PHILOSOPHICAL TRANSACTIONS OF THE ROYAL SOCIETY A

Phil. Trans. R. Soc. A (2010) **368**, 3153–3166
doi:10.1098/rsta.2010.0104

Raman spectroscopic study of four Spanish shocked ordinary chondrites: Cañellas, Olmedilla de Alarcón, Reliegos and Olivenza

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Shock metamorphism in chondritic parent bodies produces typical textures, visible under the microscope, which are a consequence of structural deformation of the crystals. Such deformations can be studied with Raman spectroscopy. The vibrational characteristics of olivines and pyroxenes, structurally deformed by weak-to-moderate shock metamorphism, have been determined on four Spanish ordinary chondrites (Cañellas, Olmedilla de Alarcón, Reliegos and Olivenza). Such deformations would affect, in principle, the band positions and widths of the Raman spectra peaks. The measured band positions and relative intensities are consistent with chemical composition for olivines and pyroxenes, but show little influence on the degree of shock. However, the full spectral band width of the silicate internal modes shows some dependence on the impact grade, which could be attributed to inhomogeneous effects produced by the impacts.

Keywords: meteorites; ordinary chondrites; Raman spectroscopy

1. Introduction

Spanish meteorites have received very limited attention so far, and only a few studies have been performed, mainly by Spanish researchers: petrological and geochemical studies (Martínez-Frías *et al.* 1989; Llorca Piqué 1997; Muñoz Sanz 1997; Muñoz Sanz *et al.* 1998; Muñoz-Espadas *et al.* 2002*a*, 2003*a*,*b*; Muñoz-Espadas 2003) and isotopic dating (Sanz & Wasserburg 1969; Sanz *et al.* 1970). A Spanish–North American Committee studied and reclassified some specimens (Keil *et al.* 1986; Casanova *et al.* 1990; McCoy *et al.* 1990). But no systematic spectroscopic analysis (infrared or Raman) has been performed on Spanish meteorites to our knowledge.

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One contribution of 12 to a Theme Issue 'Raman spectroscopic approach to analytical astrobiology: the detection of key geological and biomolecular markers in the search for life'.

Many meteorites are affected by shock metamorphism in their parent body, as a result of collisions with other asteroids. The speed of such collisions can reach 42 km s^{-1}, and produce instantaneous pressures as high as 90 GPa, and significant rises of temperature, up to 1000°C (Stöffler *et al.* 1988). Material from the target zone is consequently transformed to more dense and compacted forms, brecciated and sometimes melted.

Shock processes have an important influence on the chemical composition of meteorites, modifying their general features, the abundance and distribution of noble gases and volatile trace elements, and the radiometric age of the material (e.g. Anders 1964; Keil 1964; Heymann 1967; Neal *et al.* 1981; Walsh & Lipschutz 1982; Walsh *et al.* 1983; Friedrich *et al.* 2001, 2002, 2008; Grier *et al.* 2004; Horz *et al.* 2005; Friedrich 2006; Ferroir *et al.* 2008; Hirata *et al.* 2009). Consequently, prior to a chemical study, it is convenient to determine whether the chondrite has been affected by shock, and to what extent. In addition, shock produces typical textures, visible under the microscope, which are a consequence of structural deformation of the crystals. Such deformations can be studied with Raman spectroscopy. Several authors have used this technique to evaluate crystal deformation in highly shocked meteorites (Englert *et al.* 1980; Heymann & Cellucci 1988; Heymann 1990; Chen & Xie 1992; Miyamoto & Ohsumi 1995; Hezel *et al.* 2008; Weselucha-Birczynska & Zmudzka 2008; among others), but no specific Raman studies have been performed on minerals from weak-to-moderately shocked chondrites to our knowledge.

Moreover, the micro-Raman technique could be used to analyse individual mineral grains in a completely non-destructive way, and it offers some unique capabilities as an analytical method in the study of the extent of the shock metamorphism in a chondrite, prior to the microscopical study.

Nevertheless, the Raman spectrum is mainly sensitive to short-range effects on the mineral structure and to a lesser extent to long-range effects. Thus, the transformations experienced by the material, such as partial recrystallization, variation of the crystal size and micro-morphology caused by successive impacts, do not usually have a conspicuous effect on the positions and intensities of the bands. These effects provoke in general only subtle changes in the band width and asymmetry of the bands, which need precise band profile analysis for assessment. Studies regarding these aspects are scarce in the literature.

With these ideas in mind, a detailed micro-Raman study of several Spanish chondrites, Cañellas, Olmedilla de Alarcón, Reliegos and Olivenza, has been performed for the first time. The main aim of the present work is to determine the vibrational characteristics of olivines and pyroxenes in these Spanish meteorites, which are structurally deformed by weak-to-moderate shock metamorphism. In particular, it is of great importance to investigate the influence of such deformations on Raman band parameters (band positions and widths).

2. Experimental details

(*a*) *Samples*

The chondrites used for this study are Cañellas H4, Olmedilla de Alarcón H5, Reliegos L5 and Olivenza LL5. The specimens were provided by the Museo Nacional de Ciencias Naturales (Geology Collection) (Muñoz-Espadas *et al.* 2002*b*). Individual olivine and pyroxene crystals from hand specimens were selected under the microscope. The chemical compositions were previously determined by electron microprobe analyses as follows (Muñoz-Espadas 2003). Cañellas: (fayalite) $Fa_{16.1–17.7}$ and (enstatite) $En_{83.3–84.9}$; taenite 24–41% Ni, 0.2–0.6% Co; kamacite 5–6% Ni, 0.5–1.1% Co. Olmedilla de Alarcón: $Fa_{17.4–18.8}$ and $En_{81.1–83.0}$; taenite, 24% Ni, 0.5% Co; kamacite 6% Ni, 0.1–1.2% Co. Reliegos: $Fa_{22.9–25.2}$ and $En_{75.4–78.7}$; taenite 29% Ni, 0.6% Co; kamacite 6% Ni, 1–2% Co. Olivenza: $Fa_{28.1–30.1}$ and $En_{71.4–75.5}$; taenite 34–37% Ni, 1–3% Co.

(*b*) *Raman spectroscopy*

A total of 23 Raman spectroscopic analyses were performed at the Raman Spectroscopy Laboratory of the Unidad Asociada CSIC al Centro de Astrobiología, Universidad de Valladolid. The spectrometer used was a Hololab 5000 from Kaiser Optical System Co. with a charge-coupled device detection system of 1024 × 128 pixels and a spectral resolution of $1.5\,\mathrm{cm}^{-1}$ per pixel. The spectrometer is coupled with a reflected light optical microscope through a Raman Mark II probe-head from the same company equipped with optical fibres. The objectives used were mainly ×50 and ×100, which lead to a spot of the laser on the sample of 5–6 µm diameter. Spectra are taken in a back-scattering configuration, and to avoid incident laser light entering the spectrometer, the Raman head contains a combination of interference and SuperNotch filters. The integration time was 5–20 s, with 10 accumulations for each spectrum. The spectrometer was calibrated with both quartz and silicon crystals. The resulting spectra were later compared for identification with the Mineral Spectroscopy Server (2002) and Handbook of Raman Spectra (2002) databases as well as with our own spectral databases.

3. Results and discussion

(*a*) *Shock grade of the samples*

The shock-grade classification of the samples (Muñoz-Espadas *et al.* 2002*a*) was established according to criteria defined by Stöffler *et al.* (1991). In hand specimens, the studied chondrites show some features indicative of shock processes during their formation.

Cañellas is a fragmental breccia, consisting of centimetre-sized light clasts in a dark, fine-grained matrix. Both light and dark areas are crossed by dark melt veins. According to this, Cañellas experienced at least one shock event after the formation of the breccia (Casanova *et al.* 1990). Selected olivine crystals from Cañellas show undulatory extinction in some cases (three out of 17 grains), irregular fractures (six grains) and frequent planar fractures (seven grains), and mosaicism has been observed in just one case (figure 1). According to this, Cañellas has been assigned an S3 shock grade.

Olmedilla de Alarcón is a regolithic breccia with light–dark structure (McCoy *et al.* 1990). Its olivines show sparse undulatory extinction (only three out of 19 crystals studied), irregular fractures (three grains) and planar fractures (six grains). In addition, mosaicism and planar deformation features (PDFs) were observed in four and three crystals each (figure 2). All these features lead us to classify this chondrite in an S4 shock stage.

A clear example of undulatory extinction in olivine has been observed in Reliegos. However, seven out of 14 selected olivine crystals show planar fractures, five show mosaicism and only one shows PDFs. A melt vein has been detected in the thin section (figure 3). Following this, Reliegos is classified as S4.

Olivenza has been described as a fragmental breccia (Fodor & Keil 1978; Rubin *et al.* 1983), and the S3 shock classification by Stöffler *et al.* (1991) has been confirmed in this study. Undulatory extinction has been observed in three out of 13 olivine crystals, and irregular and planar fractures are abundant (observed in five olivine grains each; figure 4).

(*b*) *Raman spectra*

Selected Raman spectra obtained from samples of Cañellas, Olmedilla de Alarcón, Reliegos and Olivenza chondrites are shown in figures 5 and 6. The spectral position of the main bands observed is listed in table 1. They correspond mainly to olivine, pyroxene and plagioclase. Chromite and haematite have also been detected, the latter as a possible alteration phase resulting from oxidation of Fe–Ni metal.

An analysis of these results leads to several conclusions:

- The chemical composition for olivine is in a narrow range for all the meteorites. The well-known doublet for the Si$-$O vibration of olivine at 824–856 cm^{-1} can be seen at constant average values of 820–852 cm^{-1} ($\pm 2\,\mathrm{cm}^{-1}$). Using the correlation charts between the position of this doublet and the Mg/(Mg+Fe) ratio (Chopelas 1991), they correspond to forsteritic olivines, with about 10–15% Fe. This is in good agreement with the chemical compositions obtained by electron microprobe analysis.
- The positions of the pyroxene peaks at 330, 658, 676 and 1004 cm^{-1} are characteristic of the more magnesic types, with an Mg/(Mg+Fe+Ca) ratio of about 0.7. These results are also in agreement with data from electron microprobe analysis.

Nevertheless, a close inspection of these values obtained for the fundamental modes of the Si$-$O vibrations in olivines and pyroxenes shows in many cases a small red shift from the expected compositional values. Also, the observed band width of the main bands of olivines and pyroxenes appears systematically larger than that of the terrestrial minerals used as standards for identification. Thus, the possibility of some influence on the band parameters caused by the shocks undergone by the meteorites in their history should not be ruled out and will be discussed later. Previous work on Raman studies of shocked materials has been carried out by several authors. Englert *et al.* (1980) detected a shift to higher wavelength in the position of the Raman line under compressive forces. Heymann & Cellucci (1988) specifically compared the Raman spectra of chondritic laboratory-impacted olivines and non-impacted olivines. For a pressure of 22.2 GPa, the two were identical. However, for higher pressures of 59.5 and 60.7 GPa, they observed lines at 824 and 856 cm^{-1}, although weak and broad, and

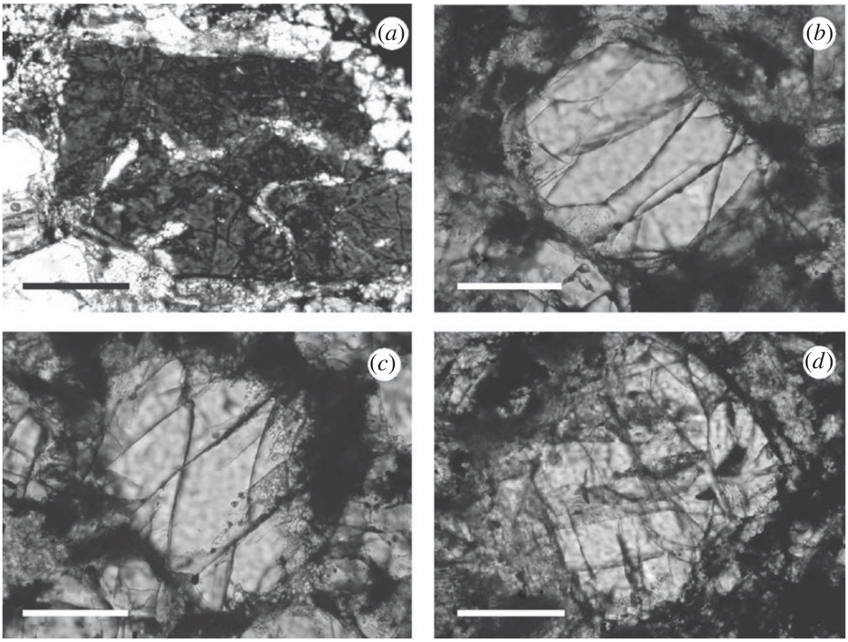

Figure 1. Shock metamorphism effects in Cañellas olivines, observed in thin section. All photographs in transmitted light. (*a*) Undulatory extinction, crossed Nicols. (*b*) Irregular fractures, parallel Nicols. (*c*) Planar fractures, parallel Nicols. (*d*) Irregular and planar fractures, parallel Nicols. Scale bars: (*a*,*d*) 150 μm, (*b*,*c*) 100 μm.

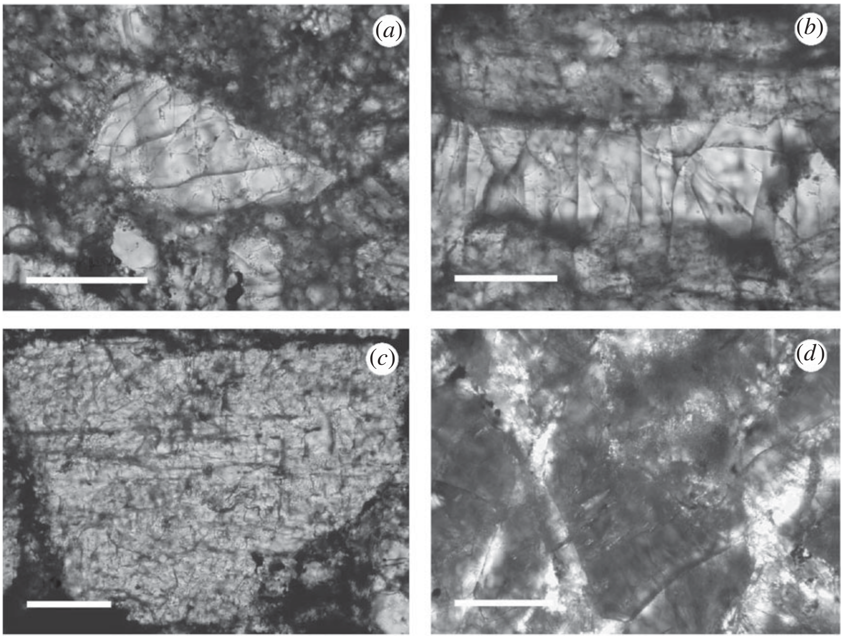

Figure 2. Shock metamorphism effects in Olmedilla de Alarcón olivines, observed in thin section. All photographs in transmitted light. (*a*) Irregular fractures, parallel Nicols. (*b*) Planar fractures, parallel Nicols. (*c*) PDFs, parallel Nicols. (*d*) Mosaicism, crossed Nicols. Scale bars: 100 μm.

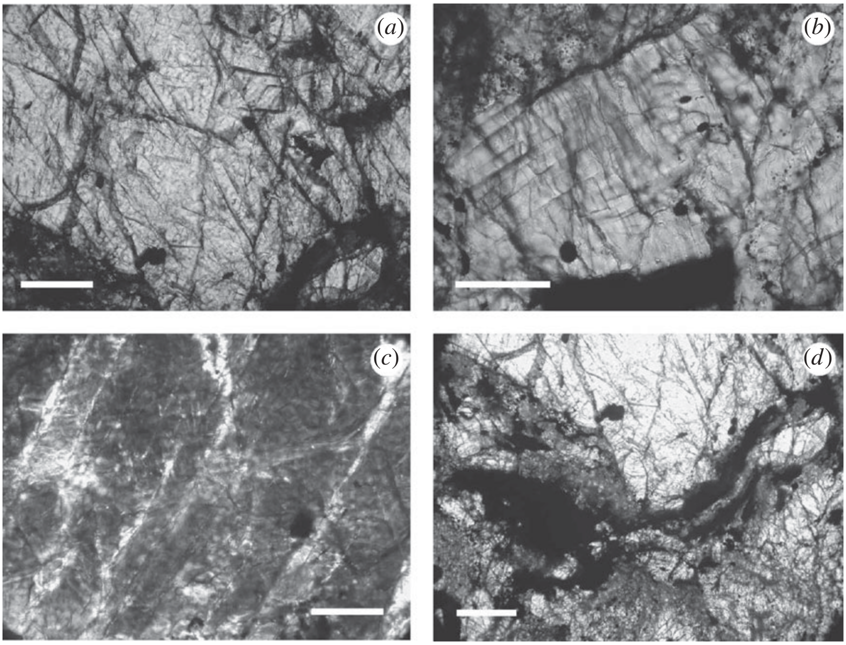

Figure 3. Shock metamorphism effects in Reliegos olivines, observed in thin section. All photographs in transmitted light. (*a*,*b*) Planar fractures, parallel Nicols. (*c*) Mosaicism, crossed Nicols. (*d*) Shock vein, parallel Nicols. Scale bars: (*a*,*c*,*d*) 300 µm and (*b*) 150 µm.

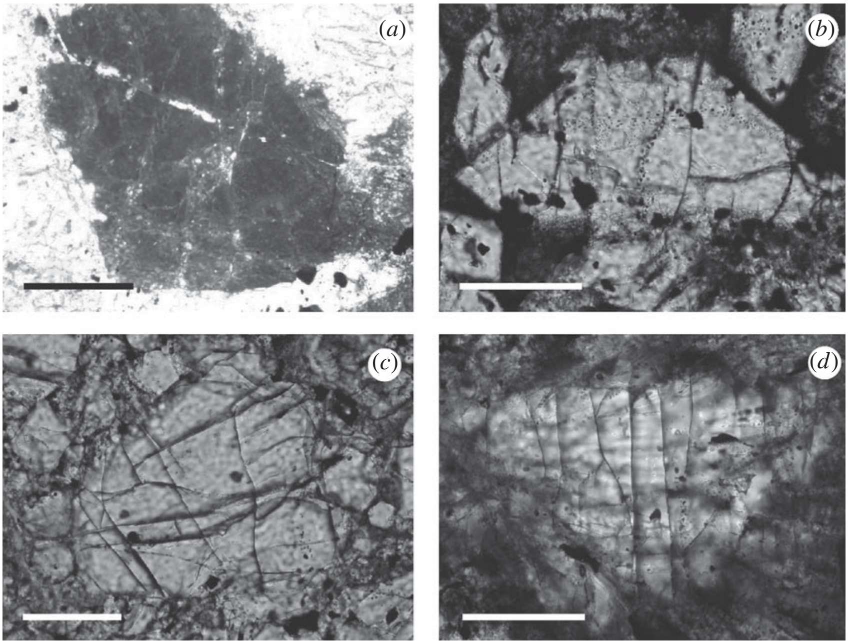

Figure 4. Shock metamorphism effects in Olivenza olivines, observed in thin section. All photographs in transmitted light. (*a*) Undulatory extinction, crossed Nicols. (*b*,*c*) Irregular fractures, parallel Nicols. (*d*) Planar fractures, parallel Nicols. Scale bars: 150 µm.

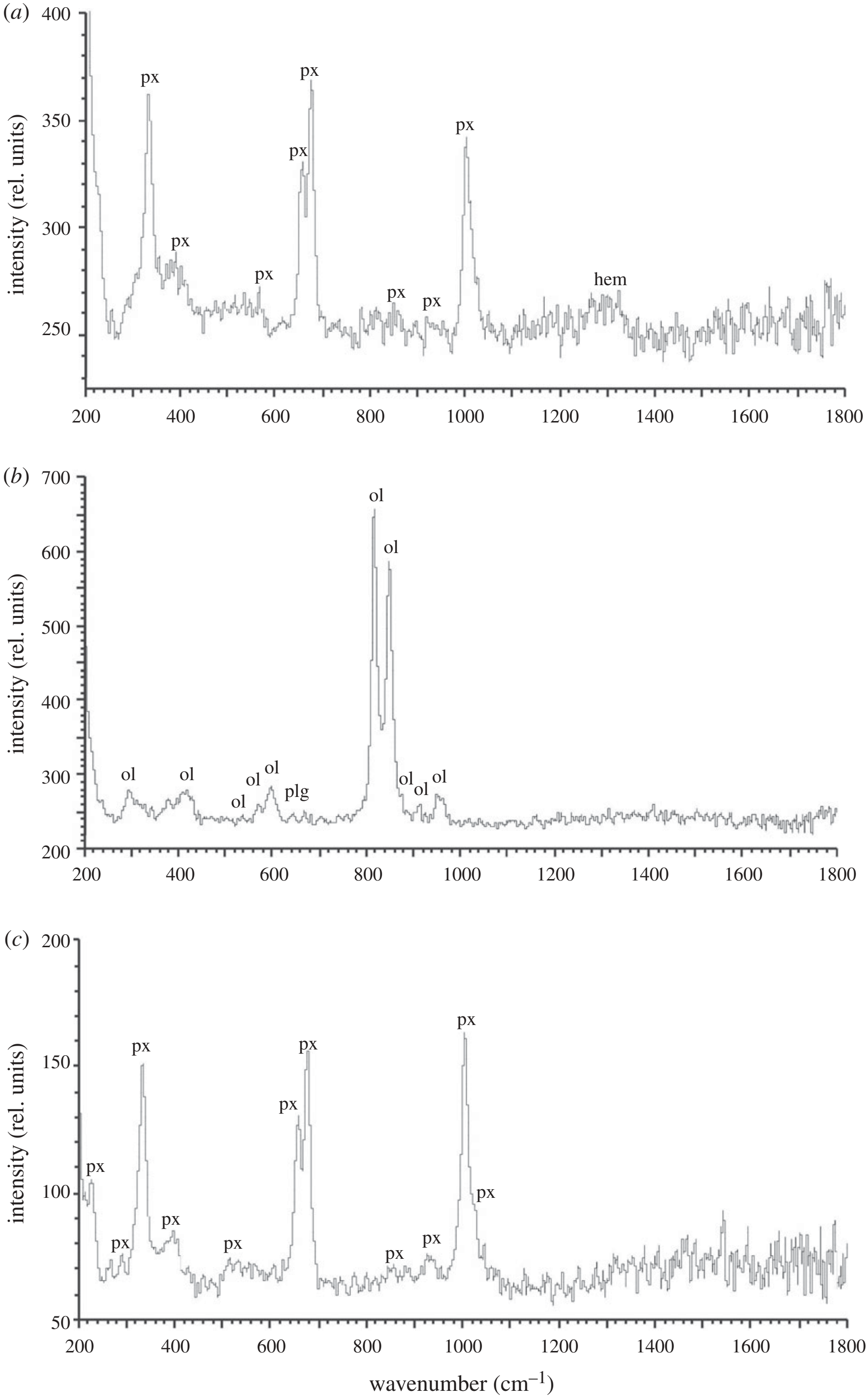

Figure 5. Selected Raman spectra performed on the ordinary chondrite samples. (*a*) Cañellas 1, (*b*) Cañellas 6, (*c*) Olmedilla de Alarcón 2, (*d*) Reliegos 2, (*e*) Reliegos 3, and (*f*) Olivenza 2. See the list of peaks in table 1.

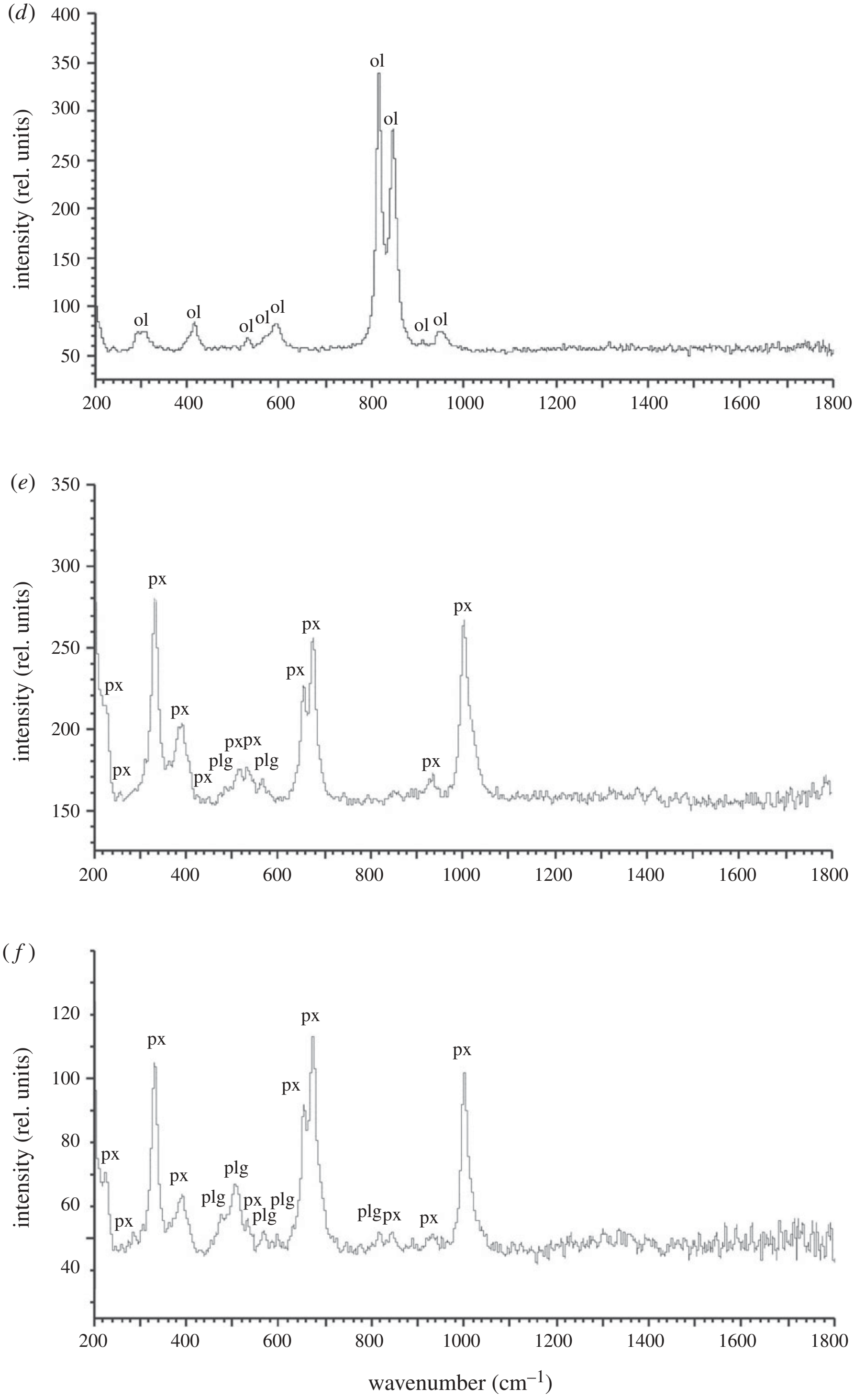

Figure 5. (*Continued.*).

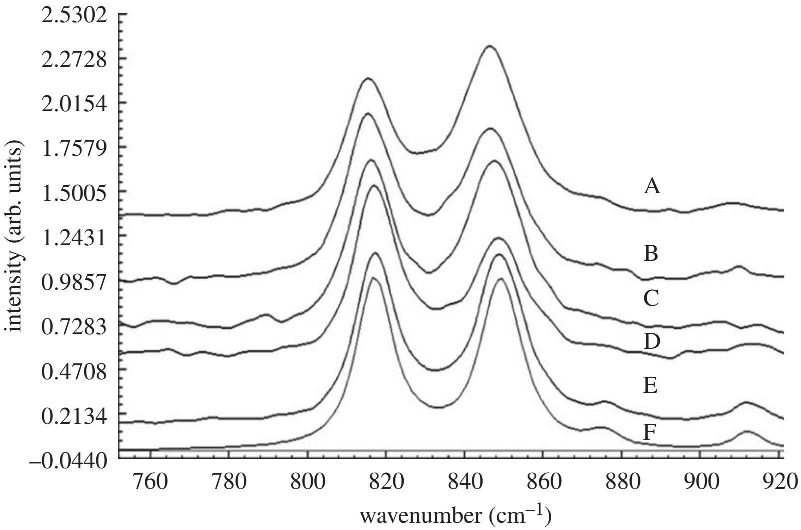

Figure 6. A comparative plot of the Raman spectra of Spanish meteorites in the spectral region of the Si–O vibrations of olivine at 824–856 cm^{-1}. A, Olivenza 4; B, Olivenza 5; C, Reliegos 7; D, Cañellas 7; E, Olmedilla de Alarcón 1; F, terrestrial olivine taken as a reference.

new peaks close to 475, 556, 572 and 1100 cm^{-1}. Those features were interpreted to be the result of the formation of olivine glass with a high degree of three-dimensional Si–O–Si bonds, which probably displayed a wide range of domain sizes, internal structure and possibly chemical composition.

Later, Heymann (1990) extended this study, with a higher number of moderately to intensely shocked samples. The main conclusion is at odds with that exposed by Heymann & Cellucci (1988): the Raman spectra of chondritic shocked olivines and orthopyroxenes are essentially identical to those of non-impacted terrestrial minerals. However, they do not rule out the possible presence of shock-produced glass in an amount too small for detection.

Miyamoto & Ohsumi (1995) detected a small shift in the position of the 820 cm^{-1} line in the Raman spectra of olivines from ordinary chondrites analysed in thin section. They attributed this feature to probable residual stress, as a consequence of the shock processes experienced by the samples. The residual stress could be caused by incomplete recovery from the shock deformation, or by deviation of the static pressure. The full width at half maximum (FWHM) of the peak is considered to be related to the degree of structural disorder of the crystal. However, these authors recognize the influence of other complex factors, such as crystal size and the presence of impurities.

The Raman spectra of orthopyroxene usually show peaks at about 331, 654, 673 and 1001 cm^{-1}. Chen & Xie (1992) detected two new bands at 800–850 cm^{-1} and 1050–1100 cm^{-1} in intensely shocked pyroxenes (pressures higher than 100 GPa and temperatures of about 2000°C), which they attribute to damage of the crystalline structure (figure 7). The new bands detected by Chen & Xie (1992) in the orthopyroxene spectra shocked at 100 GPa are not visible in the spectra obtained in our study, which could be accounted for by the fact that the analysed chondrites have not been subjected to such high pressures.

Table 1. Main peaks measured in the Raman spectra performed on the samples. Assignments: ol, olivine; px, orthopyroxene; crt, chromite; plg, plagioclase, hem, haematites.

	ol	ol	ol	ol	ol	ol	ol	ol	ol	ol	px	px	px	px	px	px	px	px	px	px	px	crt	crt	plg	plg	plg	plg	hem
Cañellas 1												338	394			576	661	680	864	941	1007							1291
Cañellas 2	299	426	538		599	820	852	874	916	956		336		423			658	680			1009							1307
Cañellas 3						819	849		914	954		334	394	424	544	591	659	678			1005							1320
Cañellas 4						824	854				294	338	394	424	544	596	661	680			1007							1315
Cañellas 5											294	337	397	425	538		659	679		937	1007			474		576	629	1288
Cañellas 6	300	419	542	576	601	820	852	876	918	958																		
Cañellas 7	304	425	538	578	589	820	852		918	954		334					662	678			1009							
Olmedilla 1	304	419	538	582	604	820	852	880	916	956	298																	
Olmedilla 2											298	338	399	523			661	680	849	936	1007							
Olmedilla 3	308	421	538		599	820	852	879	916	956	298			523														
Olmedilla 4	304			582	599	820	852	879	920			336									1007	497	680					1304
Reliegos 1	304	414	535	582	584	820	850		914	952			399	523			664	682		934	1008							
Reliegos 2	310	414	535	576	597	820	850		914	956																		
Reliegos 3											300	336	393	518			659	678		939	1005			484		567		
Reliegos 4											299	334	395	523			659	678		934	1005			474		564	624	
Reliegos 5							846				302	336	395	518			658	679	844	936	1005							1304
Reliegos 6	310	425	534	584	594	819	852		909	956																		
Reliegos 7			531	584	597	820	852		909	953												495	680					
Olivenza 1												334	392	429	539		659	677	859		1005				514	572		1321
Olivenza 2											304	336	395		534		658	677	848	937	1005			480	510	566	627	
Olivenza 3												335	393		535		658	677		935	1005				514	567		
Olivenza 4	306		529	577	595	818	850		911	954														476	510			1324
Olivenza 5	304	419	529	577	593	819	850		914	950																		

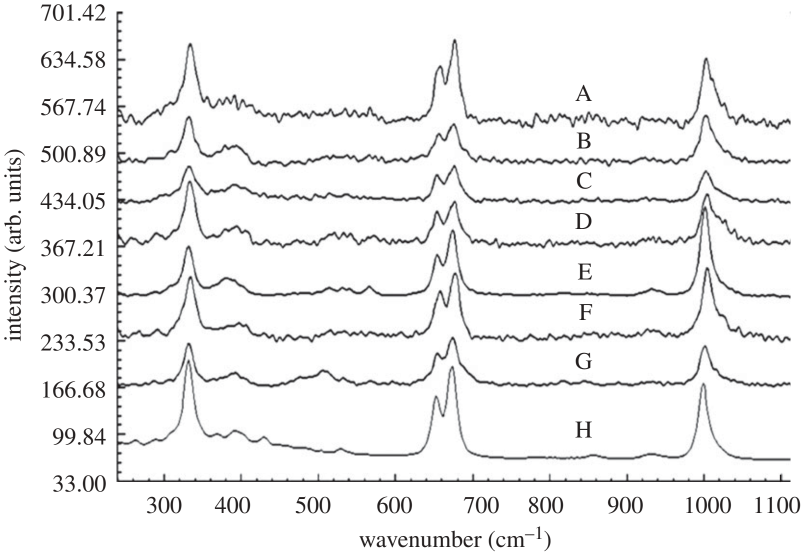

Figure 7. A comparative plot of pyroxene main Raman spectral bands for the Spanish meteorites studied. A, Cañellas 1; B, Cañellas 5; C, Reliegos 4; D, Reliegos 5; E, Olmedilla de Alarcón 2; F, Olivenza 2; G, Olivenza 3; H, terrestrial pyroxene taken as a reference.

The above considerations lead to the conclusion that the band positions alone cannot be used in general as a measure of the degree of shock. This is consistent with the previous considerations pointed out above. The composition changes affect the short-range structure while the mechanical effects of the shock affect the long-range structure. The band positions of the Raman spectra are very sensitive to the former changes.

On the other hand, the measured FWHM averaged for the observed peaks for olivines in the Spanish samples is about 12 cm^{-1} for the component at 820 cm^{-1} and 16 cm^{-1} for the component at 854 cm^{-1}. These values are higher than those observed for well-crystallized terrestrial olivines (9.5 cm^{-1} for the band at 820 cm^{-1} and 10.5 cm^{-1} for the band at 854 cm^{-1}) and a certain structural disorder can be deduced. Comparing the averaged values for each meteorite, it appears that Olmedilla de Alarcón and Reliegos underwent a greater recrystallization process than Cañellas and Olivenza. The S3 shock-grade classification of Cañellas and Olivenza corresponds to pressures of 15–20 GPa, and S4 for Olmedilla de Alarcón and Reliegos to 30–35 GPa (Stöffler *et al.* 1991). Nevertheless, the differences are small, indicating that probably only high pressures and temperatures can account for great changes in the spectra.

Moreover, it is interesting to note that, even though these values are quite homogeneous for all the chondrites studied here, they differ from grain to grain inside each meteorite, which is consistent with optical observations.

Similar results are observed for pyroxenes: the measured FWHM averaged for the observed peak at 1004 cm^{-1} is 19 cm^{-1}, ranging between 16 and 21 cm^{-1}. These values are not very different from those of well-crystallized terrestrial pyroxenes.

4. Final remarks

Micro-Raman spectroscopy has proved to be a useful technique to identify individual mineral grains in meteorites in a completely non-destructive way. The spectra can be used in accordance with complementary analytical techniques to assess the crystal chemical characteristics of the material, i.e. composition and order effects. In the case analysed here, it is found that the measured band positions and relative intensities are consistent with chemical composition for olivines and pyroxenes but show little influence on the degree of shock. On the other hand, the spectral band width of the silicate internal modes displays some dependence on the impact grade. This effect can be measured for each individual grain, showing that impacts provoke inhomogeneous effects in meteorites. In the case of the chondrites analysed, the crystal disorder observed in olivines is consistent with weak-to-moderately shocked ordinary chondrites.

The result may be extrapolated to other minerals and chondrites. The same experiences could be extended in the future to thin section analyses, so that micro-Raman spectroscopic information can be combined with textural information.

We thank Begoña Sánchez, ex-curator of the Geology Collection at the Museo Nacional de Ciencias Naturales, Madrid, for providing the samples. Antonio Sansano and Aurelio Sanz are acknowledged for their contribution to getting the Raman spectra. Also we are grateful to the Centro de Astrobiología (CSIC-INTA), associated with the NASA Astrobiology Institute. This work was partially supported by a MCyT Grant (Spanish Ministry of Science and Technology) to the second author, in the framework of the ESF-IMPACT Program.

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