieties, and for checking if a relationship between the colour/textural variations vs. the presence of specific mineral phases could be established, micro-Raman measurements were performed on 17 samples from five occurrences (Table 1). Their assignment to the IMA approved species is as follows: 14 quartz (microcrystalline, *i.e.* chalcedony - including agate and jasper varieties) and 3 opal (including silicified wood variety) samples.

The best studied occurrence was Gurasada (Apuseni Mountains), due to the remarkable diversity of gem-quality SiO_2 varieties found in Paleocene („banatitic“) pyroclastic agglomerates and tuffs: chalcedony (including agate and jasper) and opal (including silicified wood). Most of the samples show macroscopic and/or microscopic variations in colour (grey, red, blue, white, and green), transparency or texture (from massive to banded). Other chalcedonies from the Apuseni Mts. originate from Rachiş (from Cretaceous „ophiolitic“ pyroclastic rocks) and Techereu (from Paleocene rhyolites). Famous sites from Baia Mare area such as Trestia (chalcedony), and Oraşu Nou (opal) - related to the Neogene volcanism, were included for comparison.

The measurements were performed on a Dilor Labram system equipped with an Olympus LMPlan FI 50 microscope objective with a numerical aperture of 0.50, gratings 1,800 grooves/mm and an external laser with an emission wavelength of Ar^+ 514.5 nm; additionally other lasers with the following emission wavelengths He-Ne 632.8 nm were used. In the recording of the micro-Raman spectra a laser output power of about 100 mW has been employed and the measured energy after the objective was about 15 mW for the 514.5 nm laser line. The spectra obtained with the 514.5 nm excitation were used for further interpretation. In this case, the calculated lateral resolution was $0.62 \mu\text{m}$, and the spectral resolution was about 4 cm^{-1} . The measurement time was 10 s.

For each sample, several measurements were performed on a micro-scale in various areas, their number (between 10-30 spots) depending on the observed microscopic heterogeneity of the material. This allowed the local (punctual) investigation of microcrystalline and quasi-amorphous silica samples. No effect of crystal orientation with respect to the laser beam polarisation has been observed.

The identification of the mineral phases was done by the fingerprint method, using reference spectra (from free Raman databases available on the internet). The distinctive peak (or sometimes „shoulder“ of the 465 cm^{-1} quartz band, Kingma & Hemley, 1994) which is noticeable at values around 500 cm^{-1} was interpreted as an evidence for the presence of moganite intergrown with α - quartz.

RESULTS AND DISCUSSION

Raman-based characterisation of the studied silica varieties

Based on the features obtained in the Raman spectra, and for allowing a handy comparison of the large number of measurements, the studied samples were grouped into (chemically-) „homogeneous“ (or „pure“) and respectively „heterogeneous“, the terms being related to the absence/presence of other mineral phases than SiO_2 in the corresponding materials. Also, the spectra were grouped according to the main SiO_2 phase present in the sample into microcrystalline and quasi-amorphous.

In the case of homogeneous microcrystalline quartz samples (Fig. 1), one representative measured data set was selected and plotted in each case. For heterogeneous materials, one measurement was plotted for each type of phase/mixture of phases.

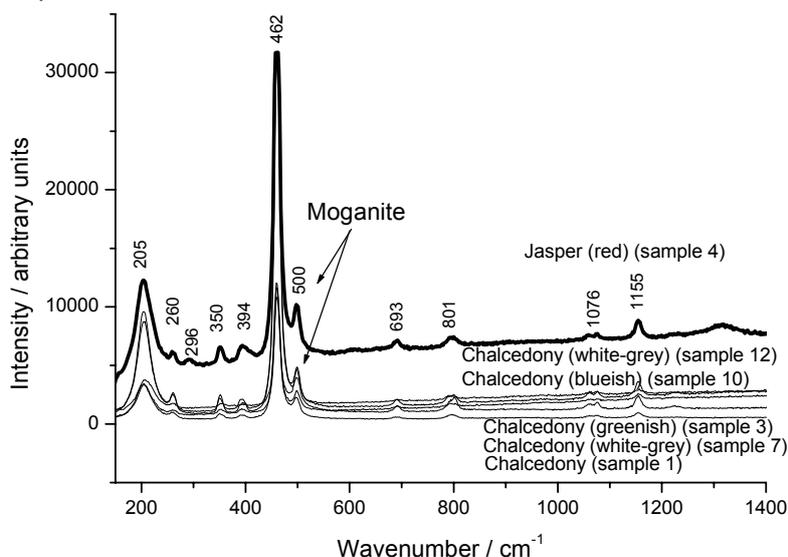


Fig. 1. Selected Raman spectra – chalcedonies and jasper from Gurasada.

Homogeneous microcrystalline quartz

Most of the chalcedonies (samples 1, 3, 7, 10 and 12: Fig. 1, and sample 6) and one jasper (sample 4: Fig. 1) from Gurasada, as well as the chalcedonies from Rachiş (sample 14), Trestia (sample 16) and Techereu (sample 17) show a bulk composition represented by α -quartz intergrown with variable amounts of moganite. No correlation has been found between the colour of the gem material and the local mineralogical composition.

Homogeneous quasi-amorphous silica

The sample from Oraşu Nou (sample 15) mineralogically consists almost exclusively of opal CT; locally, minor amounts of moganite were detected, intimately mixed with opal CT (Fig. 2). The increasing intensities of the main opal band (around 331 cm^{-1}) may suggest increasing degrees of short-range local structural ordering.

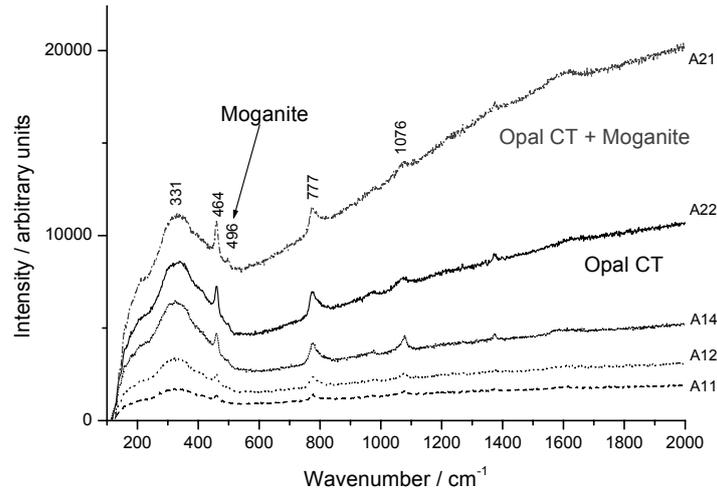


Fig. 2. Selected Raman spectra – opal from Oraşu Nou (sample 15).

The main, amorphous, phase in sample 2 from Gurasada is also defined as opal CT (Fig. 3). A full range of transitions from opal CT to α -quartz is evidenced by the set of selected spectra. Moganite occurs in the crystallized areas of the sample (where opal may be considered as absent), intergrown with α -quartz.

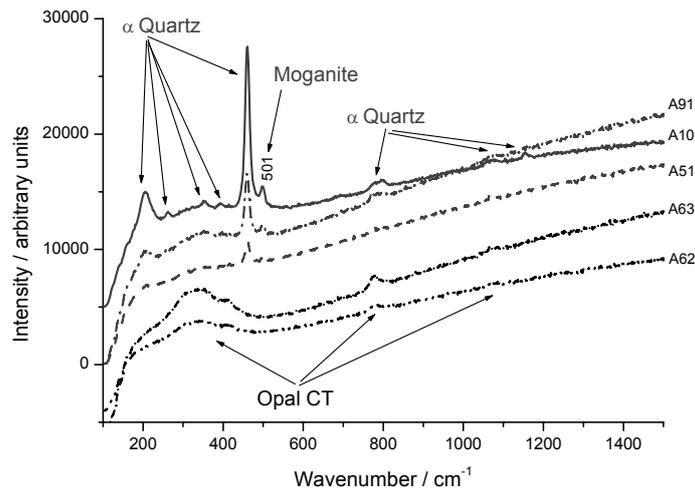


Fig. 3. Selected Raman spectra – opal from Gurasada (sample 2).

Heterogeneous microcrystalline quartz

The red-white-grey agate from Gurasada (sample 11) consists of individual bands with distinctive mineralogical composition: (α -quartz + moganite) vs. “pure” microcrystalline graphite (disordered carbon) (Fig. 4). Graphite shows broad *D* (~ 1360 cm^{-1}) and *G+D'* (~1576 cm^{-1}) bands indicating strong structural disorder, or an almost amorphous structure (Wang et al., 1989 in Nasdala et al., 2004).

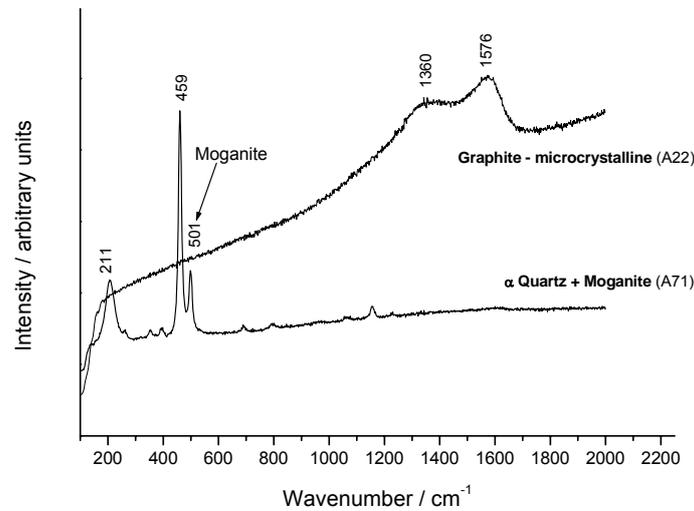


Fig. 4. Selected Raman spectra – agate from Gurasada (sample 11).

One of the jaspers from Gurasada (sample 9) is characterized by the presence of calcite, locally as an individual phase, or in mixtures with microcrystalline graphite, or α -quartz + moganite, or both (Fig. 5).

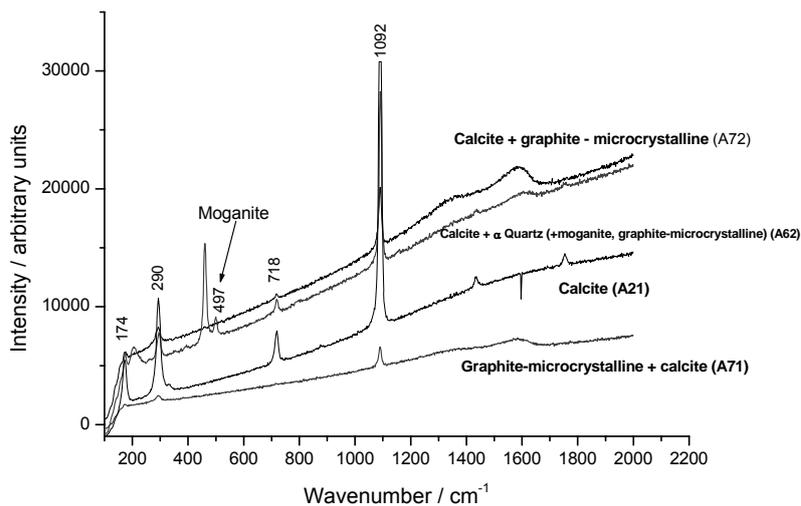


Fig. 5. Selected Raman spectra – jasper from Gurasada (sample 9).

One white chalcedony from Gurasada (sample 5) consists of clearly distinctive areas built-up of either a mixture of α -quartz + moganite, or calcite, respectively.

The red and white chalcedony from Rachiş (sample 13) consists of α -quartz (+ minor moganite) (Fig. 6); locally α -quartz is intergrown with microcrystalline graphite and an unidentified phase(s) evidenced by the bands around 300 and 550 cm^{-1} , possibly responsible for the red colour of the gem material.

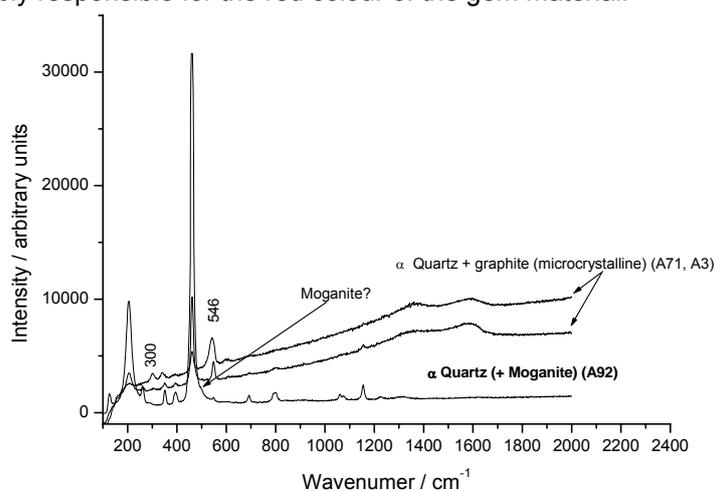


Fig. 6. Selected Raman spectra – chalcedony (red and white) from Rachiş (sample 13).

Heterogeneous quasi-amorphous silica

The silicified wood from Gurasada (sample 8) shows a very complex mineralogical composition: areas of opal (CT?) with minor amounts of α -quartz (+ traces of moganite and an unidentified phase with a band around 1000 cm^{-1}) alternate with areas consisting of α -quartz + microcrystalline graphite, and respectively “pure” microcrystalline graphite (Fig. 7).

Raman-based evaluation of the moganite-to-quartz ratio

As shown previously, all the studied materials, including the quasi-amorphous varieties locally contain moganite, in variable amounts. For a quantitative evaluation of the moganite content in the studied samples, the relative Raman calibration method proposed by Götze et al. (1998) was applied. Their model assumes that α -quartz and moganite are the only phases in the sample, and uses the α -quartz component as an internal standard. For this reason, our quantitative evaluation could be performed only in the case of microcrystalline quartz samples where non-silica phases (impurities) were absent. One exception is represented by the opal from Gurasada (sample 2), where locally moganite occurs mixed with α -quartz, in the absence of an opal phase. In the case of microcrystalline silica samples containing only minor amounts of moganite (samples 6 and 14) the fitting procedure yielded non-realistic results, thus they were also excluded from the evaluation.

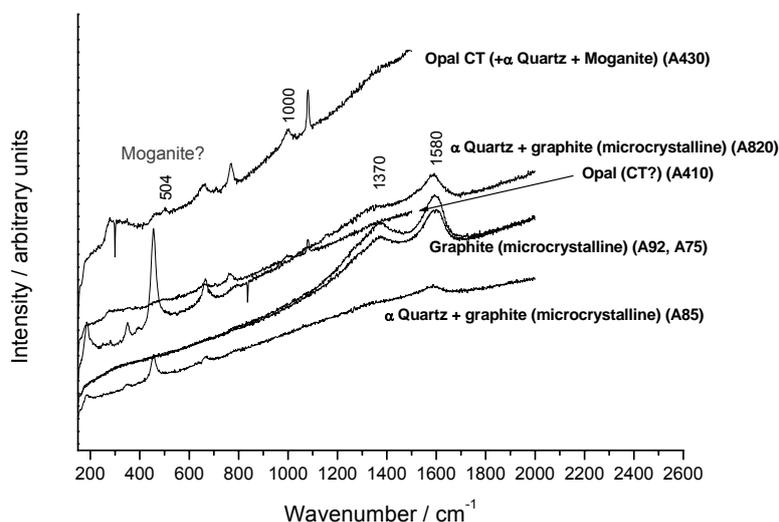


Fig. 7. Selected Raman spectra – silicified wood from Gurasada (sample 8).

The calibration curve (Fig. 3 in Götze et al., 1998) provides an estimate of the moganite content (wt. %) based on the peak integral ratio (%), where the latter is defined as the ratio of the integrals (I) of the main Raman bands of moganite ($\sim 502 \text{ cm}^{-1}$) and α -quartz ($\sim 465 \text{ cm}^{-1}$) respectively. The band integrals are calculated from the measured band heights (measured above the background) \times the corrected half-width (measured as full width at half-height), assuming Lorentzian-shaped Raman bands. The correction of the half-width (FWHM) for the apparatus function was done in accordance with Imer, 1985 (in Götze et al., 1998). The calibration curve was worked out on texture-free powders. On pieces of crushed samples (as in our case) it is recommended to use unpolarised light and microscope objectives with large numerical apertures.

The quantitative evaluation on the samples under study (Table 1) was performed taking into account the following:

- the experimental conditions were similar to those used for the calibration model (Götze et al, 1998): Ar^+ excitation (514.5 nm), unpolarised light (unoriented analyses), a similar numerical aperture (0.50 as compared to 0.55), and a similar spectral resolution (4 cm^{-1} as compared to 3 cm^{-1});
- the same experimental conditions were used for the acquisition of each data set;
- no effect of crystal orientation with respect to the laser beam polarisation has been observed;
- in the case of each sample, the spectra showing the less significant increasing background were preferred. However most of the spectra show an almost horizontal (linear) background in the $400\text{-}600 \text{ cm}^{-1}$ interval, where the main bands of α -quartz and moganite are located. According to Götze et al. (1998), even if the spectra show an increasing Raman background with increasing moganite content, this does not influence the calibration;

- an automatic two-peaks Lorentzian fitting procedure was applied in the above-mentioned interval, which included a linear baseline subtraction. Only fits with $R^2 > 0.98$ were taken into consideration (Fig. 8). No additional mathematical procedure was applied for background correction, in order to avoid introducing potential artefacts in the quantitative estimation;

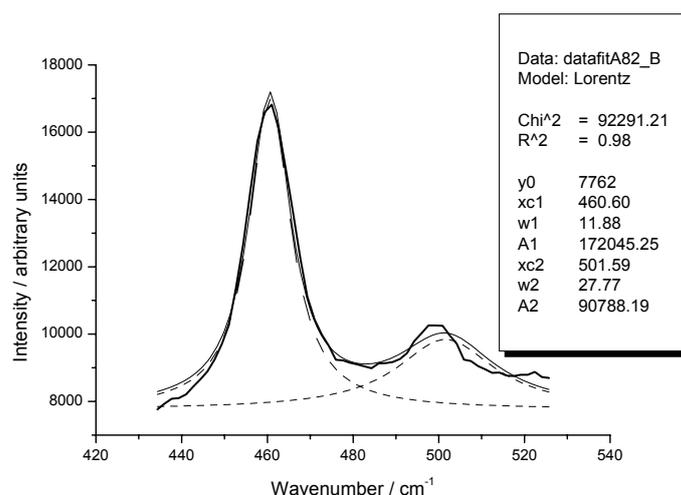


Fig. 8. Lorentzian fit for the main Raman bands of α -quartz ($\sim 465 \text{ cm}^{-1}$) and moganite ($\sim 502 \text{ cm}^{-1}$); (sample 5, measured spot A82).

- the intensities of the fitted peaks were calculated as the difference of the maximum y value of each peak read from the plot and the background (y_0) indicated by the automatic fitting procedure. For taking into account the potential errors related to the maximum y values, two readings were performed for each data set. The same calculation procedure (see below) was applied for each of these values. The automatically-calculated values for the half-widths (w_1 and w_2) were considered as the measured FWHM (Fig. 8);

- in order to deduce the real (*i.e.* corrected) FWHM, the correction for the apparatus function was applied according to the equation of Irmer (1985), considering the spectral resolution (s) of 4 cm^{-1} . As mentioned in Nasdala et al. (2001), the mathematical correction yields reliable real FWHM (b) values only if $b \geq 2s$, condition which is fulfilled in all the cases;

- the Raman band integral ratios $I_{(502)}/I_{(465)}$ (%) were calculated following the mathematical procedure in Götze et al. (1998). The error for the Raman band integral ratios in the case of the same data set (measured spot), calculated for the two maximum intensity values (see above) was smaller than $\pm 10 \%$;

- the moganite content (wt. %) was estimated based on the calibration curve proposed by Götze et al. (1998) (Fig. 9). The error of the calculation procedure (related to the maximum intensity values) was evaluated as follows: two estimations were made for each measured spot, based on the two corresponding Raman band integral ratios. The moganite content for the same data set showed a relative variation smaller than $\pm 5 \%$.

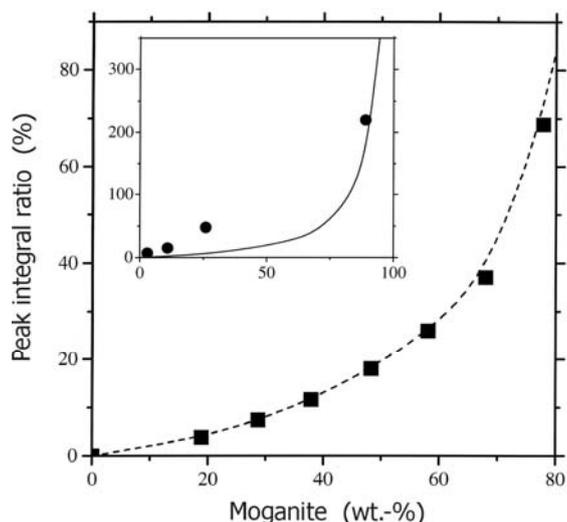


Fig. 9. Raman moganite-to-quartz ratio calibration curve (slightly modified from Götze et al., 1998). Copyright © Springer-Verlag Berlin Heidelberg.

The results are presented in Table 1. The overall moganite content in the studied samples suitable for the quantitative estimation ranges from traces (minor moganite) up to local concentrations of 75 wt. %. For the same sample, the quantitative evaluation performed on various measured spots shows variations of the moganite content between 5 - 30 wt. % (Table 1). It is common for moganite to show variations of content in the same sample, at a micro-range scale (Kingma & Hemley, 1994; Götze et al., 1998).

Geological significance of moganite in microcrystalline quartz

Maturation of cryptocrystalline SiO₂ phases (the transformation of moganite into α -quartz) produces a general decrease in the moganite content (Moxon & Rios, 2004). Thus, moganite content was proposed to be used as a tool for age evaluation.

In the studied samples, it can be noticed that the oldest chalcedonies - samples 13 and 14 from Rachiş (Cretaceous), show minor moganite contents (traces). The youngest one, sample 16 from Trestia (Neogene), revealed relatively high moganite amounts (60 - 75 wt. %). In the same time, the highest local moganite content was estimated in one of the chalcedonies (sample 5) from Gurasada (70 - 75 wt. %), of Paleocene age. However, the samples from Gurasada area are compositionally and structurally very diverse and show a wide range of moganite contents (from traces up to 75 wt. %), while from the other occurrences the number of studied samples is too small to allow a consistent discussion.

Enhanced moganite concentration (> 20 wt. %) in microcrystalline silica may suggest an evaporitic genetic setting. Moganite crystallisation is favoured by alkaline fluid chemistry combined with high activities of ferric iron. The absence of moganite in weathered and hydrothermally-altered silica samples may be a useful measure of fluid-rock interactions (Heaney, 1995).

Based on the current evaluation of the moganite content in the Romanian microcrystalline silica samples (Table 1), an evaporitic genetic setting may be assumed for all the studied occurrences, except for Rachiş (samples 13, 14).

Table 1

Raman bands integral ratios and the estimated moganite content [wt. %] in the studied samples.

No.	Material	Colour	Occurrence	Measured spot			Raman band integral ratio I(502)/I(465) [%]						Moganite [wt. %]	Observations
				A12	A17	A41	28	33	18	58	64	48		
1	Chalcedony	grey	Gurasada	A12	A17	A41	28	33	18	58	64	48	local moganite (wt. %): ~50 - 65	
2	Opal	grey-blue	Gurasada	A81	A101	A82	29	29	10	60	60	34	local moganite (wt. %): ~35 - 60	
3	Chalcedony	grey-green	Gurasada	A7	A31		36	22		65	53		local moganite (wt. %): ~50 - 65	
4	Jasper	red	Gurasada	A61	A22	A11	14	17	12	41	46	38	local moganite (wt. %): ~40 - 55	
5	Chalcedony	white	Gurasada	A61	A52	A82	62	40	63	75	68	76	local moganite (wt. %): ~70 - 75	
6	Chalcedony	white	Gurasada										minor moganite	
7	Chalcedony	white-grey	Gurasada	A15	A510	A610	40	16	54	68	44	73	local moganite (wt. %): ~45 - 75	
8	Silicified wood	brown	Gurasada										minor moganite + mixture with graphite	
9	Jasper	red	Gurasada										mixture with calcite, graphite	
10	Chalcedony	white-blue	Gurasada	A11	A41	A61	20	18	34	50	48	64	local moganite (wt. %): ~50 - 65	
11	Agate	red-white-grey	Gurasada	A71	A81	A82	28	39	38	58	66	66	local moganite (wt. %): ~60 - 65	
12	Chalcedony	white-grey	Gurasada	A51	A61	A21	33	26	27	64	58	58	local moganite (wt. %): ~60 - 65	
13	Chalcedony	white-red	Rachiş										minor moganite + mixture with graphite	
14	Chalcedony	milky-white	Rachiş										minor moganite	
15	Opal	bluish-grey	Oraşu Nou										minor moganite + opal (no α -quartz)	
16	Chalcedony	blue	Trestia	A31	A13	A21	37	30	34	66	61	64	local moganite (wt. %): ~60 - 65	
17	Chalcedony	blue	Techereu	A21	A11	A31	7	10	6	29	34	26	local moganite (wt. %): ~30 - 35	

Error evaluation: Raman band integral ratio for the same data set (measured spot) [%]: relative variation $< \pm 10$ %;
Moganite content for the same data set (measured spot) [wt. %]: relative variation $< \pm 5$ %

CONCLUSIONS

1. All the studied Romanian gem-quality SiO₂ varieties, including the quasi-amorphous ones (opals, silicified wood) locally contain moganite in variable amounts (from traces up to 75 wt. %). According to the monograph on Minerals of the Carpathians (Udubaşa et al., in Szakáll Ed., 2002), moganite has been previously mentioned from only one Romanian occurrence, in a different geological setting – a metamorphosed manganese ore deposit at Tolovanu, East Carpathians, by Hârtoşanu (in Nedelcu et al., 1997); no analytical data are given for this mineral (Hârtoşanu,

2004). Our study opens new opportunities for the investigation and identification of moganite in microcrystalline and amorphous gem-quality silica varieties in other areas in Romania.

2. The chemically - homogeneous microcrystalline samples (chalcedony, jasper) consist of a mixture of α -quartz and moganite; the homogeneous "amorphous" ones (opal) consist of opal CT with traces of α -quartz and moganite.

3. No correlation between the colour and texture of the chemically-homogeneous ("pure") microcrystalline silica samples and their mineralogical composition could yet be traced by micro-Raman measurements; further investigations are needed.

4. The chemically-heterogeneous samples (chalcedony including agate, jasper, as well as silicified wood) contain, besides the SiO_2 polymorphs, phases such as calcite and microcrystalline graphite as impurities or locally, as the main components.

5. No straightforward correlation between the moganite content and the geological age of the host-rock of the gem materials could be noticed. However the oldest microcrystalline samples from Rachiş (Cretaceous) showed minor moganite contents.

6. An evaporitic genetic setting may be assumed for the microcrystalline silica samples in all the studied occurrences except for Rachiş. Further investigations are needed for documenting this presumption.

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11	Agate	red-white-grey	Gurasada	A71	A81	A82	28	39	38	58	66	66	local moganite (wt. %): ~60 - 65	
12	Chalcedony	white-grey	Gurasada	A51	A61	A21	33	26	27	64	58	58	local moganite (wt. %): ~60 - 65	
13	Chalcedony	white-red	Rachiş										minor moganite + mixture with graphite	
14	Chalcedony	milky-white	Rachiş										minor moganite	
15	Opal	bluish-grey	Oraşu Nou										minor moganite + opal (no α -quartz)	
16	Chalcedony	blue	Trestia	A31	A13	A21	37	30	34	66	61	64	local moganite (wt. %): ~60 - 65	
17	Chalcedony	blue	Techereu	A21	A11	A31	7	10	6	29	34	26	local moganite (wt. %): ~30 - 35	

Error evaluation: Raman band integral ratio for the same data set (measured spot) [%]: relative variation $< \pm 10$ %;
Moganite content for the same data set (measured spot) [wt. %]: relative variation $< \pm 5$ %

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