
Electronic Absorption Spectra of Lunar Minerals

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Electronic absorption spectra of lunar minerals

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Transition metals play an important rôle in the lunar rock-forming silicates, occurring particularly in pigeonite, augite, olivine and pyroxferroite. Measurements of the absorption spectra of lunar silicates can provide information on the oxidation states and co-ordinations of the transition metal ions. Such measurements lead us to conclude that Ti^{3+} as well as Fe^{2+} and Ti^{4+} ions are present in lunar silicates. Fe^{3+} ions do not occur in concentrations greater than a few parts per million, and the spectral evidence for Cr^{2+} ions in olivines requires further substantiation – whereas occurrence of Cr^{3+} is very likely. The occurrence of low oxidation states in the transition elements of the lunar rocks suggests that low oxygen fugacities prevailed during the formation of the lunar crust. However, other reduction mechanisms postulated include release of solar-wind gas, and pressure-induced reduction on meteorite impact or during magma genesis at depth.

Correlations of laboratory measurements with remote telescopic measurements confirm that major features in remote reflectance profiles arise from absorption by pyroxenes, enabling average pyroxene compositions to be determined for areas of the lunar surface. Intense absorption towards the ultraviolet-blue end of the spectrum corresponds to high titanium contents, as confirmed by molecular orbital calculations which support the use of remote spectral measurements to map titanium-rich areas of the Moon.

INTRODUCTION

Certain metallic elements of the first transition series are important constituents of the lunar rock-forming silicate minerals. They occur particularly in the pyroxene (pigeonite, augite, orthopyroxene), pyroxenoid (pyroxferroite), olivine and glass phases, but significant amounts of iron also occur in lunar plagioclase feldspars. The concentrations, oxidation states, co-ordinations and site occupancies of these elements have a major influence on the stabilities and the optical properties of lunar materials. It is essential to determine the oxidation states, co-ordination symmetries and site occupancies of these transition metal ions in order to assess temperatures and oxygen fugacities during the crystallization of the lunar materials. Also, an understanding of the variations in optical properties of the lunar minerals is required for determining the compositions of areas of the lunar surface by remote sensing reflectance spectral techniques.

Considerable information on the crystal chemistry of the transition metal ions can be obtained from measurement of the electronic absorption spectra of lunar minerals, provided that peaks in the spectra are correctly assigned to particular electronic transitions. Numerous chemical analyses of lunar samples have shown that iron, titanium, chromium, and manganese predominate over the other transition elements in lunar silicates. In fact, the concentrations

of Sc, V, Co, Ni and Cu are well below 0.1 mass % in the silicate phases and spectral contributions from ions of these elements are most unlikely to be resolved amidst the numerous absorption bands and peaks arising from Fe, Ti, Cr and Mn. Furthermore, the relatively low abundance of manganese in the lunar rocks, together with the weak (spin-forbidden) spectral features of Mn^{2+} ions, make this element an unlikely contributor to the electronic spectra of lunar minerals. Therefore only the Ti^{4+} , Ti^{3+} , Cr^{3+} , Cr^{2+} , Fe^{3+} and Fe^{2+} ions are likely to produce detectable spectral features in the near infrared-visible-near ultraviolet region examined in electronic absorption spectroscopy.

EXPERIMENTAL TECHNIQUES AND THEORY OF SPECTRAL INTERPRETATION

The silicate crystals in lunar rocks are normally smaller than 1 mm in diameter, so that techniques incorporating microscopes in the spectrometer system are required. Burns (1966) has described a system in which two petrological microscopes (one in the reference beam), to which three-axis universal stages can be attached, are mounted in a Cary model 14 or 17 recording spectrophotometer. The calcite Nicol polarizers of the microscopes produce polarized light over the relevant spectral region, and the universal stage enables single crystals of minerals in petrological thin sections to be oriented with respect to the plane of polarization. The intensities of spectral features are accentuated by using thick, mounted, single crystals or rock sections (50–100 μm). This system has been used to measure the spectra of minerals in Apollo lunar samples with dimensions exceeding 0.75 mm (750 μm) in diameter (Burns *et al.* 1972*a, b*, 1973). The microscope system of Burns (1966) has been modified by Mao & Bell (1973), so that measurements can be undertaken on single crystals as small as 50 μm in diameter and 5 μm thick, for example, extracted from lunar samples (Bell & Mao 1972*a, b*; Mao & Bell 1971, 1973).

The theory of transition metal spectra is described in several authoritative texts and reviews (e.g. Ballhausen 1962; Cotton 1963; Figgis 1967; Lever 1968; Orchin & Jaffé 1971), and can only be given cursory mention here. The absorption spectra of transition metal ions in the visible and nearby regions originate mainly from excitations of electrons between filled and unfilled 3d orbitals. These excitations may be confined to individual ions giving rise to crystal field or intra-electronic transitions, or take place between neighbouring ions in a crystal structure producing charge-transfer transitions. Electron transfer between adjacent elements may involve cation \longrightarrow cation, cation \longrightarrow anion, and anion \longrightarrow cation transitions.

If, for example, a transition metal ion is octahedrally coordinated by oxygens in a silicate mineral the 3d orbitals are split into a more stable group of t_{2g} orbitals (which project between the surrounding oxygens) and a higher energy group of e_g orbitals (which are directed towards the negatively charged oxygens). Electronic transitions between these orbitals (crystal field transitions) lead to absorption of energy in the visible and near-infrared regions (25000–4000 cm^{-1}). The precise energies depend on the nature of the cation, its oxidation state, coordination symmetry, and the metal–oxygen distance. Many of the transitions within cations in low-symmetry coordination sites are polarization dependent. Spin-allowed transitions, in which the electronic configurations of the ground and excited states have identical numbers of unpaired electrons, are usually one or two orders of magnitude more intense than spin-forbidden transitions in which electron pairing occurs in excited states.

Light absorption in the visible region also results from electron transfer between two

ELECTRONIC ABSORPTION SPECTRA OF LUNAR MINERALS 251

different cations (or different oxidation states of the same cation) in adjacent coordination sites sharing a common edge or face. Such metal-metal charge transfer processes may also be strongly polarization dependent (Burns 1973). Charge transfer between metal and oxygen results mainly in the absorption of energy in the near ultraviolet.

Detailed discussions of the spectra of individual transition metal ions are given in the previously cited texts and reviews; discussions relating specifically to transition metals in silicate minerals and in lunar silicates are provided by Burns (1970*a*) and Burns & Vaughan (1975).

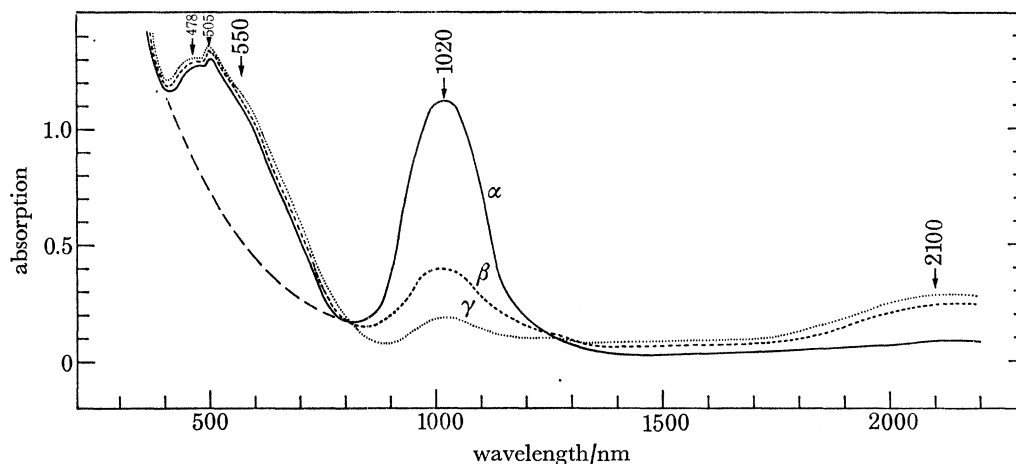


FIGURE 1. Polarized absorption spectra of augite single crystals in rock 74275., γ spectrum; ---, β spectrum; —, α spectrum (from Sung *et al.* 1974).

LUNAR MINERAL SPECTRA

Pyroxenes

Polarized absorption spectra of pyroxenes have been measured from lunar samples obtained by the Apollo 11 (Burns *et al.* 1972*a, b*), Apollo 12 (Mao & Bell 1971, 1973; Burns *et al.* 1972*b*; Cohen 1972; Bell & Mao 1972*a, b*), Apollo 14 (Bell & Mao 1972*b*), Apollo 15 (Burns *et al.* 1972*b*, 1973), Apollo 17 (Sung *et al.* 1974*a, b*) and Luna 20 (Adams *et al.* 1973) missions. For example, figure 1 shows the spectra of pyroxene single crystals from Apollo 17 rock 74275. Prominent absorption bands in the near infrared around $10\,000\text{ cm}^{-1}$ and $5\,000\text{ cm}^{-1}$ represent spin-allowed crystal field transitions in Fe^{2+} ions located mainly in the non-centrosymmetric M2 sites of the pyroxene structure (Hafner & Virgo 1970; Hafner *et al.* 1971*a*; Ghose *et al.* 1972; Malysheva 1972). However, Fe^{2+} ions in M1 sites also contribute to the spectra around $10\,000\text{ cm}^{-1}$. Shifts in the absorption maxima of these spin-allowed transitions can be correlated with pyroxene type and composition (Burns *et al.* 1972*b*; Adams *et al.* 1973; Adams 1975).

Assignment of the peaks in the visible region was controversial. For example, the doublet or asymmetric peak around $19\,800\text{ cm}^{-1}$ has been variously assigned to charge transfer processes involving Fe or Ti, spin-forbidden peaks in Fe^{3+} or spin-forbidden peaks in Fe^{2+} . Of these alternatives, a spin-forbidden transition in Fe^{2+} ions appears to be the most plausible explanation (Runciman *et al.* 1973*a*). The peak at $18\,180\text{ cm}^{-1}$ is also assigned to a spin-forbidden Fe^{2+} transition. These peaks are located on the flanks of a broad absorption band which has a maximum at $20\,920\text{ cm}^{-1}$. This band, and similar peaks in other lunar pyroxene spectra and in

pyroxenes from the Allende meteorite, has been assigned to crystal field transitions in Ti^{3+} ions located in the distorted M1 sites of the pyroxene structure (Burns *et al.* 1972*a, b*; Burns & Huggins 1973; Mao & Bell 1974). However, Dowty & Clark (1973*a, b*) have proposed alternative assignments for the two bands occurring in many lunar pyroxenes in terms of $\text{Fe}^{2+} \longrightarrow \text{Ti}^{4+}$ ($21\,000\text{ cm}^{-1}$) and $\text{Ti}^{3+} \longrightarrow \text{Ti}^{4+}$ ($16\,500\text{ cm}^{-1}$) charge transfer transitions.

Burns *et al.* (1973) have cited spectral evidence for the presence of Cr^{3+} ions in certain Apollo 15 pyroxenes. Broad, weak bands at $16\,670$ and $22\,200\text{ cm}^{-1}$ observed from measurements on pigeonite cores of the pyroxene phenocrysts have been assigned to crystal field transitions in Cr^{3+} ions. The high Cr/Fe ratio and low Fe^{2+} concentration enabled the Cr^{3+} peaks to be resolved in these crystals.

Pyroxferroite

The pyroxenoid mineral, pyroxferroite $\text{CaFe}_6(\text{SiO}_3)_7$ (Chao *et al.* 1970) is unique to lunar mineralogy and occurs, for example, as large phenocrysts in Apollo rock 15058. These phenocrysts, which contain negligible Cr, Mn and Ti, have been used for polarized spectral measurements (Burns *et al.* 1973). The spectra, illustrated in figure 2, show broad, intense bands at 9800 , 7300 and 4750 cm^{-1} and weaker though sharper peaks at 18200 and 19800 cm^{-1} . Determination of the crystal structure of pyroxferroite (Burnham 1971) has shown that Fe^{2+} ions

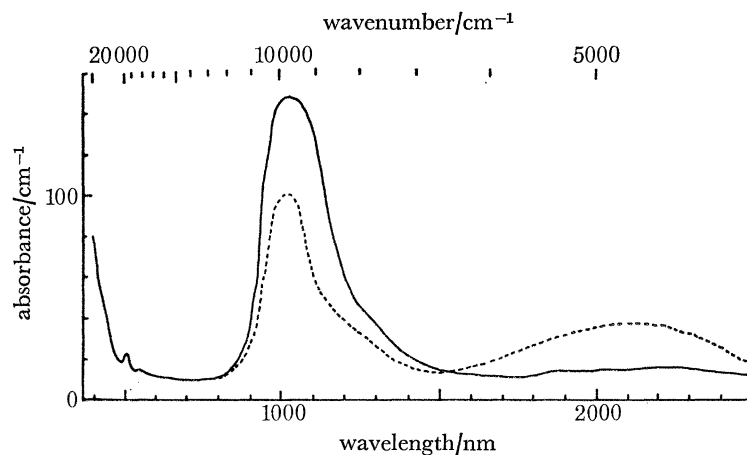


FIGURE 2. Polarized absorption spectra of pyroxferroite from Apollo 15 rock 15058. The composition of this pyroxenoid is $\text{Ca}_{0.13}\text{Mg}_{0.03}\text{Fe}_{0.83}\text{Al}_{0.98}\text{Si}_{0.98}\text{O}_6$. —, β spectrum; ---, α spectrum.

occupy at least five of the seven cation sites. So, the broad bands in the near-infrared region may be attributed to closely overlapping spin-allowed transitions in Fe^{2+} ions located in several coordination sites of the pyroxferroite structure. Spectra of the isostructural terrestrial mineral pyroxmangite ($\text{CaMn}_6(\text{SiO}_3)_7$) have been determined by Manning (1968) but peaks attributable to Mn^{2+} are absent in the lunar pyroxferroite. Since large amounts of other transition metal ions are absent from pyroxferroite, the weak peaks in the visible region are attributed to spin-forbidden transitions in the Fe^{2+} ions. Consequently, the visible-region spectrum of pyroxferroite (figure 2) has enabled certain peaks in the structurally related pyroxenes to be distinguished as spin-forbidden Fe^{2+} transitions, and helped in the overall assignment of peaks in lunar pyroxene spectra (Burns *et al.* 1973).

Olivines

Polarized absorption spectra of olivines have been reported for samples collected during the Apollo 11 (Haggerty *et al.* 1970; Burns *et al.* 1972*a*), Apollo 12 (Mao & Bell 1971; Bell & Mao 1972*a, b*), Apollo 15 (Bell & Mao 1972*a, b*; Mao & Bell 1973; Burns *et al.* 1973), and Luna 20 (Adams *et al.* 1973) missions. The spectra from Apollo rock 15555 shown in figure 3 are typical and are very similar to the spectra obtained from terrestrial olivines (Burns 1970*a, b*; Runciman *et al.* 1973*b*). The broad bands at 8680 and 11210 cm^{-1} are assigned to the spin-allowed transitions of Fe^{2+} ions in the olivine M1 positions (Burns 1970*b*; Mao & Bell 1973; Runciman *et al.* 1974). The more intense band at 9500 cm^{-1} originates from similar transitions in Fe^{2+} ions in the non-centrosymmetric M2 positions. The variations in band maxima can be correlated with compositional variations in the olivines, and agree well with trends established for terrestrial Mg-Fe olivines (Burns 1970*b*; Burns *et al.* 1972*b*).

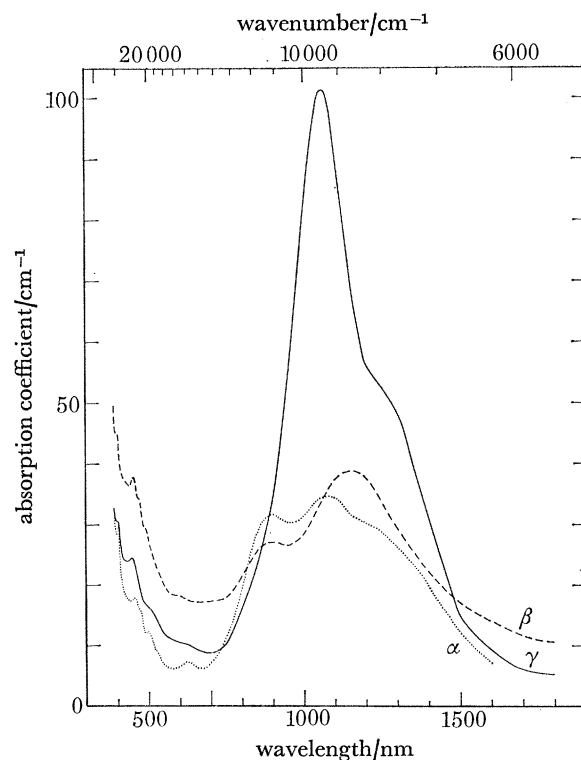


FIGURE 3. Polarized absorption spectra of a lunar olivine from Apollo 15 rock 15555, 205 (from Bell & Mao 1972*b*; Mao & Bell 1973).

The assignment of peaks in the visible region is still a matter of debate, particularly for the peaks at 16800 and 22200 cm^{-1} which have been assigned to $\text{Fe}^{2+} \rightarrow \text{Fe}^{3+}$ charge transfer (Mao & Bell 1971; Bell & Mao 1972*a, b*), Cr^{3+} spin-allowed transitions (Burns *et al.* 1973), Fe^{3+} spin-forbidden transitions when they occur in terrestrial olivines (Burns *et al.* 1973), and Fe^{2+} spin-forbidden transitions (Runciman *et al.* 1973*b*; Burns 1970*b*). Further study is required to resolve these assignments.

Spectral evidence for the presence of Cr^{2+} ions in lunar olivines is also a matter of discussion, particularly in the case of Apollo 11 samples (Burns *et al.* 1973). Spectral features reported

from these samples at 13900 cm^{-1} and 9600 cm^{-1} have been assigned to crystal field transitions in Cr^{2+} ions (Haggerty *et al.* 1970). Although crystal chemical arguments have been proposed in support of the existence and stability of Cr^{2+} ions in olivines (Burns 1975) and forsterites containing Cr^{2+} have been synthesized (Scheetz & White 1972), it has been suggested that the high Fe/Cr ratios in lunar olivines may prevent the resolution and detection of Cr^{2+} spectral features among the numerous features arising from the abundant Fe^{2+} ions (Burns *et al.* 1973). Additional work is also required to solve this problem.

Plagioclase feldspars

Spectra of plagioclase feldspars in Apollo 14 (Bell & Mao 1972*a, b*; Mao & Bell 1973) and Luna (Adams *et al.* 1973) samples exhibit a broad, strongly polarized band around 8000 cm^{-1} with pronounced asymmetry on the long wavelength side. This absorption band may be attributed to Fe^{2+} ions in a variety of sites in the structure. Mössbauer spectroscopy of lunar plagioclases confirms that Fe^{2+} ions occur in the 8–12 coordinated calcium sites (Appleman *et al.* 1971; Hafner *et al.* 1971*b*; Schürmann & Hafner 1972) and in the tetrahedral sites (Appleman *et al.* 1971). The large width of the absorption band may therefore be attributed to overlapping contributions from Fe^{2+} ions in two or more of the four calcium positions of the plagioclase structure (Megaw *et al.* 1962). The asymmetry on the long wavelength shoulder of this band may be due to tetrahedrally coordinated Fe^{2+} ions which exhibit crystal field transitions at lower energies (Slack 1964; Bates *et al.* 1966). Small amounts of Fe^{3+} ions have been reported from e.s.r. measurements on lunar plagioclases (Kolopus *et al.* 1971; Weeks *et al.* 1972; Weeks 1972; Hafner *et al.* 1973) but are most unlikely to be sufficient to produce the weak spin-forbidden bands expected for appreciable concentrations of Fe^{3+} ions.

APPLICATIONS TO LUNAR SCIENCE

Oxidation states of transition metal ions in the lunar minerals and the conditions of their crystallization

Debate in the literature regarding the oxidation states of Fe, Ti and Cr in the Apollo lunar samples as determined from electronic absorption spectra has centred around subtleties of spectral interpretation and tends to obscure the large extent of agreement between different workers (Burns *et al.* 1973). It has been established independently by Mössbauer spectroscopy that almost all of the iron in lunar minerals occurs as Fe^{2+} (Hafner & Virgo 1970; Hafner *et al.* 1971*a*; Ghose *et al.* 1972; Malysheva 1972). Mössbauer studies have failed to detect Fe^{3+} ions in the ferromagnesian silicates and the generally accepted detectability limit for the Mössbauer technique lies in the range 0.3–1.0% of the total iron as Fe^{3+} . However, e.s.r. measurements of lunar plagioclases have clearly shown the presence of several parts/ 10^6 Fe^{3+} (e.g. Weeks 1972; Hafner *et al.* 1973) although this may have been formed after crystallization by shock and radiation effects (Weeks 1973).

The occurrence of Ti^{3+} in addition to Ti^{4+} in lunar pyroxenes was proposed on analytical and crystal chemical grounds from the earliest studies (e.g. Agrell *et al.* 1970; Lovering & Ware 1970; Dence *et al.* 1970; Bence & Papike 1972; Boyd & Smith 1971). The identification of peaks attributable to Ti^{3+} in spectra of lunar pyroxenes has established the presence of Ti^{3+} (Burns *et al.* 1972*a, b*, 1973; Sung *et al.* 1974*a*). Titaniferous pyroxenes from mare basalt samples collected in Apollo 11 and Apollo 17 missions have been estimated to contain 30–40%

ELECTRONIC ABSORPTION SPECTRA OF LUNAR MINERALS 255

of total titanium as Ti^{3+} (Sung *et al.* 1974*b*). Lunar glasses have also been shown to contain substantial amounts of Ti^{3+} (Vaughan & Burns 1973; Bell & Mao 1974). The presence of Cr^{2+} in lunar olivines was suggested on the basis of spectra from Apollo 11 samples (Haggerty *et al.* 1970) and subsequently used to explain the crystal chemistry of Apollo 12 (Boyd & Smith 1971; Weill *et al.* 1971) and Apollo 15 (Boyd 1972) pyroxenes. Its presence has not been convincingly established whereas the presence of Cr^{3+} ions in lunar olivines has been proposed on the basis of reproducible spectral evidence (Burns *et al.* 1973).

Thus, the presence of appreciable amounts of Fe^{2+} , Ti^{4+} and Ti^{3+} in lunar silicate minerals is well established. It is also likely that Cr^{3+} ions are present in olivines, although the spectral evidence for Cr^{2+} ions requires further substantiation. Fe^{3+} ions, certainly present at concentrations of a few parts/ 10^6 in some minerals are unlikely to exceed these concentrations. Even the $FeOOH$ phase reported in such samples as the Apollo 16 'rusty rock' (66095) is now known to be akaganéite (β - $FeOOH$) formed by terrestrial oxyhydration of lawrencite ($FeCl_2$) and native iron (Taylor *et al.* 1974*a, b*). By contrast, metallic iron is widespread in Apollo and Luna samples.

The occurrence of low oxidation states in the transition elements of the lunar rocks indicates the low oxygen fugacities prevalent during the formation of the lunar crust. On the basis of a significant Ti^{3+}/Ti^{4+} ratio and the possible presence of Cr^{2+} , values of P_{O_2} at 1000 °C in the ranges of 10^{-13} – 10^{-14} atm (10^8 – 10^9 Pa) (Brett *et al.* 1971) and 10^{-15} – 10^{-16} atm (Haggerty 1972) have been proposed. However, Mao *et al.* (1973), from studies of Apollo 17 glasses, have proposed that this material could have cooled under conditions of higher P_{O_2} (10^{-8} – 10^{-9} atm). These observations, and the fact that Ti^{3+} concentrations appear to be much greater in Apollo 11 and Apollo 17 samples than any other lunar material, suggest other factors contribute to the high Ti^{3+}/Ti^{4+} ratio in lunar minerals. Bell & Weeks (1974) suggest the release of solar wind gas is an important reduction mechanism and Sung *et al.* (1974*b*) suggest pressure induced reduction of Ti^{4+} to Ti^{3+} occurring as a result of impact shock events or at depth in the lunar interior where the highly titaniferous magmas formed by partial fusion.

Remote sensing reflectance spectra of the lunar surface

Information on the gross mineralogy and petrology of different regions of the Moon's surface may be obtained from remote reflectivity measurements of the lunar surface (Adams & Jones 1970; Adams & McCord 1970, 1972; Pieters *et al.* 1973). The interpretation of the reflectivity measurements depends on the laboratory spectral studies of single crystals, and of whole rock powders using diffuse reflectance methods (Adams 1975). Also, polarized absorption spectra are now being measured over all solid angles of a crystal to clarify the relationship between data from oriented sections and from powders (Gaffey & Burns 1974).

The major features in the remote reflectance profiles arise from absorption by the pyroxenes (Adams & Jones 1970). Correlations between laboratory and remote sensing data have enabled classification of lunar rocks and soils in terms of average pyroxene composition (Adams & McCord 1972). The two major bands in the absorption spectra of pyroxenes (figure 1), which occur around 5000 cm^{-1} ($2\ \mu\text{m}$) and 10000 cm^{-1} ($1\ \mu\text{m}$), are due to spin-allowed transitions in Fe^{2+} ions. These appear as troughs in the reflectance spectrum and compositional variations of the $1\ \mu\text{m}$ band can be used to determine average pyroxene compositions for areas of the lunar surface. Also, plots of the $1\ \mu\text{m}$ band versus the $2\ \mu\text{m}$ band provide a method of obtaining Fe/Mg ratios of pyroxene phases (Adams 1974).

Spectral reflectivity curves of a large number of widely scattered areas on the Moon's surface have revealed that they may be classified into a limited number of spectral types on the basis of different absorption characteristics in the visible and near-infrared regions (McCord & Adams 1973). One type of reflectivity profile, obtained from 'lunar black spots' and 'dark mantling material' surrounding mare (Pieters *et al.* 1973, 1974), is characterized by relatively high reflectance in the ultraviolet and blue region of reflectivity curves ratioed to a standard area in central Mare Serenitatis. The darkness and blueness of the lunar continuum in the visible and ultraviolet was attributed to charge transfer transitions in the regolith involving Fe^{2+} , Ti^{3+} and Ti^{4+} . Furthermore, the steep slope and relatively blue spectral character of the lunar black spots were deduced to indicate high concentrations of Fe and Ti. Charette *et al.* (1974) further distinguished the dark mantling material spectral type from mare, mare crater, upland and bright upland crater spectral types on the basis of plots of reflectance slopes at 0.402–0.564 μm against 0.564–0.948 μm . They further showed that the titanium content of soil samples collected at each Apollo and Luna landing site is related to the slope between 0.402 and 0.564 μm of the telescopic reflectance spectral curves of areas centered on the landing sites.

Clarification of the processes contributing to the visible-region spectra of the Moon's surface is being obtained from molecular orbital calculations on charge transfer transitions. Thus, oxygen \longrightarrow metal charge transfer transitions are important in the near ultraviolet region (Vaughan *et al.* 1973; Loeffler *et al.* 1974) while metal \longrightarrow metal charge transfer involving $\text{Fe}^{2+} \longrightarrow \text{Ti}^{4+}$ and $\text{Ti}^{3+} \longrightarrow \text{Ti}^{4+}$ transitions in ilmenite, ulvöspinel, armalcolite and pyroxene (Loeffler *et al.* 1975) are responsible for broad spectral features spanning 0.5–0.6 μm (*ca.* 20000–16000 cm^{-1}).

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ELECTRONIC ABSORPTION SPECTRA OF LUNAR MINERALS 257

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