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Micro-Raman spectroscopy of plagioclase and maskelynite in Martian meteorites: Evidence of progressive shock metamorphism

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Abstract: We present the first systematic Micro-Raman spectroscopic investigation of plagioclase of different degree of shock metamorphism in Martian meteorites. The equilibrium shock pressure of all plagioclase phases of seventeen unpaired Martian meteorites was determined by measuring the shock-induced reduction of the refractive index. Systematic variations in the recorded Raman spectra of the plagioclase phases correlate with increasing shock pressure. In general, the shock induced deformation of the plagioclase crystal lattice leads to an increase of luminescence background and a broadening of all Raman bands. The most persistent bands are those at 505 and 590 cm^{-1}. For shock pressures above ~40 GPa, the post shock temperature in the meteorites was high enough to initialise reordering of the O-tetrahedra in the structure of the diaplectic glass, leading to a decrease of intensity for the band near 590 cm^{-1}. At higher shock pressure (>45 GPa) and post-shock temperature plagioclase starts to recrystallise. This results in a reduced luminescence background and a decrease of the full width of half maximum of the characteristic Raman bands.

key words: Martian meteorites, diaplectic plagioclase, maskelynite, Raman spectroscopy, shock metamorphism

1. Introduction

At present, the group of Martian meteorites consists of about 51 single specimens of 32 unpaired meteorites with a total mass of ~89 kg (Meyer, 2003). All of them were ejected from Mars in the last 20 million years in 4–8 impact events (Nyquist *et al.*, 2001). These rocks document an interplanetary exchange of solid matter, which raises questions regarding the mechanism and the physical conditions during the ejection events. Especially the *P/T* conditions have strong implications for the launching of Martian surface rocks (Artemieva and Ivanov, 2002), for the retention of remnant magnetisation (Weiss *et al.*, 2000), the noble gas content (Schwenzer *et al.*, 2004), and a possible transfer of viable micro-organisms from Mars to Earth (Horneck *et al.*, 2001). While being accelerated above the escape velocity of 5 km/s the meteorites experienced a certain amount of shock loading (*e.g.*, Melosh, 1984; Nyquist *et al.*, 2001). Estimates of the equilibrium shock pressures (Fritz *et al.*, 2002, 2003) and numerical simulations of oblique impacts on Mars (Artemieva and Ivanov, 2004) define a launch window for Martian meteorites of 5 to 50 GPa. These pressures caused significant shock effects in the constituent phases of the meteorites, *i.e.*, strong mosaicism, mechanical twinning, shear fractures and high dislocation densities in pyroxene and, deformation bands, planar fractures, planar deformation features, and also strong mosaicism and high dislocation densities in olivine. Plagioclase is transformed to diaplectic glass and to vesiculated glass and various minerals are transformed into their high-pressure polymorphs (*e.g.*, Sharp *et al.*, 1999; Langenhorst and Poirier, 2000; Beck *et al.*, 2004).

In 1872, Tschermak discovered an isotropic form of plagioclase which shows a conspicuous lack of flow features and vesiculation in the achondrite Shergotty that he termed *maskelynite*. The origin of this material by shock compression was demonstrated by Milton and DeCarli (1963) and maskelynite was since found in numerous shocked terrestrial, lunar and meteoritic rocks (*e.g.*, Bischoff and Stöffler, 1992; Nyquist *et al.*, 2001). Different stages of shock metamorphism of plagioclase ranging from diaplectic plagioclase (birefringent shocked plagioclase), diaplectic glass (maskelynite) and normal (thermal) glass in the Nördlinger Ries impact crater were first described systematically by v. Engelhardt *et al.* (1967) and Stöffler (1967). The definition of these terms is given by Stöffler (1972). Detailed investigations of naturally and experimentally shocked plagioclase showed that the refractive index of maskelynite decreases with increasing shock pressure (Dworak, 1969; Stöffler and Hornemann, 1972; Ostertag, 1983). Based on the experimentally calibrated refractive index of shocked plagioclase, the peak shock pressure (final equilibrium shock pressure) of the host meteorite can thus be deduced (Stöffler *et al.*, 1986). For several Martian meteorites the progressive stages of shock metamorphism of plagioclase including the shock induced reduction of the refractive index were determined to deduce their equilibrium shock pressures (Fritz *et al.*, 2002, 2003; Fritz, 2005). According to these studies, the different degrees of shock metamorphism in order of increasing shock pressures are manifested by:

a) birefringent plagioclase in nakhlites (5–20 GPa) and Chassigny,

b) partly birefringent plagioclase and diaplectic glass in Chassigny (26–32 GPa) and Dhofar 019 (26–29 GPa).

c) diaplectic glass with a refractive index intermediate between the one of a plagioclase crystal and a normal glass with identical An content in Shergotty (30.5 $\pm$ 2.5 GPa), Zagami (29.5 $\pm$ 0.5 GPa), ALH 84001 (32 $\pm$ 1 GPa) and EETA79001 (36 $\pm$ 4 GPa),

d) diaplectic glass with refractive index near the values of synthetic plagioclase glass in DaG 476, and SaU 005 (40–45 GPa) and QUE 94201, Los Angeles, and LEW 88516 (~45 GPa),

e) vesiculated plagioclase glass in ALHA77005 ($>$45 GPa) with a refractive index identical to their synthetic plagioclase glass, indicating the highest degree of shock metamorphism in the Martian meteorites.

The aim of this paper is to correlate the Raman spectra of diaplectic plagioclase phases to the equilibrium shock pressure determined by refractive index measurements (Fritz *et al.*, 2002, 2003; Fritz, 2005) and to evaluate the applicability of Micro-Raman spectroscopy for obtaining the equilibrium shock pressure in shock metamorphosed plagioclase-bearing rocks.

2. Samples and analytical techniques

We investigated plagioclase, maskelynite and plagioclase glass in seventeen unpaired Martian meteorites belonging to the groups of SNCO's. These samples cover the entire range of known lithologies from Mars. Quantitative mineral analyses were performed with a JEOL JXA-8800L electron microprobe operating at 15 kV and a beam current of 10–15 nA. To avoid potential loss of volatile elements, a 5–10 μm sized electron beam was used. Suitable mineral standards including anorthoclase, basaltic glass, chromite, chromium augite, diopside, ilmenite, microcline, and plagioclase, all certified by the United States National Museum as reference samples for electron microprobe analysis (Jarosewich *et al*., 1980) were applied to calculate the mineral compositions. These parameters yielded stoichiometric results for plagioclase normative phases of all meteorites studied.

Micro-Raman spectroscopy was carried out using a notch filter based Dilor LabRam with a HeNe laser of 632 nm wave length. The sample excitation and Raman scatter collection was performed using a 100$\times$ optical lens on the Raman microscope. An energy of 3 mW was transferred to the sample surface with a spot size of 2.5 μm. The Raman spectra were collected from 200 to 1200 cm^{-1}. The final spectra represent the accumulation of 20 single spectra each recorded with a collecting time of 5 s, at the same spot. All measurements were performed on thin sections except for terrestrial plagioclase, where single grains were used. The Raman spectra were collected at three to nine different spots in three to five grains of each meteorite. The analysed plagioclase phases were located at some distance away from any localised shock melts which were present in some of the investigated thin sections. The band intensity relative to the luminescence background was determined in the unprocessed spectra. Backgrounds of the final spectra were graphically reduced by linearly interpolating over discrete frequency ranges. In the most sensitive frequency range we interpolated from 350 to 700 cm^{-1}. Finally, Gauss Lorenz fitting was used to deconvolute the recorded Raman spectra. All spectra were processed with the LabSpec v. 2.08 software by Dilor SA & Université de Reims.

3. Results

3.1. Chemical composition

Representative analyses of plagioclase phases from all analysed meteorites are shown in Fig. 1 and the data is presented Tables 1 and 2. The composition of plagioclase phases in Shergottites range from An_{43-71} with a low K content (Or_{1-6}). In comparison, the ultramafic nakhlites, chassignites and orthopyroxenites, contain Na- and K-enriched plagioclase phases with An_{22-38} and Or_{3-13}. These results are compatible with previous analyses reported for plagioclase and maskelynite in Martian meteorites (*e.g.*, Greenwood and McSween, 2001; Meyer, 2003). In all investigated seventeen unpaired Martian meteorites, analyses of the plagioclase phases are stoichiometric. The composition of the terrestrial plagioclase from Ytterby is Na-rich with $Ab_{77-88}\,An_{19-21}Or_{2-3}$.

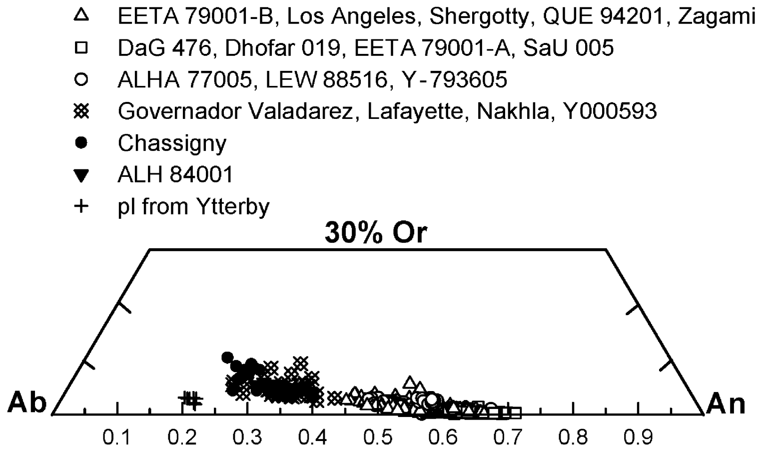

Fig. 1. Quadrilateral diagram showing the chemical composition of plagioclase normative phases in Martian meteorites. Shergottites are displayed with open symbols, nakhlites, chassignites and orthopyroxenites have filled symbols, and the terrestrial reference sample from Ytterby is represented by crosses.

Table 1. Chemical composition of plagioclase phases in Martian meteorites.

	G. V.		Lafayette		Nakhla		Y 000593		Chassigny	
	1	2	1	2	1	2	1	2	1	2
SiO_2	58.9	59.3	59.8	59.3	59.6	59.5	60.0	60.1	62.5	61.6
Al_2O_3	24.9	24.7	24.9	25.5	25.2	25.0	24.8	24.0	23.7	23.9
FeO	2.01	1.07	0.75	0.74	0.71	0.84	0.81	1.27	0.13	0.16
MgO	n. d.	< 0.03	n. d.	< 0.03	n. d.	n. d.	n. d.	n. d.	n. d.	< 0.03
CaO	6.57	7.03	7.05	7.33	7.23	7.42	6.97	6.88	4.33	4.79
Na_2O	6.44	6.64	6.41	6.29	6.76	6.73	6.75	6.49	7.46	7.46
K_2O	0.98	0.49	1.03	0.73	0.53	0.57	0.78	0.97	1.74	1.48
Total	99.76	99.29	99.92	99.91	99.97	100.07	100.12	99.72	99.83	99.45
Ab	60.1	61.2	58.4	58.1	60.9	60.1	60.7	59.4	67.9	67.3
An	33.9	35.8	35.5	37.4	36.0	36.6	34.6	34.8	21.8	23.9
Or	6.0	3.0	6.2	4.5	3.1	3.3	4.6	5.8	10.4	8.8

	ALH 84001		Shergotty		Zagami		QUE 94201		Los Angeles	
	1	2	1	2	1	2	1	2	1	2
SiO_2	60.6	59.8	55.4	53.8	57.1	55.7	56.0	52.9	54.1	53.6
Al_2O_3	25.0	25.7	27.8	28.9	27.3	27.4	27.3	29.4	28.9	28.8
FeO	0.10	0.08	0.68	0.60	0.58	0.62	0.60	0.89	0.66	0.55
MgO	n. d.	n. d.	n. d.	0.08	n. d.	n. d.	n. d.	< 0.03	n. d.	< 0.03
CaO	6.30	6.71	10.7	11.7	9.37	10.1	10.5	12.7	11.6	12.4
Na_2O	7.24	6.98	5.00	4.68	5.50	5.16	4.71	3.93	4.50	4.37
K_2O	0.83	0.78	0.28	0.21	0.36	0.27	0.95	0.09	0.29	0.12
Total	100.02	100.03	99.95	99.94	100.18	99.28	100.08	99.95	100.01	99.96
Ab	64.3	62.3	45.1	41.5	50.4	47.3	42.3	35.7	40.6	38.6
An	30.9	33.1	53.3	57.4	47.5	51.1	52.1	63.7	57.8	60.7
Or	4.8	4.6	1.6	1.2	2.1	1.6	5.6	0.5	1.7	0.7

Table 2. Chemical composition of plagioclase phases in Martian meteorites and the reference sample of terrestrial plagioclase from Ytterby Sweden.

	EETA 79001 B		EETA 79001 A		Dhofar 019		SaU 005		DaG 476	
	1	2	1	2	1	2	1	2	1	2
SiO_2	52.9	52.2	52.9	53.0	51.4	51.5	55.3	51.5	52.4	51.1
Al_2O_3	29.7	30.4	29.3	29.4	30.7	30.7	28.0	30.7	30.2	30.5
FeO	0.58	0.47	0.49	0.73	0.31	0.38	0.22	0.46	0.47	0.38
MgO	n. d.	n. d.	< 0.03	n. d.	< 0.03	0.12	0.11	0.12	0.21	0.20
CaO	12.5	12.7	12.8	12.8	13.7	13.8	10.7	13.4	13.3	14.1
Na_2O	4.20	3.94	4.26	3.95	3.55	3.53	5.09	3.70	3.52	3.50
K_2O	0.18	0.14	0.12	0.09	0.08	0.03	0.17	0.09	0.06	0.09
Total	100.00	99.82	99.91	100.02	99.74	100.03	99.63	99.97	100.10	99.90
Ab	37.4	35.6	37.4	35.6	31.8	31.7	45.7	33.1	32.3	30.9
An	61.5	63.6	62.0	63.8	67.7	68.2	53.3	66.4	67.3	69.0
Or	1.0	0.8	0.7	0.5	0.5	0.1	1.0	0.5	0.4	0.1

	ALHA 77005		LEW 88516		Y-793605		Ytterby
	1	2	1	2	1	2	
SiO_2	55.0	55.0	56.1	53.7	55.8	57.6	64.8
Al_2O_3	28.6	28.2	27.9	29.5	28.0	27.0	22.7
FeO	0.81	0.51	0.32	0.32	0.22	0.34	< 0.06
MgO	0.30	0.14	n. d.	n. d.	< 0.03	< 0.03	n. d.
CaO	9.74	10.6	9.93	11.8	10.2	8.78	3.79
Na_2O	5.19	5.25	5.37	4.50	4.82	5.37	8.23
K_2O	0.41	0.28	0.29	0.17	0.24	0.28	0.47
Total	99.99	99.91	99.91	99.93	99.31	99.40	100.00
Ab	47.9	46.5	48.6	40.4	44.9	51.1	77.4
An	49.6	51.9	49.7	58.6	53.6	47.2	19.7
Or	2.5	1.6	1.7	1.0	1.5	1.8	2.9

3.2. Micro-Raman spectroscopy

The progressive stages of shock metamorphism of the analysed plagioclase phases and Raman spectroscopic characteristics are presented in Table 3 and the recorded Raman spectra of the plagioclase phases are shown in Figs. 2, 4, and 5.

As a reference sample of *unshocked plagioclase*, a terrestrial oligoclase from the island Ytterby near Stockholm, Sweden was used. The terrestrial oligoclase exhibits the characteristic Raman bands at 290, 481, 509, and 574 cm^{-1} (Fig. 2a). Minor bands were observed at 170, 200, 285, 330, 400, 625, 760, 810, 975, and 1100 cm^{-1}. The observed frequencies correlate well with the factor group analysis of vibrational modes for plagioclase (Matson *et al.*, 1986). The full widths at half maximum (FWHM) of the indicative bands at 485 and 510 cm^{-1} are in the range of 12 and 16 cm^{-1} wave numbers, respectively.

Among the Martian meteorites examined in this study, the nakhlites experienced the lowest shock pressures. This is evident by the presence of *birefringent diaplectic plagioclase*. The plagioclase in the nakhlites Y000593 and Lafayette displays no reduction of the refractive index. A slight shock induced decrease of the refractive index was detected for plagioclase of Nakhla and Governador Valadarez (Fritz *et al.*, 2002, 2003). However, no differences in the Raman spectra of plagioclase in these four meteorites are indicated (Fig. 2a). Diaplectic plagioclase in all nakhlites studied displays Raman bands with only a minor shift of the 574 cm^{-1} band to 570 cm^{-1}. As a result of shock metamorphism, however, all bands show a slight band broadening compared to unshocked plagioclase. The FWHD of the bands at 480 and 510 cm^{-1} average at 19 and 16 cm^{-1} wave numbers, respectively. Also, the maximum intensities (I) of Raman bands relative to the luminescence background (LB) are significantly lower (I_{485}/LB = 1.12, I_{509}/LB = 1.2, I_{575}/LB = 1.02) compared to unshocked plagioclase (I_{485}/LB = 1.8, I_{509}/LB = 2.6, I_{575}/LB = 1.2). This reflects an increase of the lumines-

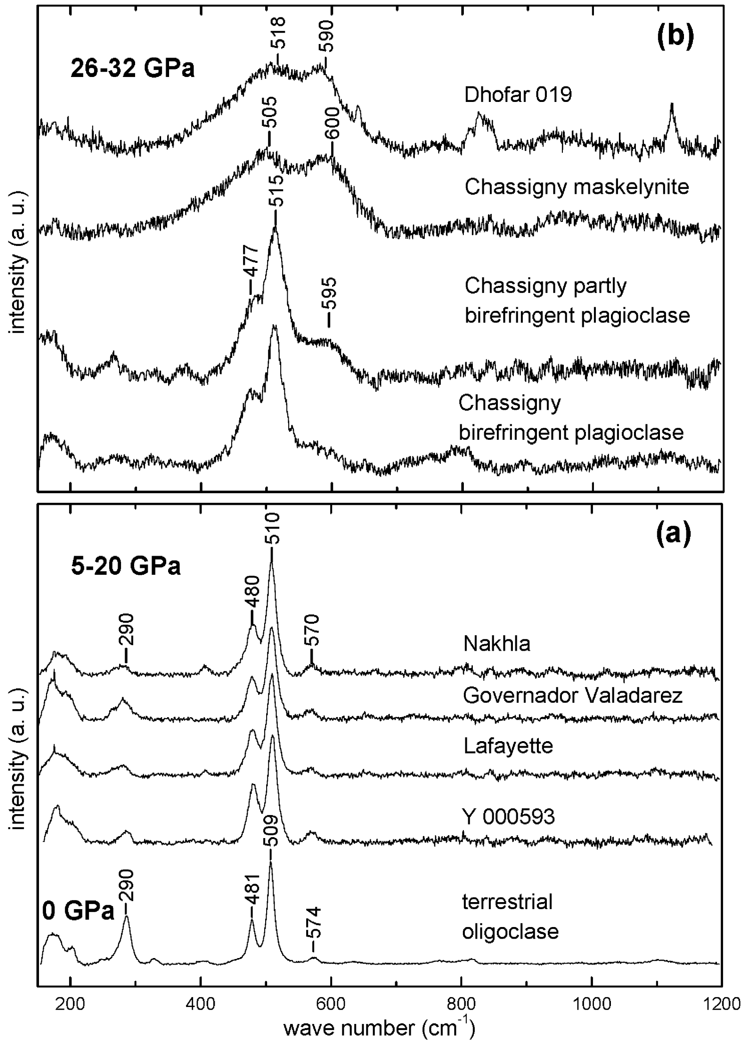

Fig. 2. Raman spectra of plagioclase arranged in the order of increasing shock compression. (a) Raman spectra of unshocked terrestrial oligoclase from Ytterby, Sweden and diaplectic plagioclase from nakhlites in the lower diagram display characteristic Raman bands. Shock metamorphism is indicated by band broadening and a rising of the luminescence background. (b) The transition of plagioclase to maskelynite is accompanied by an intense band broadening and a relative shift of the Raman band 570 to 600 cm^{-1}.

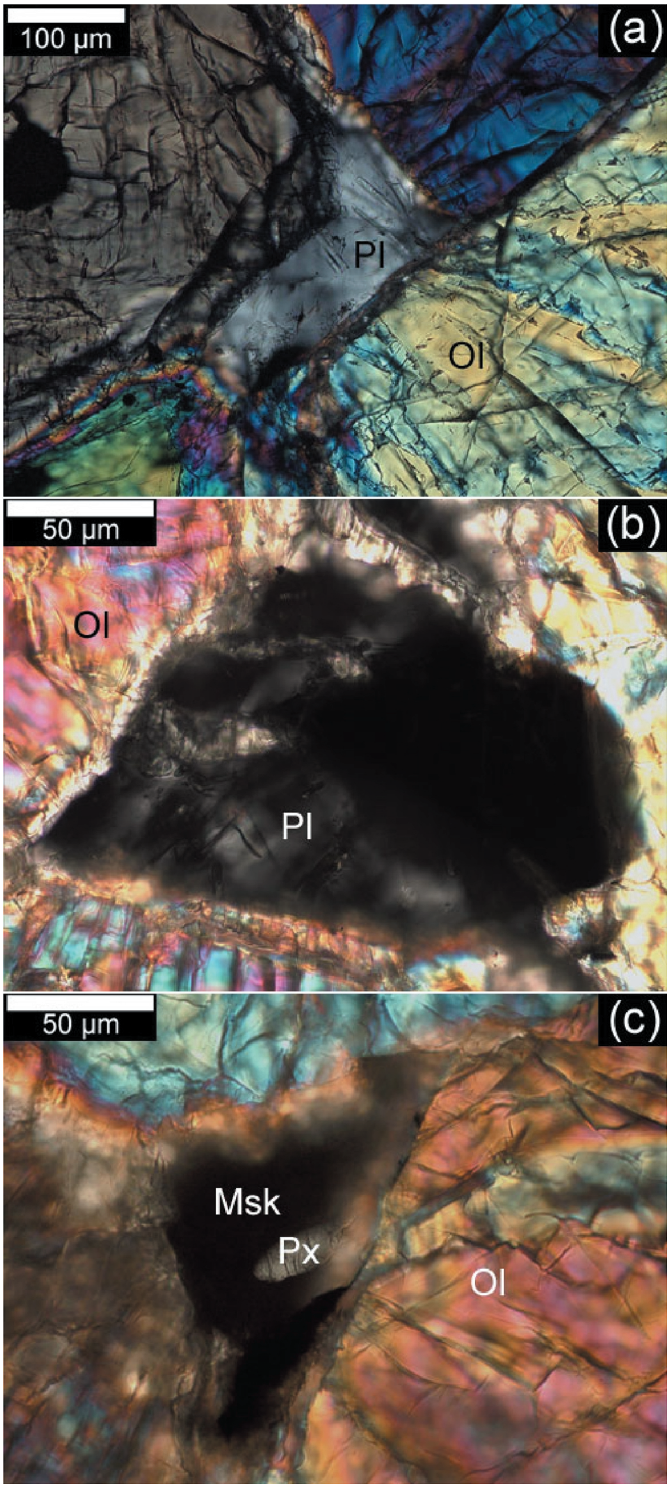

Fig. 3. Transmitted light images of Chassigny with crossed nicols. Plagioclase in Chassigny displays different stages of shock metamorphism : (a) birefringent and (b) partly birefringent diaplectic plagioclase as well as (c) maskelynite. All phases are surrounded by olivine.

cence background in all nakhlites due to shock metamorphism.

The transformation of plagioclase (An_{30}) to partly birefringent plagioclase, and to diaplectic glass is achieved by a shock compression of 28 and 32 GPa, respectively (Stöffler *et al*., 1986). In the case of Chassigny ($An_{25\text{-}30}$), all stages of the transition from plagioclase to diaplectic glass, *i.e*., birefringent and partly birefringent plagioclase, and isotropic maskelynite, are present, probably due to the inhomogeneous distribution

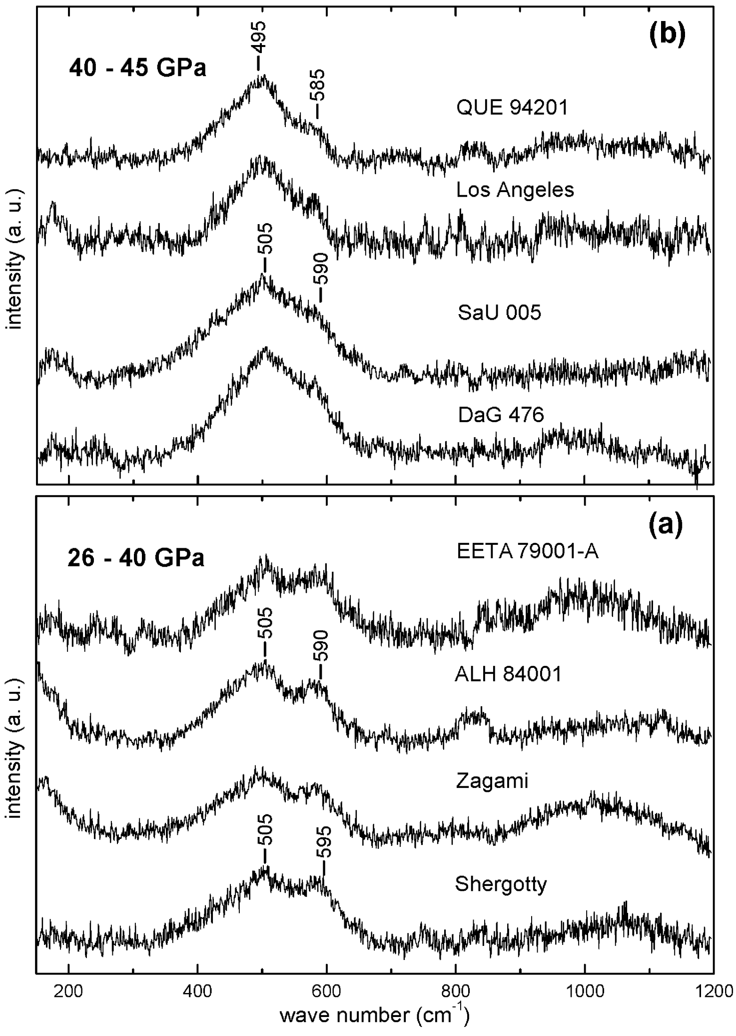

Fig. 4. Raman spectra of maskelynite in Martian meteorites. The spectra of maskelynite formed by shock pressures of (a) < 40 GPa and (b) > 40 GPa are arranged in order of increasing shock pressure as determined by the refractive index of plagioclase (Fritz et al., 2002, 2003). Maskelynite of different An-composition and shock pressure are similar and show characteristic double band structure with peaks at approximately 505 and 590 cm^{-1}. At shock pressure above approximately 40 GPa the relative intensity of I_{590}/I_{505} cm^{-1} is decreasing.

of the shock wave (Figs. 3a–c). Therefore, Chassigny offers the unique opportunity to obtain Raman spectra of plagioclase in different structural states in the same sample (Fig. 2b).

The *diaplectic plagioclase* in Chassigny shows a 100% increase of the FWHM to 36, 25 and 70 cm^{-1} wave numbers for the Raman bands at 475, 515, and 595 cm^{-1} (Fig. 2b) compared to unshocked plagioclase. Additionally, a pronounced shift of the Raman bands at 485, 510, and 570 cm^{-1} to 475, 515, and 595 cm^{-1} occurs. With increasing

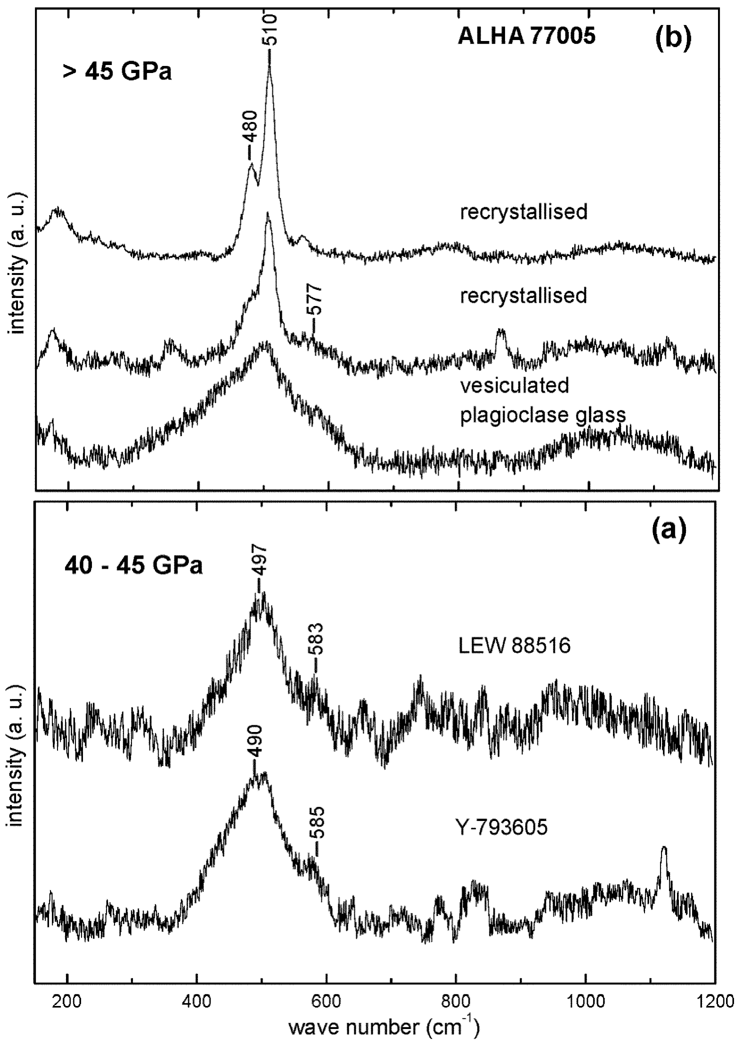

Fig. 5. (a) Raman spectra of maskelynite in lherzolitic shergottites shocked to >40 GPa. The relative intensity of Raman bands at 590 to band at 505 cm^{-1} is low, indicating a high equilibration shock pressure. (b) In the spectra from vesiculated plagioclase glass of ALH 77005 the band at 590 cm^{-1} is obscured by the broad band at 505 cm^{-1}.

Table 3. Progressive stages of shock metamorphism of plagioclase. For the analysed plagioclase phases the structural state, their optical properties and the characteristics of the recorded Raman spectra are shown.

Plagioclase phase	Structural state	Optical property	Raman bands (cm^{-1})	Analysed samples
Unshocked plagioclase	Crystal	Birefringent	290, 481, 509, 574	Terrestrial plagioclase from Ytterby (Sweden)
Weakly shocked plagioclase (5-27GPa*)	Diaplectic crystal	Birefringent	290, 480, 510, 570 band broadening and increasing background	Nakhla, Lafayette, G. V.**, Y000593, Chassigny
Shocked plagioclase (27-30 GPa*)	Diaplectic crystal and diaplectic glass	Birefringent and isotropic	475, 515, 570-600 merging of bands at 475 and 515, increase of band at 570-600.	Chassigny, Dhofar 019, Zagami
Maskelynite (30 – 45 GPa*)	Diaplectic glass	Isotropic	505, 580- 600 both bands form a broad plateau	Chassigny, ALH 84001, Shergotty, Zagami, Los Angeles, QUE 94201, EETA 79001, DaG 476, SaU 005, Dhofar 019, LEW 88516, Y-793605
Quenched plagioclase melt (> 45 GPa*)	Normal glass	Isotropic	broad band near 510, with shoulder near 580	ALHA 77005
Recrystallised plagioclase	Crystal	Birefringent	480, 510, 580	ALHA 77005

* Shock pressures for plagioclase phases with An_{50} according to Stöffler *et al.* (1986).
** G.V. = Governador Valadarez.

shock pressure, the intensities of the bands at 475 and 515 cm^{-1} further decrease relative to the luminescence background for $I_{475}/LB = 1.08$ and $I_{515}/LB = 1.15$ (Fig. 2b); only the band at 595 cm^{-1} shows a slight increase of $I_{595}/LB = 1.03$ compared to diaplectic plagioclase in nakhlites.

The variations of the Raman spectra in diaplectic plagioclase can be expressed by the relative change of the band intensity. I_{510}/I_{480} decreases to 3 to 2 in Y000593 and Lafayette, to ~2 in Nakhla and Governador Valadarez, and to 1.5–2 for the diaplectic plagioclase in Chassigny. In contrast, the I_{590}/I_{505}-ratio increases with increasing shock pressure from 0.05 to 0.1 in diaplectic plagioclase from nakhlites and to 0.2 in diaplectic plagioclase from Chassigny.

Raman spectra obtained from *partly isotropic plagioclase* indicate that upon the onset of isotropisation, the reduction of band intensities and band broadening continue and minor Raman bands vanish in the luminescence background (Fig. 2b). The Raman band at 480 cm^{-1} is present as a shoulder on the flank of the band at 505 cm^{-1} and the Raman band near 570 cm^{-1} shifts to higher frequencies with increasing intensity. Complete isotropisation is indicated by a merging of the Raman bands at 475 and 515 cm^{-1} to a broad band with a maximum at approximately 510 cm^{-1}. A broad plateau with maxima at 510 and 600 cm^{-1} is finally characteristic for Raman spectra of maskelynite. Additionally the intensity of the band at 600 cm^{-1} increases to $I_{600}/LB =$

1.12. The spectra of *maskelynite* in Chassigny are very similar to Raman spectra obtained from oligoclase experimentally shocked above 32 GPa (Heymann and Hörz, 1990).

Plagioclase with An_{65} turns partly and totally isotropic at shock pressures of 26 and 29 GPa, respectively (Stöffler *et al*., 1986). According to the refractive index of maskelynite Dhofar 019 experienced a shock pressure of about 27 GPa (Fritz *et al*., 2003; Fritz, 2005). At this pressure, the plagioclase (An_{57-71}) is almost totally converted to maskelynite with only few weak birefringent domains remaining. However, no differences could be detected in the Raman spectra of the very weakly birefringent areas compared to the total isotropic areas in plagioclase phases of Dhofar 019 (Fig. 2b). Similar results were obtained in plagioclase phases from Zagami. The recorded Raman spectra of the very weakly birefringent domains of diaplectic plagioclase in Chassigny, Dhofar 019 and Zagami can not be discriminated from the Raman spectra of the totally isotropic maskelynite in Chassigny, Shregotty, Zagami, ALH 84001, and EETA79001 (Figs. 2b and 4a).

In the pressure range of 30 to 40 GPa, the refractive index of diaplectic glass strongly decreases (Ostertag, 1983). However, a comparison of the Raman spectra of diaplectic glass in the Martian meteorites which formed at shock pressures in the range of 30 to 40 GPa revealed no significant changes (Fig. 4a). Despite strongly varying An-content (An_{25-70}) and shock compression (26–40 GPa), the diaplectic glass from Chassigny, Dhofar 019, EETA79001-A, Shergotty, Zagami, and ALH 84001, display very similar Raman spectra (Figs. 2b and 4a) with bands near 505 and 590 cm^{-1} being the most persistent; the relative intensities of I_{590}/I_{505} vary between 0.3 and 1.1.

Raman spectra of maskelynite that was formed at shock pressures higher than 40 GPa (Fig. 4b) exhibit significant differences compared to spectra of maskelynite shocked below 40 GPa. Diaplectic glass in the olivine phyric shergottites DaG 476 and SaU 005 and in the basaltic shergottites Los Angeles and QUE 94201 is characterised by a low refractive index with values close to normal glass. This is indicative of high shock compression in the range of 40 to 45 GPa (Fritz *et al*., 2002, 2003). Raman spectra obtained from maskelynite in these meteorites show a decrease of the relative intensity of the band near 590 cm^{-1} (Fig. 3b). While for maskelynite shocked below ~40 GPa, the ratio of I590/I505 is about 0.6, it decreases for maskelynite shocked above 40 GPa to $I_{590}/I_{505} = 0.1–0.4$.

The reduction of the relative intensity of the band at 590 cm^{-1} is accompanied by a reduction of the FWHM from 40–70 cm^{-1} wave numbers in moderately (26–40 GPa) shocked maskelynite to 20–40 cm^{-1} wave numbers for strongly (40–45 GPa) shocked maskelynite.

The Raman spectra of plagioclase phases of the lherzolitic shergottites LEW 88516, Yamato-793605, and ALHA77005 are shown in Fig. 5. The maskelynite found in LEW 88516 is characterised by an extremely low refractive index indicating a shock pressure of about 45 GPa. Apart from maskelynite and recrystallised plagioclase, ALHA77005 also contains abundant vesiculated, shock melted plagioclase glass indicating a significant higher shock pressure compared to LEW 88516; for Y-793605 no data for refractive index are available. Similar to diaplectic glass in basaltic and olivine-phyric shergottites, the most persistent Raman bands of maskelynite in the lherzolitic shergottites are near 500 and 590 cm^{-1} showing a minor shift to lower frequencies. The band at 585 cm^{-1} is characterised by a low relative intensity of $I_{585}/I_{490}=0.05–0.2$ and emerges only as a shoulder on the broad band at 490 cm^{-1}. The shoulder at 585 cm^{-1} is least pronounced in the spectra of plagioclase glass from ALHA77005. Due to the low intensity ($I_{585}/LB=1.00–1.06$), this band is difficult to identify in the broad band at 490 cm^{-1}. The recorded spectra are very similar to Raman spectra reported from labradorite experimentally shocked to 50 GPa (Velde *et al*., 1989).

Thermal plagioclase glass partly rimmed by recrystallised plagioclase is present in ALHA77005 (Ikeda, 1994; Figs. 6a and b). The Raman spectra shown in Fig. 5b illustrate the different stages of recrystallisation of plagioclase in this meteorite. From vesiculated plagioclase glass, which is characterised by an extremely low FWHM of Raman band near 585 cm^{-1} of ~10 cm^{-1} wave numbers to recrystallised plagioclase, the minor band at 585 cm^{-1} shifts to lower frequencies and shows only very small intensities.

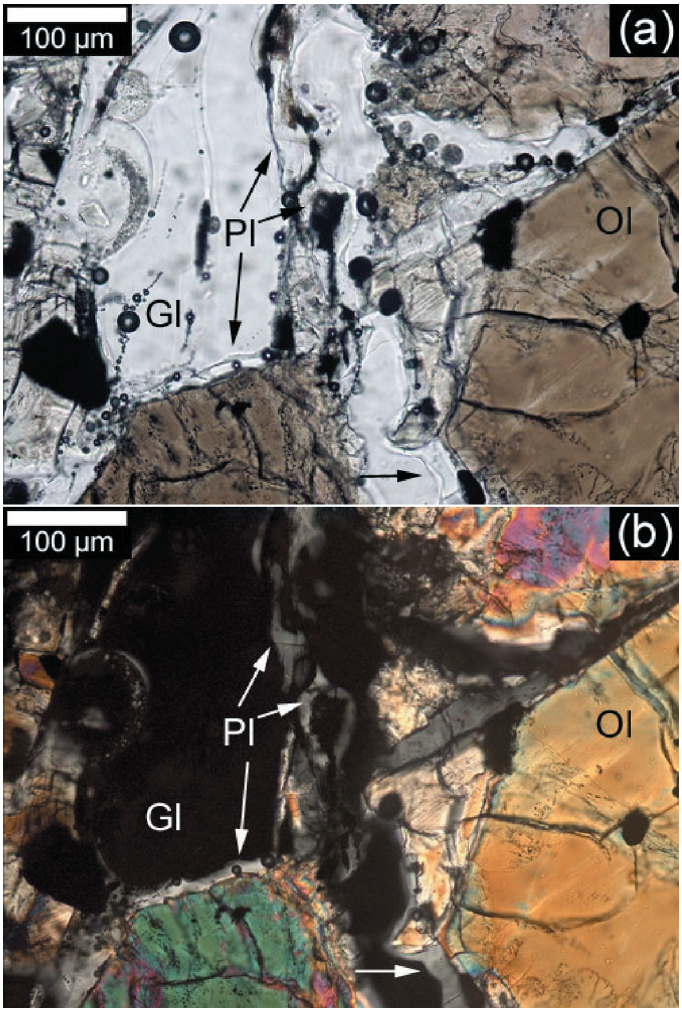

Fig. 6. Transmitted light images of ALHA 77005 (a) plain polarised and (b) with crossed nicols. Shock melted vesiculated plagioclase glass is rimmed by recrystallised plagioclase indicative for shock pressures above 45 GPa.

The FWHM of the Raman band at 485 cm^{-1} decreases and with increasing degree of recrystallisation the band splits up to separate bands at 480 and 510 cm^{-1}. The dominant bands at 510 and 480 cm^{-1} which are characteristic for crystalline plagioclase, have I_{510}/I_{480}-ratios between 1.2 to 2. This value is slightly lower than the I_{510}/I_{480}-ratios of 2.0 to 2.4 of unshocked oligoclase. The FWHM of the Raman band at 510 cm^{-1} is 13–20 cm^{-1} wave numbers, which is slightly higher than the FWHM of 12 cm^{-1} wave numbers of unshocked plagioclase. This observation is in good agreement with the partly fibrous structure of the birefringent plagioclase rim, that display inhomogeneous to undulatory extinction, indicating incomplete recrystallisation of the plagioclase glass.

4. Discussion

4.1. Raman bands diagnostic of shock?

The results presented above show that Raman spectra obtained from plagioclase phases in Martian meteorites systematically change with progressive shock metamorphism. With increasing shock pressure, band broadening and reduction of the band intensities are the main effects observed in the Raman spectra.

All spectra obtained from moderately to strongly shocked maskelynite (26–45 GPa), of variable An contents (An_{22-71}) display a broad plateau with a maximum near 505 and 590 cm^{-1} (Figs. 2 and 4). Variations of band positions between 490 to 520 and 585 to 600 cm^{-1} do not correlate with differences in composition nor with the degree of shock compression as determined by Fritz *et al*. (2002, 2003). These observed variations were not regarded to be diagnostic, since band broadening and reduction of intensity leads to a superposition of three individual broad Raman bands which all have a relative low intensity compared to the luminescent background. This may lead to some artificial variations of the determined positions of the Raman bands. Especially the position of the broad band at 505 cm^{-1} which actually represents the merged bands at 480 and 510 cm^{-1} can be influenced by the relative intensity of the two merged bands. On the other hand, at the transition from plagioclase to maskelynite the narrow Raman band at 570 cm^{-1} increases in intensity and shifts of to higher values (580–600 cm^{-1}) which is clearly documented (Fig. 2b) and hence regarded to be diagnostic. While Chen and El Goresy (2000) and Treiman and Treado (1998) report broad Raman bands at ~800 and ~1050 cm^{-1} in the spectra of maskelynite from Martian meteorites, in our measurements these Raman bands could not be clearly identified due to strong luminescence.

4.2. Isotropisation of plagioclase

Nakhlites are the least shocked Martian meteorites and contain exclusively birefringent plagioclase that is characterised by no or only a minor reduction of the refractive index. The Raman spectra of this plagioclase show a band broadening and an increase of the luminescence background in the Raman spectra compared to unshocked plagioclase in accordance with its weakly shocked nature (Fig. 2a). Comparing vibrational Raman spectra of tectosilicate crystals and their isochemical glass, Matson *et al*. (1986) concluded that the broadness of bands increases with decreasing structural order. Raman spectroscopic investigations of experimentally shocked oligoclase and andesine for shock pressures up to 32 GPa clearly demonstrate that the broadening of bands 476 and 505 cm^{-1} is due to shock metamorphism (Heymann and Hörz, 1990). In addition to shock effects observed in constituent minerals of the nakhlites, *i.e.*, undulatory extinction of olivine and pyroxene, and polysynthetic twinning of pyroxene, the broadening of Raman bands of plagioclase proves that the nakhlites are weakly shocked. This is further evidence for the fact that unshocked nakhlites do not exist.

In Chassigny, the plagioclase phases display a significant reduction of the refractive index and a 100% increase of the FWHM for characteristic Raman bands. The shift of the Raman band at 480 to 475 cm^{-1} (Fig. 2b) is most likely an artefact due to the Gauss Lorenz fitting, since the band at 480 cm^{-1} is present as a shoulder on the broadening band at 510 cm^{-1}. Band broadening together with a decrease of band intensities of the Raman bands at 480 and 510 cm^{-1} finally lead to a single band with a maximum at about 505 cm^{-1} (Fig. 2b). These Raman bands near 500 cm^{-1} can be explained by considering the structure of diaplectic glass. Although the long-range order is destroyed in diaplectic glass, the intermediate-range order (TO_4 ring structures (T=Al, Si)) of such glass is preserved, and can be inferred from the frequency of the dominant symmetric stretch v_s (T-O-T) band in its Raman spectra. In silica glass and tectosilicates the Raman bands at 490 and 505 cm^{-1} are attributed to the motion of the oxygen atom along a line bisecting the T-O-T angle in four-membered siloxan rings (Sharma *et al.*, 1983; Galeener, 1982; Matson *et al.*, 1986).

The Raman spectra of all plagioclase phases in the investigated Martian meteorites show a Raman band near 500 cm^{-1}, indicating distinct structural similarities between plagioclase and maskelynite. With progressive shock metamorphism, a decrease of I_{510}/I_{490} is observed, ultimately leading to a pronounced band broadening that makes the identification of the former separate bands impossible in diaplectic glass. The variation of band intensities near 500 cm^{-1} indicates a change in the proportion of vibrational modes causing the Raman bands at 480 and 510 cm^{-1}. Velde *et al.* (1989) measured Raman spectra of experimentally shocked plagioclase and attributed the relative increase of band 480 compared to 510 cm^{-1} to an increase in structural disorder. Such distortion of the four-fold (terahedral) coordination bonds of Si and Al to weaker, less polymerised bonds that approach six-fold (octahedral) co-ordination is also confirmed by emissivity and hemispherical reflectance spectra of experimentally shocked anorthosite (Johnson *et al.*, 2002). Additionally, several studies of experimentally shocked feldspar suggest that diaplectic glass is intermediate between a truly glassy state and the crystalline state (Ostertag, 1983; Heyman and Hörz, 1990; Yamaguchi and Sekine, 2000; Johnson *et al.*, 2002). Despite differences in the Raman spectra, the strong similarities of the v_s (T-O-T) modes between plagioclase and maskelynite should be noted. The Raman spectra with bands near 500 cm^{-1} show that the medium range order of the T-O tetrahedra is strongly distorted but not substantially rearranged.

Plagioclase phases in the lherzolitic shergottites show the strongest band at a lower frequency (490 cm^{-1}) compared to diaplectic glass from basaltic and olivine-pyhric shergottites (495 to 520 cm^{-1}) (Fig. 5). However, since the obtained Raman bands have low intensities, a strong luminescence, and overlapping band positions, the slightly different band positions may not be diagnostic.

With increasing shock pressure, plagioclase phases in all studied samples show significant variations in band position, intensity, and FWHM for the Raman band at 570 to 606 cm^{-1} (Figs. 2, 4, and 5). This Raman band at 606 cm^{-1} is often referred to as the "defect" band (Matson and Sharma, 1985; Mysen *et al*., 1980) and commonly assigned either to broken Si-O bonds (Seifert *et al*., 1982), or to symmetric stretching vibrations of oxygen in three-membered rings (Galeener, 1982). Studying the structure of quenched melts on the SiO_2-$NaAlO_2$ joints, Seifert *et al*. (1982) observed a shift of the band at 605 cm^{-1} with increasing Al/(Al+Si)-ratios to lower frequencies. Shoulders appearing in the spectra of the alumosilicate glasses between 550 and 580 cm^{-1} correspond to the SiO_2 "defect" band at 606 cm^{-1} (Matson *et al*., 1986). Compared to the obtained Raman spectra for unshocked terrestrial oligoclase, the Raman band at 575 cm^{-1} shifts to slightly lower frequencies in the weakly shocked nakhlites (Fig. 2a), suggesting a preferred breaking of the weaker Al-O bonds (compared to the stronger Si-O bonds) in the tetrahedra network of the tectosilicates. Increasing shock compression leads to a higher defect density in the crystal lattice and to more disrupted Si-O bonds. Hence, the Raman band at 570 cm^{-1} shifts to higher frequencies and increases in intensity (Fig. 2b). In the recorded Raman spectra of diaplectic glass, the band positions vary between 580 and 600 cm^{-1} (Figs. 2, 4, and 5). While achieving an isotropic state, the amount of defect T-O (T=Al, Si) bonds reaches a saturation level at a relative maximum with $I_{590}/I_{505}=0.3$–1.1. Despite the decreasing reduction of the refractive index of diaplectic glass in samples experimentally or naturally shocked between 30 and 40 GPa (Ostertag, 1983; Fritz *et al*., 2002; 2003), no significant change in the recorded Raman spectra of the maskelynite was found within this pressure range.

4.3. *Annealing of maskelynite*

Diaplectic glass from samples shocked above approximately 40 GPa is characterised by a low refractive index, which is near the values for normal glass. Raman spectra of this type of diaplectic glass shocked to very high pressures, show a decrease in the relative intensity of I_{590}/I_{505} compared to moderately shocked diaplectic glass. The relative intensity of I_{590}/I_{505} is 0.3–1.1 for maskelynite formed by shock pressures between 29 to 40 GPa and 0.1–0.4 for maskelynite formed by shock pressures in the range of 40 to 45 GPa.

As outlined above several studies suggest that diaplectic glass is intermediate between a truly glassy state and the crystalline state (Ostertag, 1983; Heyman and Hörz, 1990; Yamaguchi and Sekine, 2000; Johnson *et al*., 2002). For experimentally shocked anorthite glass, Reynard *et al*. (1999) found a correlation of the relative intensity of the Raman band at 580 cm^{-1} with density and refractive index. All parameters reached a maximum at a shock compression of 24 GPa. The decrease of the 580 cm^{-1} band intensity, the refractive index, and the density of diaplectic anorthite glass at shock compressions of more than 24 GPa was attributed to partial annealing immediately after decompression due to high post shock temperatures above 630°C (Reynard *et al*., 1999). It should be noted that, since plagioclase glass is more compressible than crystalline plagioclase, a high post shock temperature is already achieved in the glass at relatively low shock pressures. Ahrens *et al*. (1969) calculated for Oligoclase shocked to ~42

GPa a post shock temperature increase of 600 to 670 K. Starting at temperatures above 450°C and more pronounced above 630°C a partial relaxation of the structural modification was also observed by Raman-spectroscopy in shocked silica glass (Okuno *et al.*, 1999). The decrease of the relative intensity of the Raman band at 580 cm^{-1} observed for maskelynite in the highly shocked Martian meteorites (Fig. 4) can therefore be explained by annealing of the diaplectic glass due to high post shock temperatures.

4.4. Recrystallisation of plagioclase

In vesiculated plagioclase glass of ALHA77005, the Raman band at 585 cm^{-1} is no longer detectable due to the broad band at 485 cm^{-1} (Fig. 5b). Very similar results were obtained for labradorite experimentally shocked to 50 GPa (Velde *et al.*, 1989). Different degrees of recrystallisation of plagioclase glass resulting in crystalline plagioclase with characteristic Raman spectra were observed in ALHA77005. However, broader Raman bands and lower relative band intensities for I_{510}/I_{490} compared to unshocked plagioclase indicate that recrystallisation did not go to completion (Figs. 2a, 5b); this conclusion is confirmed by an inhomogeneous extinction of the birefringent areas (Fig. 6). Annealing experiments of maskelynite show that labradorite shocked to 45 GPa requires 0.5 h at 1000°C or 10 h at 900°C to display signs of recrystallisation (Ostertag, 1982). Using again data of experimentally shocked gabbro (Trunin *et al.*, 2001) and applying the calculation cited above (Artemieva and Ivanov, 2004), a post shock temperature of about 950°C corresponds to a shock pressure of 54 GPa. Additionally, brown staining of olivine and melting of plagioclase strongly suggest that ALHA77005 experienced shock pressures $>$45 GPa (Bauer, 1978; Stöffler and Hornemann, 1972). The high post-shock temperature most likely induced recrystallisation of parts of the vesiculated plagioclase glass.

4.5. Formation of maskelynite

It was suggested that the index of refraction of plagioclase and maskelynite is not indicative for the peak shock pressure, since maskelynite may not be a diaplectic glass but represents a glass quenched from a shock induced dense melt at high pressures (Chen and El Goresy, 2000). This interpretation is based on the observation that maskelynite from Shergotty (a) is smooth and lack any cleavage, cracks and fractures, although neighbouring olivine and pyroxene are pervasively fractured; (b) shows off shots into fractures in the surrounding pyroxene; and (c) entrains fragments from the adjacent pyroxene, (Chen and El Goresy, 2000). However, this does not take into account results from shock recovery experiments on plagioclase, which indicate a change from brittle to ductile behaviour with increasing shock pressures (Grady *et al.*, 1975; Grady, 1980; Yamaguchi and Sekine, 2000). The ductile behaviour of plagioclase in shock compression experiments is a well known phenomena, as shown by (a) sound velocity measurements on feldspar under shock compression indicating a complete loss of strength at stress above the Hugoniot elastic limit; (Grady *et al.*, 1975; Grady, 1980) and (b) shock recovery experiments of feldspar crystals by Ostertag (1983) which reveal that samples shocked between 22 and 26 GPa were recovered powdery and white, whereas above 26 GPa the samples became hard and translucent. Yamaguchi and Sekine (2000) investigated in shock recovery experiments the texture of grain boundaris between plagioclase and pyroxene, and observed in samples shocked at 36 GPa, (too low to produce shock melting in plagioclase) that diaplectic glass was deeply injected into fractures of the adjacent pyroxene and that small pyroxene fragments drifted into the smooth diaplectic glass. Consequently none of the observations discussed by Chen and El Goresy (2000) represents clear evidence for abundant shock melting of plagioclase in Shergotty.

Additionally, the maskelynite from Shergotty shows a refractive index which is intermediate between the one of a plagioclase crystal and the one of a normal glass, all with identical composition (*e.g.*, Tschermak, 1872; Stöffler *et al.*, 1986; Fritz *et al.*, 2003). Furthermore, the recorded Raman spectra show a strong band near 600 cm^{-1} (Fig. 4a; Chen and El Goresy, 2000). Since a significant decrease in the refractive index of the shock densified plagioclase phase and a decrease in the intensity of the Raman band near 600 cm^{-1} is documented by shock recovery and subsequent annealing experiments on anorthite glass above 630°C in less than one minute (Reynard *et al.*, 1999), it can be concluded that Shergotty was not subjected to temperatures in excess of 630°C after the release from shock compression. Chen and El Goresy (2000) proposed that the plagioclase in Shergotty was shock molten and quenched at high pressures. Such a scenario would require unrealistic high quenching rates of the shock molten plagioclase to temperatures below 630°C immediately after passage of the shock wave to preserve the index of refraction observed in maskelynite from Shergotty. In conclusion, maskelynite is not a plagioclase melt quenched at high pressures but a diaplectic glass formed by shock compression at pressures below 45 GPa.

4.6. Calibration of shock pressure

The Raman spectra of all plagioclase phases in the seventeen unpaired Martian meteorites investigated display systematic variations that can be correlated with the equilibrium shock pressure derived from the shock-induced reduction of the refractive index of plagioclase/maskelynite (Fritz *et al.*, 2002, 2003) and other shock induced features observed in olivine and pyroxene (Fritz, 2005). Based solely on Raman spectra, different stages of shock metamorphism in plagioclase phases can be discriminated:

a) For shock pressures below 26 GPa, diaplectic birefringent plagioclase differs from unshocked plagioclase in the FWHM of characteristic Raman bands and reduced band intensities compared to the luminescence background.

b) The Raman spectra of isotropic maskelynite formed between 26 to 40 GPa are characterised by a broad plateau with maxima at approximately 505 and 590 cm^{-1}.

c) The spectra for maskelynite formed by shock in the range of 40 to 45 GPa also show similar characteristics. Additionally in this pressure range the high post shock temperature initiates a partial relaxation in the structure of diaplectic glass, which leads to a reduction of the band near 590 cm^{-1}.

d) For shock pressures significantly exceeding 45 GPa, post-shock temperatures of more than 900°C lead to recrystallisation of plagioclase, as indicated by the splitting of the band at approximately 505 cm^{-1} into two bands at 490 and 505 cm^{-1}.

The described results can be used to estimate equilibrium shock pressures of Martian meteorites for which no refractive index data of plagioclase phases are available, such as Y-793605. Microscopic observations revealed undulatory extinction, strong mosaicism, and planar fractures in olivine and pyroxene as well as maskelynitisation of plagioclase. Shock metamorphic features indicative of moderate to strong shock metamorphism were also reported by Ikeda (1997) and Mikouchi and Miyamoto (1997). Ikeda (1997) pointed out that Y-793605 lacks any melt-pockets commonly observed in ALHA77005 and, thus, concluded a lower shock pressure for Y-793605 compared to ALHA77005. This conclusion is compatible with the absence of vesiculation in maskelynite and the observation that no signs of recrystallisation to birefringent plagioclase could be detected by microscopic observations. Based on the above presented Raman spectra of maskelynite in Y-793605 with $I_{585}/I_{490}=0.15$, a shock pressure of 40 to 45 GPa could be deduced. This is compatible with the observed strong mosaicism and planar fractures in olivine and pyroxene of Y-793605.

5. Conclusion

This is the first systematic study that combines the results of refractive index measurements and Micro-Raman spectroscopy of plagioclase phases in Martian meteorites. It could be demonstrated that systematic variations of Raman spectra with progressive shock metamorphism can be used to identify different shock stages in plagioclase-bearing rocks. Significant variations in the Raman spectra of plagioclase and diaplectic plagioclase are described. However, the strong decrease of the refractive index in the range of 30 to 40 GPa is not reflected in spectra obtained by Micro-Raman spectroscopy. For higher shock compression, annealing due to high post shock temperatures is recorded in the Raman spectra. Micro-Raman spectroscopy is thus capable to discriminate between unshocked plagioclase, diaplectic plagioclase and diaplectic glass with high and low refractive index. However, the differences of diaplectic glass with low refractive index and normal glass can not unequivocally be resolved by Raman spectroscopy. Raman spectroscopy has the advantage of being non destructive and can be applied to unprocessed material. Since plagioclase is a major constituent in most meteorites and planetary bodies, Micro-Raman spectroscopy has potential for application in space missions. The usefulness of Raman spectroscopy on space missions was first explored by Wang *et al.* (1995). Since lander and rover missions get increasingly important in solar system exploration, remote Raman systems are currently developed (Horton *et al.*, 2001; Stopar *et al.*, 2003). This paper outlined the possible application of Raman spectroscopy to calibrate shock metamorphism in plagioclase bearing rocks and may thus be of importance for future space missions especially to Mars.

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