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Mössbauer and EPR spectra for glasses and glass-ceramics prepared from simulated compositions of Lunar and Martian soils

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Abstract

In situ resource processing and utilization on planetary bodies is an important and integral part of NASA's space exploration program. Within this scope and context, our general effort is primarily aimed at developing glass and glass-ceramic type materials using Lunar and Martian soils, and exploring various application potentials of these materials for planetary surface operations. This paper reports the successful preparation of glasses from the melts of simulated composition of Lunar and Martian soils, and thermal and structural characterization using differential thermal analysis (DTA), Mössbauer and electron paramagnetic resonance (EPR) spectroscopy. Glass-ceramic materials were developed from these glasses using selective heat treatment, and were also analyzed by Mössbauer and EPR spectroscopy. The crystalline phases formed at different stages of heat treatment were analyzed and identified by X-ray diffraction (XRD). Potential applications envisaged up to this time of these glasses/glass-ceramics on the surface of Moon or Mars are discussed. © 2006 Elsevier B.V. All rights reserved.

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1. Introduction

The long term space exploration goals of NASA include establishing a self-sufficient, affordable and safe human and robotic presence on planetary bodies, especially on Moon and Mars, for an extended period of time [1]. Some of the essential items required for such a permanent and self sufficient extra-terrestrial habitat include structural materials for habitat construction, materials for crew life support including oxygen and water, power generation, and materials and components for scientific investigations. In this context, in situ processing and utilization of resources on the non-terrestrial planetary bodies becomes an integral part of the space exploration mission. This approach would reduce significantly the level of up-mass (transporting materials from earth to planetary bodies) and, hence, reduce the overall work load and cost of the exploration mission. Using the Si present in Lunar or Martian soil to fabricate thin film solar cells for electrical power generation is one such example [2]. The possibility of extracting structural metals and producing refractory materials and glasses from the soils of Moon or Mars has also been proposed [3,4].

A general effort aimed primarily at developing glass and ceramic type materials using simulated compositions of Lunar and Martian soils, and exploring various application

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potentials of these materials on the respective planetary surface is the premise of the present research. The role of glass and ceramics on the progress of human civilization is well known and recognized. Examples of applications of these materials range from common structural components (building and sealing, composites, containers, automotive components, substrate materials for solar cell and electronic applications, rocket nozzles, heat resistant ceramic tiles for space vehicles) to such sophisticated applications as laser and photonic devices, optical glass fibers, gas sensors, bio-materials, electronic ceramics (capacitor, magnet, semiconductor), and many more. Specific applications of a material are controlled primarily by the composition and processing methods and conditions. The major objective of this on-going research is to investigate what type of glass and ceramic materials can be developed from the Lunar and Martian soil compositions, and explore the potential application areas of these materials through extensive property analysis and material characterization. The preliminary results obtained up to this time, namely, a few selected thermal properties, and structural characterization by measuring Mössbauer, EPR, and XRD spectra for the glasses and their devitrified counterparts prepared from the simulated compositions of Lunar and Martian soils are reported and discussed in this paper.

The most abundant component in both Lunar and Martian soils is SiO₂, which makes these soils conducive for fabricating important glass and ceramic products. However, the composition of the Lunar or Martian soil is highly diverse and varies considerably with location. The diverse nature of soil composition of Moon or Mars makes it difficult for the researchers to choose the right composition to work with. Also, the amount of actual Lunar and Martian soil reserve on Earth is limited, and cannot be made available to all the researchers. For these reasons, the Johnson Space Flight Center (JSC) of NASA has developed simulants that closely represent the average composition of the soils on Moon and Mars for use of the research community.

The Lunar soil simulant, designated as JSC-1 (Lunar), was developed from a volcanic ash deposit located in the San Francisco volcano field near Flagstaff, Arizona. This ash, which was erupted from vents in the south flank of Merriam Crater ($35^{\circ}20'$ N, $111^{\circ}17'$ W), was mined, processed (crushing, grinding, mixing, sieving), and stored for future use. The composition of this JSC-1 Lunar simulant, which was made available through the Office of the Curator, Johnson Space Flight Center [5] and is used in the present investigation, closely resembles to that of the

low TiO₂ Lunar soil collected from the Maria geological terrain of the Moon by the Apollo 14 Mission [6,7] (Table 1). However, in terms of composition there is a little difference between the actual Apollo 14 Lunar soil and the JSC-1 Lunar simulant. Since, a near vacuum environment exists in the Moon's atmosphere, the actual Lunar soil does not contain any Fe₂O₃, where as, the JSC-1 Lunar stimulant, which is also re-designated as L01 in this paper (Table 1), contains about 3.4 wt% Fe₂O₃.

The simulant developed [8] by Johnson Space Flight Center for the Martian soil, designated as JSC-1 (Mars) whose average composition closely resembles to the volatile-free composition of the Martian soil retrieved by the Viking mission (Lander site 1) [9] is not currently available. Thus, a simulant for the Martian soil with composition identical to that reported [8] for JSC-1 (Mars) was prepared for the present investigation by mixing the required raw materials in appropriate proportions, and the final composition designated as M01, is shown in Table 1.

This paper reports the preparation of glasses from the melts of simulated composition of Lunar (JSC-1 or L01) and Martian (M01) soils along with their thermal and structural characterization using techniques that include differential thermal analysis (DTA), XRD, Mössbauer and EPR spectroscopy. Glass-ceramic materials were also developed from these glasses using selective heat treatment, and were analyzed by Mössbauer and EPR spectroscopy. The crystalline phases formed at different stages of heat treatment were analyzed and identified by X-ray diffraction (XRD) analysis.

2. Experimental procedure

Glasses from the simulated compositions of Lunar and Martian soils, L01 and M01 in Table 1, were prepared by melting the batches between 1500 and 1550 °C in platinum crucibles in air for about 3 h. A typical melt size was approximately 50 g. Melts were quenched on steel plates and glasses were annealed for 6 h near the respective glass transition temperature. The vitreous characteristics of the as-quenched melts were confirmed by X-ray diffraction analysis (XRD), and the composition of the as-made glasses was determined by chemical analysis (ICP-AES) at the Acme Chemical Laboratories, Vancouver, Canada. As shown in Table 2, the composition determined by chemical analysis for the as-made glasses is in excellent agreement with the batch composition (Table 1).

Glass powders of particle size between 75 and 125 μ m were used for differential thermal analysis (DTA, Perkin

Table 1

Simulant compositions	(wt%) for the	Lunar (JSC-1), L01	, and Martian, M01, soils
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ID	SiO ₂	Al_2O_3	TiO ₂	Fe ₂ O ₃	MnO	CaO	MgO	K ₂ O	Na ₂ O	P_2O_5	FeO
Lunar, L01	47.7	15.0	1.6	3.4	0.2	10.4	9.0	0.8	2.7	0.7	7.4
Lunar, Apollo 14	47.3	17.8	1.6	0.0	0.1	11.4	9.6	0.6	0.7	0.0	10.5
Mars, M01	43.2	23.2	3.8	15.6	0.3	6.2	3.4	0.6	2.4	0.9	0.0

The composition of the soil collected from the Maria terrain on Moon by the Apollo 14 Mission is also shown for comparison.

Chemical analysis by ICP-AES for the glasses prepared from the simulated compositions of Lunar (L01) and Martian (M01) soils										
Glass ID	SiO ₂	Al_2O_3	TiO ₂	Fe ₂ O ₃	MnO	CaO	MgO	K ₂ O	Na ₂ O	P ₂ O ₅
L01	46.1	18.3	1.5	11.3	0.2	10.2	8.6	0.8	2.7	0.7
M01	41.7	24.4	4.0	16.2	0.3	6.6	3.2	3.1	0.1	1.0

Chemical analyses were performed at the Acme Analytical Laboratories, Vancouver, Canada

Elmer DT7) experiments at a heating rate of 10 °C/min in a flowing nitrogen gas (30 mL/min) to determine the glass transition temperature, $T_{\rm g}$, and crystallization temperature, $T_{\rm c}$. The estimated error in determining $T_{\rm g}$ and $T_{\rm c}$ is ± 2 °C.

The glasses were crystallized by heating at temperatures around T_c for ten hours in air, and the crystalline phases were determined using powder X-ray diffraction analysis (Scintag XDS2000) and reference data from the JCPDS files.

The Mössbauer spectra for the as-made glasses and their crystalline counterparts were obtained at room temperature on a spectrometer provided with a 10 mC rhodium matrix cobalt-57 source. The amount of iron found at the sample holder was 4 mg/cm^2 . The velocity of the source was calibrated using a pure iron foil that was also used as a reference for the isomer shift value. The EPR spectra of the powdered samples, the as-made glass as well as its crystalline form, were taken at room temperature with a Bruker EMX homodyne, X-band spectrometer.

3. Results

Table 2

3.1. DTA and XRD

The XRD patterns shown in Fig. 1 for the as-made glasses prepared from the Lunar and Martian simulant compositions are typical of amorphous materials, and do not contain lines representative of any crystalline or unmelted phases. Qualitative assessment also indicates that the cooling rate of the melts required to produce these glasses was less than 40 °C/min. This observation along with the XRD results suggest that the ability to form glass for the melts of Lunar and Martian soils simulants is reasonably high, and glasses with practically no crystalline inclusions can be prepared easily from the melts of these soils.

The typical DTA patterns for the L01 (Moon) and M01 (Mars) glasses are shown in Fig. 2(a) and (b), respectively. The glass transition temperature (T_g) as determined from the DTA curves is about 680 °C and 710 °C for the L01 and M01 glasses, respectively. The DTA curves for both glasses show two exothermic crystallization peaks. For the L01 glass (Moon), a week, but broad exothermic hump centered around 750 °C appears as the first crystallization peak, and a stronger sharp second crystallization peak appears at about 820 °C. For the M01 glass (Mars), the first and second crystallization peaks occur at about 760 °C and 1140 °C, respectively.

Since the compositions of both simulants are complicated multi-component systems, the crystallization mecha-

Fig. 1. XRD for the glasses prepared from simulated compositions of (a) Lunar (L01) and (b) Martian (M01) soils.

nism for these glasses is highly complex, and more than one phase are found to form upon crystallization of these glasses. The XRD patterns for the glasses heated in air for 10 h at the crystallization temperatures determined by DTA are shown in Figs. 3 and 4 for the crystallized L01 and M01 samples, respectively. Heating at about the middle of the first crystallization peak (~750 °C, Fig. 2(a)), the L01 glass crystallizes only Fe₃O₄ (JCPDS-771545), Fig. 3(a). When heated at the second crystallization temperature (\sim 820 °C), this glass crystallizes CaMg(SiO₃)₂ (JCPDS-750945), CaFeSi₂O₆ (JCPDS-711499) and NaFe (Si_2O_6) (JCPDS-711496) along with Fe₃O₄, Fig. 3(b). Like wise, the first crystallization peak for the M01 glass (760 °C) occurs due to the crystallization of Fe_3O_4 only. When heated at the second crystallization temperature (1140 °C), this glass crystallizes α -Fe₂O₃ (JCPDS-771545), Fe₂SiO₄ (JCPDS-832073), and Na(AlSi₃O₈) (JCPDS-711150). The crystalline phases formed by heat treating these glasses at different temperatures are summarized in Table 3.

3.2. Electron paramagnetic resonance spectroscopy

Both the Lunar and Martian soils contain a significant amount of iron in the form of ferrous and/or ferric oxide,





Fig. 2. DTA patterns for the glasses prepared from simulated compositions of (a) Lunar (L01) and (b) Martian (M01) soils.



Fig. 3. XRD for the glass from simulated composition of Lunar (L01) soil after heating at (a) 750 °C and (b) 820 °C for 10 h.



Fig. 4. XRD for the glass from simulated composition of Martian (M01) soil after heating at (a) 760 °C and (b) 1140 °C for 10 h.

Table 3 Heat treatment temperature/time, and crystallized phases determined by XRD

Crysta	llization	Crystalline Pl	hases					
Glass ID	Temperature (°C)	Fe ₃ O ₄ PDF: 771545	Fe ₂ O ₃ PDF: 391346	CaMg(SiO ₃) ₂ PDF: 750945	NaFe(Si ₂ O ₆) PDF: 711492	CaFeSi ₂ O ₆ PDF: 711499	Fe ₂ SiO ₄ PDF: 832073	Na(AlSi ₃ O ₈) PDF: 711150
L01	750/10 h 820/10 h	X X		- X	- X	- X	_	-
M01	760/10 h 1140/10 h	Х	X	-		_	– X	_ X

Table 1. The iron could be detected by electron paramagnetic resonance (EPR) in the glass matrix as Fe^{3+} ions by the appearance of resonance absorptions at $g_{\text{ef}}^{-1} \approx 4.3$, 6.0 and 2.0 [10]. The relative intensity of the absorption lines depends strongly on composition [10]. The $g_{\text{ef}} \approx 4.3$ resonance line is characteristic for isolated Fe^{3+} ions situated predominantly in rhombically distorted octahedral or tetrahedral oxygen environments [10–12]. The $g_{\text{ef}} \approx 6.0$ resonance line arises from axially distorted sites [10–12]. The $g_{\text{ef}} \approx 2.0$ feature is assigned to a ferromagnetic resonance (FMR) of fine-grained precipitates of ferro- or ferrimagnetic single domains encountered in fine-grained precipitates in the glasses [13]. This $g_{\text{ef}} \approx 2.0$ absorption line also appears when iron ion (Fe²⁺ and Fe³⁺) dimers or clusters are present at the interstitial positions [14].

The EPR spectra of the as-made glasses and crystallized Lunar (L01) and Martian (M01) samples obtained in the present study, which are dominated by a broad ferromagnetic resonance (FMR) at $g_{ef} \approx 2$, are shown in Fig. 5. The EPR spectra for both glass and crystallized M01 samples, Fig. 5(b), are broader and more asymmetric compared to those for the L01 samples, Fig. 5(a). Also for crystallized M01 samples there is a small shoulder at $g_{ef} \approx 4$ that is ascribed to a remnant paramagnetic resonance of isolated Fe³⁺ ions located in tetrahedral FeO₄ and/or octahedral FeO₆ local sites having strong rhombic distortion.

3.3. Mössbauer Spectroscopy

⁵⁷Fe Mössbauer spectroscopy was used to determine the valence state and site occupancy of the iron ions. The Mössbauer spectra for the as-made glasses and their crystallized forms for the Lunar and Martian simulants are shown in Fig. 6(a) and (b), respectively. Two Lorentzian

¹ $g_{\text{ef}} \approx (h(\omega)v)/(\beta(\omega_{\beta})B)$, ω being the microwave angular frequency, $\beta(\omega_{\beta})$ the Bohr magneton and *B* the resonance magnetic field.



Fig. 5. EPR spectra for the as-made glass and its crystallized form prepared from the (a) Lunar (L01) and (b) Martian (M01) soil simulants.

doublets have been used to fit each spectrum except that an extra sextet was used to fit α -Fe₂O₃ for the Martian glass treated at 1140 °C for 10 h, and each doublet has been assigned to the Fe²⁺ and Fe³⁺ ions, respectively. The use of two Lorentzian doublets was found sufficient to give a reasonable fit, and the results (average) were the same within experimental error as those obtained by fitting the spectra with four or more doublets. The two ⁵⁷Fe hyperfine parameters; isomer shift δ and quadrupole splitting ΔE_Q , calculated from the two doublets fitting of the Mössbauer spectra are given in Tables 4 and 5. As shown in Fig. 6 and Tables 4 and 5, both Fe²⁺ and Fe³⁺ ions are present in both L01 and M01 as-made glasses, and some of the Fe²⁺ ions in the as-made glass are oxidized to Fe³⁺ after heat treatment.

4. Discussion

All the characteristic transformation temperatures, namely, glass transition, crystallization and melting as



Fig. 6. Mössbauer spectra for the as-made glass and its crystallized form prepared from the (a) Lunar (L01) and (b) Martian (M01) soil simulants.

Table 4	
Room temperature Mössbauer hyperfine parameters, isomer shifts $\langle \delta \rangle$),
quadrupole splitting $\langle \Delta E_Q \rangle$, fraction of Fe ²⁺ for simulated lunar sample	s

Glass samples ID	$\langle \delta \rangle$ (mm/s)		$\langle \Delta E_{\mathbf{Q}} \rangle$	(mm/s)	Fraction ^a of
	Fe ²⁺	Fe ³⁺	Fe ²⁺	Fe ³⁺	Fe^{2+} (%)
L01 'as made glass'	0.91	0.35	1.95	0.74	34
L01, 820° for 10 h	0.92	0.37	1.96	0.76	3

The estimated error in $\langle \delta \rangle$ and $\langle \Delta E_Q \rangle$ is ± 0.03 mm/s.

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^a $Fe^{2+}/[Fe^{2+} + Fe^{3+}]$ was calculated from the Mössbauer spectra.

determined by DTA are consistently higher for the M01 glass, which suggests that the Martian soil has a higher refractoriness than the Lunar soil. The higher refractoriness of the Martian soil probably occurs due to a higher amount of Al_2O_3 content in its composition, Table 1. The working temperature range (difference between the first crystallization and glass transition temperatures) is slightly larger for the L01 glass (about 70 °C) than that of the M01 glass (about 50 °C). This result suggests that

Table 5

Room temperature Mössbauer hyperfine parameters, isomer shifts $\langle \delta \rangle$, quadrupole splitting $\langle \Delta E_{\rm Q} \rangle$, fraction of Fe (III) for simulated martian samples

Sample $\langle \delta \rangle$ (mm/s)		$\langle \Delta E_{ m Q} angle \ ({ m mm/s})$		Fraction ^a of Fe ²⁺	Fraction ^a of α -Fe ₂ O ₃	
	Fe ²⁺	Fe ³⁺	Fe ²⁺	Fe ³⁺	(%)	(%)
M01 'as made glass'	0.91	0.33	1.95	0.68	48	0
M01, 1140° for 10 h	0.98	0.40	1.98	0.77	15	20

The estimated error in $\langle \delta \rangle$ and $\langle \Delta E_{\Omega} \rangle$ is ± 0.03 mm/s.

^a $Fe^{2+}/[Fe^{2+} + Fe^{3+}]$ was calculated from the Mössbauer spectra.

drawing glass fibers from the L01 melt would be slightly easier than from the M01 melt, which is demonstrated to be true in a later phase of this continuing investigation.

The ferromagnetic resonance (FMR) predominant at $g_{ef} \approx 2$ of the EPR spectra for both L01 and M01 (Fig. 5) samples could be related to Fe³⁺ ions occupying positions closer than what it would have been if they were at the tetrahedral substitutional sites, giving rise to exchange interactions between the neighboring spins [10–12]. Furthermore, the presence of dimers or clusters containing more than two iron ions (Fe²⁺ or Fe³⁺) at the interstitial positions [14] can be another possibility. In many oxide glasses the intensity of the line with $g_{ef} \approx 2$ increases much faster than that of $g_{ef} \approx 4.3$ with increasing Fe³⁺ ion concentration resulting in Fe³⁺ clustering [14], which effectively contributes to the broadening of line width such as one observed in the EPR spectra of Martian (M01) glass.

Another interesting feature observed in Fig. 5 is the decrease of the EPR spectral intensity for both L01 and M01 heat treated samples compared to those for their glassy counterparts. This behavior generally implies for an eventual reduction or Fe^{3+} ions to Fe^{2+} , since only Fe^{3+} contributes to the EPR spectra at room temperature. However, Mössbauer analysis in Fig. 6 showed exactly the opposite results, namely, an oxidation of Fe^{2+} ions to Fe^{3+} . Similar results showing oxidation of Fe^{2+} ions to Fe^{3+} , have been reported [15,16] for many iron phosphate glasses after heat treatment. The reason for this apparently opposite results observed by EPR and Mössbauer spectroscopy is not understood at this time, and additional work is continuing for a better understanding of the underlying processes involved during heat treatment of these glasses. It may be possible that the observed reduction in the EPR signal intensity in Fig. 5 that also indicates a reduction of the super-paramagnetic behavior is associated not with a reduction of Fe^{3+} to Fe^{2+} , but with the precipitation and growth of non-paramagnetic crystallites containing substantially larger, perhaps of multi-domain, ferromagnetic phases containing Fe^{3+} .

There is a strong tendency for the precipitated phases in glasses to be spherical and well dispersed due to their interfacial surface tension and homogeneous nucleation [13], so that shape anisotropy and particle-particle interactions can be eventually neglected. In ordinary cases, the precipitates are treated as single domain particles and the occurrence of some anisotropy of the FMR spectra will be interpreted as a magneto crystalline anisotropy.

The ratio of $Fe^{2+}/[Fe^{2+} + Fe^{3+}]$ for the as-made L01 and M01 is 0.34 and 0.58, respectively, Tables 4 and 5. The isomer shift, $\langle \delta \rangle$ -values, of 0.33–0.40 mm/s and guadrupole splitting, $\langle \Delta E_{\rm O} \rangle$, of 0.68–0.77 mm/s are characteristics for the isolated Fe^{3+} ions occupying the distorted octahedral sites as suggested also by the present EPR spectra. The isomer shift of Fe^{2+} ions ranges from 0.91 to 0.98 mm/s and the values for quadrupole splitting range from 1.95 to 1.98 mm/s. These values of isomer shifts and quadrupole splitting correspond to octahedral coordination for Fe²⁺ ions in these glasses [17]. The presence of α -Fe₂O₃ in the heat treated M01 sample is evident from its Mössbauer spectrum in Fig. 6(b), see also Table 5 for the amount present, which is further supported by the X-ray diffraction analysis of the sample, Fig. 4(b). The α -Fe₂O₃ particles have been shown [18] to act as typical super-paramagnetic particles and their presence in the heat treated M01 could lead to develop a paramagnetic behavior. The magnetic characteristics of the heat treated M01 glass have been demonstrated separately in an earlier study [19].

5. Conclusions

The soils on the surface of Moon or Mars is expected to form excellent glasses when melted and quenched as has been demonstrated in the present investigation using simulated compositions of Lunar and Martian soils. The characteristic transformation temperatures, namely, the glass transition, crystallization, and melting temperatures for these glasses are generally very high (higher than commercial silicate glasses), indicating the suitability of these glasses for high temperature applications. The presence of both Fe²⁺ and Fe³⁺ ions, most likely in octahedral coordination, has been detected both in the glass and its devitrified form, the Fe³⁺ ions occupying substitutional and interstitial sites in the network. The α -Fe₂O₃detected by Mössbauer spectroscopy and XRD in the crystallized Martian sample (M01), which also caused an asymmetry in its EPR spectra could be related to a more paramagnetic behavior of this sample. The results of the present investigation demonstrate the feasibility of developing glass and ceramic type materials for use on the surface of Moon or Mars from the soils of these planets. Drawing continuous glass fibers from these melts has been also demonstrated, which may find applications for producing glass fiber reinforced composite materials for structural applications.

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