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Abstract:

Apollo-era seismic profile remains controversial on the existence of a lunar inner core composed of face centered cubic (fcc) structured iron (Fe), although selenophysical observations support its presence. In particular, it is strongly challenged by mineralogy because the inferred seismic velocity is 27% lower than that of fcc-Fe. Here we report in-situ high-pressure and high-temperature X-ray diffraction (XRD) and acoustic velocity measurements of iron-carbon (Fe-C) solid solution. Fe alloyed with 0.2-1.7 wt.% C undergoes an isostructural phase transition around 4-5 GPa accompanied by a notable velocity drop of ~1 km/s. The inferred seismic velocity profile of the lunar inner core can be directly matched by this new phase in Fe-C alloy (hereafter denoted as fcc-II; conventional fcc-Fe is referred as fcc-I). These findings provide compelling evidence for the existence of the lunar inner core composed of fcc-II Fe-C with a temperature potentially below 1500 K.

Introduction

The Chang'e missions stimulate a new upsurge after the Apollo missions in the exploration of the Moon. Far-side lunar regolith samples returned by the Chang'e-6 mission have advanced insights into surface volcanic processes¹, the origin of the ancient lunar magnetic field², and mantle water distribution^{3,4}. These new findings extend our knowledge of the Moon from its surface to deep mantle. When referring to the lunar core, it is commonly accepted that the Moon possesses a molten core composed of Fe and sulfur (S)⁵⁻⁹. To date, it is still debated whether an inner core exists. The moment of inertia, tidal deformation, dissipation-related quality factor, and paleomagnetism obtained from selenophysical observations¹⁰⁻¹³, thermodynamic constraint^{9,10}, lunar dynamo model¹⁴⁻¹⁶, and phase equilibria¹⁷ strongly indicated an inner core with a radius up to 280 km. Compared with those methods, seismic core phases can directly detect the structure of the lunar core. Unfortunately, the seismic observations from the Apollo-era seismic data can not conclusively resolve the existence an inner core^{5,6,11,18}.

The inferred seismic profile of the lunar outer core (~4.1 km/s) is about ~7% lower than that of liquid Fe⁷. Nickel (Ni) and S could reduce the V_P of liquid Fe individually or jointly at lunar core conditions^{7,19-21}. Similarly, 3.5 wt.% carbon is reported to lower down the V_P of liquid Fe by ~2%, but

its effect is reversed in the presence of Ni²²⁻²⁴. To date, the ~7% V_P deficit of lunar outer core can be well explained by the incorporation of 4-10.5 wt.% S^{7,19,21}. With this composition, if crystallization occurs in the lunar core, pure fcc-Fe would crystallize from the liquid core with negligible S, according to the partitioning coefficient of S between solid and liquid Fe, which is nearly zero at lunar core conditions²⁵⁻²⁷. Thus, if the moon possesses an inner core, the seismic velocities of the solid metallic core would be ~4.3 km/s for the compressional wave velocity (V_P)^{5,11}. It has been reported that the V_P of fcc-Fe at lunar core conditions are ~5.6 km/s^{28,29}. Nickel (Ni) is expected to alloy with Fe in the terrestrial cores, while the incorporation of Ni has negligible effect on the acoustic velocity of Fe under lunar core conditions³⁰. This leaves the 23% velocity discrepancy between the lunar inner core and pure fcc-Fe unresolved, which profoundly challenges the existence of a solid inner core in the Moon.

Trace light elements like silicon (Si), hydrogen (H), and oxygen (O) rarely partition into metallic lunar core due to the high oxygen fugacity and relatively low pressure¹⁸. In contrast, phase equilibria experiments¹⁷ and mass balance calculations³¹ indicate that C could reside in the lunar core with a content ranging from 0.3-4.8 wt.%. Fe is known to present a liquid phase and four solid phases, which are body-centered cubic (bcc, including α and δ), fcc (or γ), and hexagonal close-packed (hcp or ϵ) phases. Recently, the incorporation of C into Fe is reported to induce a liquid-liquid (L-L) phase transition in molten Fe around 5 GPa³²⁻³⁷. The L-L transition in molten Fe-C has been attributed to $\delta - \gamma$ solid phase transition in Fe³³, but such L-L phase transition does not occur in liquid Fe³⁸, invalidating it as the mechanism for the L-L phase transition in Fe-C. The expected transition pressure of ~5 GPa is exactly the pressure in the lunar inner core, prompting us to test whether similar structural changes occur in solid bcc-Fe-C (hereafter bcc means only α phase) and fcc-Fe-C. If such structure changes exist in solid Fe-C, the ultra-low inferred seismic velocity of lunar inner core might be explained. To address these questions, we conducted in-situ energy-dispersive X-ray diffraction (XRD) and acoustic velocity measurements on Fe alloyed 0.2 wt.% C (Fe-0.2C) and 1.7 wt.% C (Fe-1.7C) solid solutions up to 7 GPa and 1300 K in a Paris-Edinburgh press coupled with Pulse-echo overlap technique.

Room-temperature behaviors of Fe-C alloys

The acoustic velocities of Fe-C alloys at room temperature are illustrated in Fig. 1. The V_P and

79 shear wave velocity (V_S) of bcc-Fe obtained from parallel experimental studies have been reported²⁸.
80 The V_P is in the range of 6.0-6.5 km/s and linearly increases as a function of pressure (0-7.0 GPa),
81 while its V_S varies little and remains nearly constant at 3.5 km/s. The results of bcc-Fe are consistent
82 with prior work³⁹. C doping fundamentally alters these properties of bcc-Fe. At pressures below 4.0
83 GPa, 0.2 wt.% C could increase the V_P of bcc-Fe by ~ 0.4 km/s, while it slightly decreases the V_S by
84 0.1-0.3 km/s. The effect of C on V_P of bcc-Fe is the same as that of Si^{28,40}. Recently, the V_P of alleged
85 Fe-2.2C has been measured by picosecond acoustic in diamond anvil cell up to 15 GPa at room
86 temperature⁴¹. The incorporation of 2.2 wt.% C seems to reduce the V_P of bcc-Fe. However, the sample
87 was deposited and may have had porosity. In particular, C might distribute along the boundary of Fe
88 particle/grain and hardly diffuse into the lattice of Fe during deposition. The measured V_P likely
89 represent the mixture of Fe and C instead of Fe-C solid solution. Light elements are generally supposed
90 to increase the V_S of Fe⁴²⁻⁴⁶, however, we find that the incorporation of C could decrease the V_S of Fe.
91 This effect is same as our recent results on the bcc-Fe-Si alloys²⁸. Above 4.0 GPa, we observe a gradual
92 decrease in the V_P of Fe-0.2C and the V_P drops to ~ 5.7 km/s above 5.5 GPa. Its V_S , meanwhile,
93 increases linearly with pressure below 4.0 GPa, but plateaus at ~ 3.4 km/s above 4.0 GPa. The acoustic
94 velocity discontinuities in both V_P and V_S indicate a phase transition in Fe-0.2C around 5 GPa. The
95 XRD characterization (Fig. 1b) shows that the bcc structure phase persists up to 7.3 GPa. The XRD
96 and acoustic velocity results evidence that it is an isostructural phase transition (ISPT). We denote the
97 low-pressure phase structure as bcc-I (<4.0 GPa) and the high-pressure phase as bcc-II (>5.5 GPa),
98 respectively. At pressures between 4.0 GPa and 5.5 GPa, the V_P lies between those of bcc-I and bcc-II,
99 indicating a mixed-phase region of Fe-0.2C. A $\sim 1.6\%$ volume collapse is also observed at ~ 4.5 GPa,
100 further confirming the ISPT in Fe-0.2C (Fig. 1c).

101 Further, the starting Fe-1.7C sample was employed to clarify how varying carbon concentration
102 affects the aforementioned ISPT. Fe-1.7C starts as a bcc-fcc mixture at room temperature. Below 4.5
103 GPa, the V_P stays constant at 6.2 km/s and the V_S linearly increases from 2.5 km/s to 3.3 km/s (Fig.
104 1a). 1.7 wt.% C is supposed to significantly increase the V_P of bcc-Fe, but the V_P of bcc/fcc-Fe-1.7C
105 is smaller than that of Fe-0.2C and comparable to that of pure bcc-Fe. Notably, 1.7 wt.% C reduces the
106 V_S of Fe by 6-29%, which is much larger than the effect of 0.2 wt.% C (3-8%) likely due to both carbon
107 doping and the fcc phase coexistence. Above 4.5 GPa, the V_P suddenly drops to ~ 5.6 km/s and the V_S
108 keeps constant at ~ 3.2 km/s. These results illustrate that the Fe-1.7C sample also undergoes an ISPT

around 4.5 GPa at room temperature. We also obtained the V_P and V_S of Fe-1.7C during decompression. The V_P and V_S are ~ 5.6 km/s and ~ 3.2 km/s above 4.0 GPa which are same with their compression values. Below 4.0 GPa, V_P suddenly increases to ~ 6.2 km/s and V_S drops to 2.5-3.3 km/s, which means both V_P and V_S recovers to the values in compression process below 4.5 GPa. The variation trend of V_P and V_S indicates that the ISPT is reversible.

Mechanism of the ISPT in iron-carbon alloys

For decades, Fe is believed to present a liquid phase and four solid phases which are bcc (including α and δ), fcc, and hcp. A L-L phase transition in molten Fe-C was reported around 5 GPa³²⁻³⁷. Here one more isostructural phase, bcc-II, is observed in solid Fe with the presence of C. In order to investigate the mechanism of ISPT in Fe-C alloy, synchrotron Mössbauer spectra (SMS) and x-ray emission spectra (XES) were conducted at room temperature. Fe-0.2C persists in ferromagnetic state up to 12.3 GPa and then transform to nonmagnetic above 15.4 GPa (Supplementary Fig. S4). The magnetic change can be attributed to the bcc-hcp phase transition. No macroscopic magnetic change is observed around 5 GPa, indicating it would not be the reason for ISTP in Fe-C alloys. In contrast, XES provides a way to probe local magnetic momentum. The K_β emission is due to a radiation decay of the 1s core hole from a 3p level. Due to a net magnetic moment (μ) in the 3p-3d exchange interaction, the K_β emission spectrum splits into a main line $K_{\beta 1,3}$ and a satellite line $K_{\beta'}$. The intensity of $K_{\beta'}$ is proportional to the net spin of the 3d shell of the transition metal. It had been reported that the $K_{\beta 1,3}$ of bcc-Fe peaked at ~ 7057 eV during compression and had negligible pressure-dependence, however, the $K_{\beta'}$ decayed as pressure increased^{47,48}. For the case of Fe-0.2C (Fig. 2), its $K_{\beta 1,3}$ peak shifts to the lower energy as pressure increases. The shift can be attributed to the responses of Coulomb repulsion to the pressure, indicating the incorporation of C into Fe raise the sensitivity of Coulomb repulsion to pressure. A weaker broad satellite $K_{\beta'}$ peak is observed for Fe-0.2C compared with that of Fe at same pressure condition. Unlike the gradual decay of $K_{\beta'}$ in Fe⁴⁷, $K_{\beta'}$ in Fe-0.2C slightly increases from 1.3 GPa to 3.4 GPa and then generally decays to 11.5 GPa. We calculated the integrals of the absolute values of the difference spectra (IAD)⁴⁹ to analyze the shift of K_β of Fe-0.2C by using the data at 11.5 GPa as the reference spectrum. Fig. 2(C) shows the IAD values of Fe-0.2C under high pressure conditions. The IAD value increases as a function of pressure below 4.6 GPa, while a sudden drop in the IAD is observed at 5-6 GPa. Above 5-6 GPa, the IAD value monotonously decreases as a function

of pressure. This feature differs from that of Fe, whose IAD monotonously decreases in the bcc-stabilized pressure range. The XES results illustrates that the incorporation of C into Fe induces a sudden decrease of Coulomb repulsion between Fe-Fe atoms, which might be responsible for the observed ISTP in Fe-C alloys (Fig. 1).

High-temperature behaviors of Fe-C alloys

The observed ISPT in Fe-1.7C with mixed bcc and fcc phases at room temperature indicates that the ISTP may also occur in fcc-Fe-C. We conducted high-temperature acoustic velocity measurements of fcc-Fe-C alloys up to 1300 K (Fig. 3). Above 1000 K, both Fe-0.2C and Fe-1.7C are in fcc phase (Supplementary Fig. S5 and S6). The V_P of fcc-Fe-0.2C is 5.4 km/s below 4.5 GPa. It is about 1.0 km/s lower than that of its bcc-I counterpart. Temperature has invisible effect on V_P in these P-T conditions. However, significant temperature effect can be observed in the V_S (Supplementary Fig. S7). At 1100 K, the V_S is ~2.5 km/s, while it is ~2.2 km/s at 1300 K. We did not obtain V_P datum for fcc-Fe-0.2C above 4.5 GPa, but the sudden V_S drop around 5.3 GPa indeed confirm the ISPT in fcc phase. The V_S is ~2.0 km/s at 1100 K, which is ~0.5 km/s lower than its lower pressure (<4.5 GPa) counterpart. We denote the ISPT in fcc phase as fcc-I and fcc-II. The fcc ISTP can be more clearly observed in Fe-1.7C. Below 4.0 GPa, the V_P and V_S of fcc-I Fe-1.7C are ~5.0 km/s and ~2.6 km/s at 1100 K, respectively. While above 4.0 GPa, the V_P and V_S drop to ~4 km/s and ~2.1 km/s, respectively.

Implications for the lunar core

As mentioned above, selenophysical observations¹⁰⁻¹², thermodynamic constraint^{9,10}, lunar dynamo model¹⁴⁻¹⁶, paleomagnetism¹³, and phase equilibria¹⁷ suggested an inner core in the Moon's deep interior. The seismic velocities estimated from the Appollo data are ~4.3 km/s for V_P and ~2.3 km/s for V_S or lower^{5,11}. Lunar core conditions (5-6 GPa and 1300-1900 K)²⁹ favor fcc-Fe in its inner core⁵⁰⁻⁵³. The V_P of pure fcc-Fe has been measured up to 19 GPa and 1100 K²⁹ using inelastic X-ray scattering and 2.8 GPa and 1073 K using Pulse-echo overlap technique²⁸. At lunar inner core conditions, the V_P of fcc-Fe is ~5.6 km/s with estimated V_S of ~3.2 km/s. The gap between seismologic and mineralogic constraints in the lunar core leads to an outstanding issue in determining the existence and

the composition of lunar inner core, especially considering the conventional wisdom that the incorporation of light elements would boost the acoustic velocities of solid iron^{40,42-46,54}. S was suggested the main light element in the lunar outer core^{7,19-21,55,56}. However, the content of S in the lunar inner core would be negligible, because the partitioning coefficient of S between solid and liquid Fe increases from 0 at ~20 GPa to 0.1 at ~40 GPa²⁵⁻²⁷. The ISPT in Fe-0.2C and Fe-1.7C could induce a V_P collapse from 5.0-5.5 km/s to ~4-4.5 km/s and the V_S to ~2.1 km/s. These data are in good agreement with the inferred seismic velocity profile of the lunar core when the data scattering is considered, which means the incorporation of 0.2-1.7 wt.% C into fcc-Fe could reproduce the Apollo seismically inferred velocities of the lunar core above 5 GPa (Fig. 3). The C-rich lunar core deduced from the acoustic velocity of fcc-II Fe-C is also supported by phase equilibria experiments in Fe-S-C system^{17,57} and mass balance calculations combined with metal-silicate partitioning and bulk silicate Moon abundance of C³¹. The melting experiments of low C content Fe-C-S ternary system⁵⁸ showed that the C partitioning coefficient in liquid and solid Fe is around 0.5. If there is 0.2-1.7 wt.% C in Moon's inner core, the C content in its liquid outer core would be 0.1-0.8 wt.% (Fig. 4). It is reported that 4-10.5 wt.% S^{7,19,21} is required to reproduce the V_P of the Moon's outer core^{14,21,23}. The incorporation of C would not significantly affect the structure of Fe-S liquid, but slightly increase its density and sound velocity^{59,60}. That means more S is required in the lunar outer core to compensate the effect of carbon. The carbon solubility in Fe-12.5S melts is up to 2 wt.% according to the melting experiments⁵⁷. It is compatible for 0.1-0.8 wt.% C co-exist Fe-S melt in the lunar outer core.

Compared with V_P , the V_S of Fe-C is more sensitive to the temperature (Supplementary Fig. S7). The temperature-dependence of V_S can be employed to constrain the temperature of the lunar core if carbon is the sole light element. A reduction of $\sim 0.7 \times 10^{-3}$ km/K for V_S can be deduced from the results in Supplementary Fig. S7. The V_S of fcc-II Fe-C is around 2.1 km/s at 1300 K and lunar core pressure. Both V_P and V_S of fcc-II Fe-C at 1300 K could reproduce the seismic velocities of the alleged lunar inner core. As temperature increases to 1500 K, although the V_P for fcc-II Fe-C is comparable to the Apollo seismic estimation, the upper bound of V_S for fcc-II Fe-C is below the inferred seismic velocity of the lunar inner core, indicating the temperature profile of the lunar core is likely smaller than 1500 K. We calculated the density of fcc-Fe-0.2C at 5 GPa and 1300-1500 K according to the thermal equation of state under the assumption of interstitial substitution of C in Fe lattice⁶¹. The constrained

density of lunar inner core is 7.71–7.82 g/cm³, which is in good agreement with the constraint from tidal deformation ($7822 \pm 1615 \text{ kg/m}^3$)¹⁰.

Method

Sample preparation: The Fe-0.2C and Fe-1.7C are synthesized by piston cylinder apparatus at 1 GPa and 1273 K with iron powder and graphite (purity 99.9%, purchased from Alfa Aesar Company). All sample are 2 mm in diameter and 5 mm in height. The chemical composition of the synthesized sample was analyzed using EPMA-8050G Electron Probe Microanalyzer at Shanghai Institute of Measurement and Testing Technology. The electron beam conditions are 15 kV and 200 nA with 10 s peak counting time and 10 s background counting time. Five well-characterized Fe-C alloys with carbon contents of 0.0067, 0.160, 0.382, 0.620, and 0.870 wt.% were used as standards to establish a calibration curve for carbon measurement. Neither the sample nor the standards were coated by conductive layer in order to prevent carbon contamination, while charging was prevented by carbon tape. LiF/CH₄ and PbST/CH₂ were used for carbon and iron analysis, respectively. The measured carbon contents of the two synthesized samples range from 0.15-0.26 wt.% with an average of 0.2 ± 0.06 wt.% (denoted as “Fe-0.2C”) and 1.65-1.81 wt.% with an average of 1.7 ± 0.11 wt.% (denoted as “Fe-1.7C”) (Fig. S1).

Acoustic velocity measurements: Synthesized samples were carefully cut to disks with thickness of ~0.7 mm. All samples were finely double-polished with 6 μm diamond lapping film. The final thicknesses of all samples were about 0.65 mm. The experiments were conducted using the Paris-Edinburgh press at 16-BM-B, High Pressure Collaborative Access Team (HPCAT) at the Advanced Photo Source, Argonne National Laboratory. Two types of PE cells are employed (Supplementary Fig. S2). Sintered diamond buffering rod and SiO₂ backing were used for room temperature measurements, while alumina buffering rod and backing were used in the high temperature cell. The buffer rod and the backing were placed next to the sample, and ultrasonic signals were reflected between the interfaces. An MgO ring surrounded the sample and it was used to calibrate the pressure, with uncertainties of ~0.3 GPa at room temperature and 0.5 GPa above 1000 K⁶². The temperature was read by pre-

calibrated power-temperature relations⁶³. Pulse-echo overlap technique was employed to obtain the ultrasonic V_P and V_S signals. An electrical sine wave was generated by waveform generator. Ultrasonic signals from 12 MHz to 40 MHz were collected with a digital oscilloscope (Tektronix DP05104). The wave was reflected in the buffer-rod/sample and sample/backing, which were R_1 and R_2 for both P-wave and S-wave (Supplementary Fig. S3). The travel time (t) of ultrasonic signal in the sample could be determined by R_1 and R_2 . Sample length (l) was measured by white X-ray radiography image. The acoustic velocity can be calculated by

$$V_P \text{ (or } V_S) = \frac{2l}{t} \quad (1)$$

Where l is the length of the sample, and t is the travel time of ultrasonic signals determined by R_1 and R_2 , respectively. The uncertainty of acoustic velocity is $\sim 0.5\%$. The detailed method can be found in ref. ⁶². The energy-dispersed x-ray diffraction (EDXRD) patterns were collected by a Ge solid state detector at $2\theta = 15^\circ$. The collection time ranged from 30 s to 200 s.

X-ray emission spectra (XES): The XES were performed at 16-IDD, HPCAT. A symmetric diamond anvil cell with culet size of 300 μm was used to generate high pressure for XES measurement. A rhenium gasket was pre-indented at 20 GPa and a 150 μm chamber hole was drilled by laser ablation. A ~ 25 μm thick Fe-0.2C pellet with diameter of ~ 70 μm was loaded into the chamber. Silicon oil served as pressure medium. The pressure was determined by ruby fluorescence with an uncertainty of 0.1 GPa. All XES experiments were conducted at room temperature. An incident X-ray beam with an energy of 11.3 keV and a bandwidth of ~ 1 eV was employed for the experiments. The collection time was ~ 40 minute for each spectrum.

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AUTHOR CONTRIBUTIONS

Conceptualization, B.C., J. L., and M.H.; investigation, M.H., H.L., Y.K., J. L., and X.C.; visualization,

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257 L; funding acquisition, M.H., X.S., S.N., and H.S.; project administration, M.H. and X.S.; writing –
258 original draft, M.H. and H.L.; writing – review & editing, all authors.

259

260 **DECLARATION OF INTERESTS**

261 The authors declare no competing interests.

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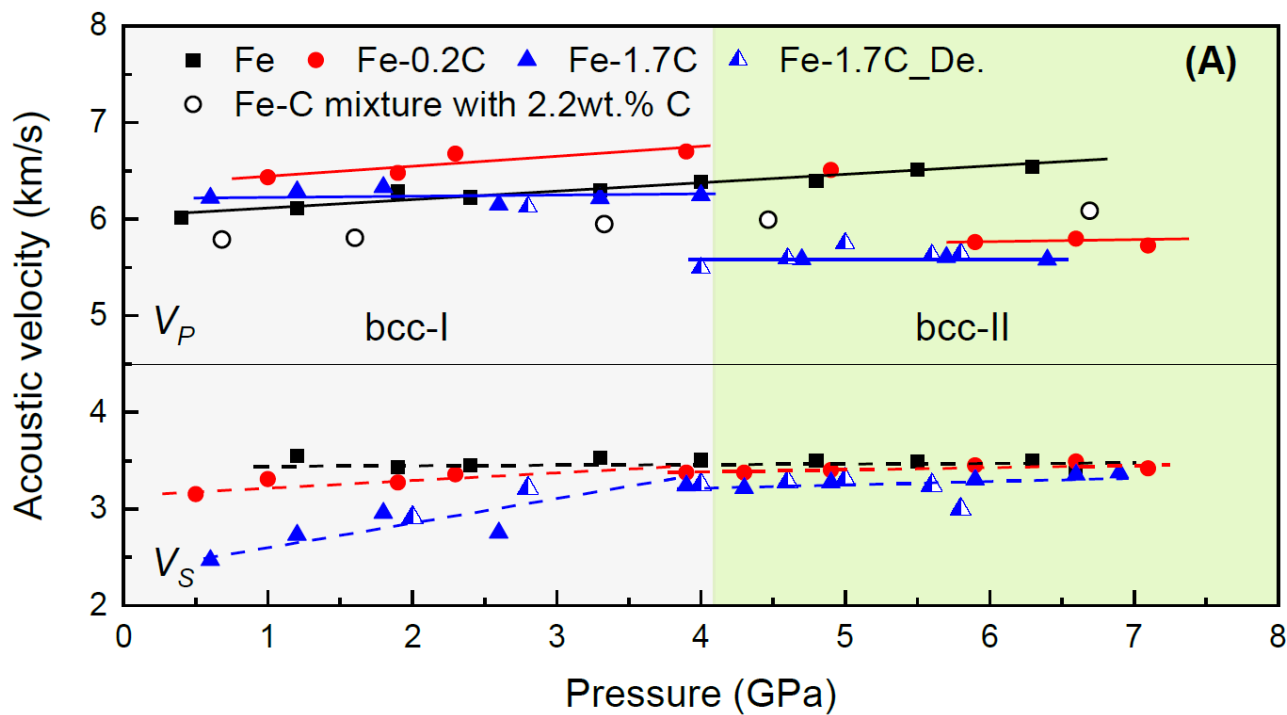
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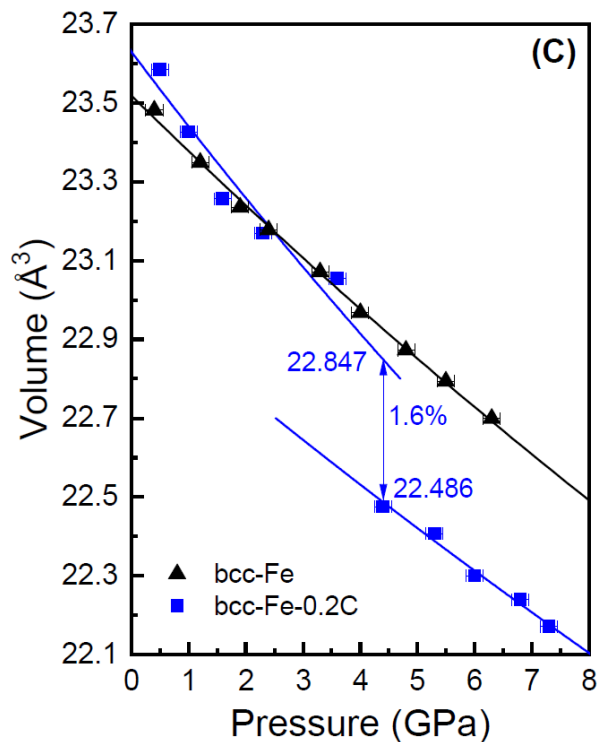
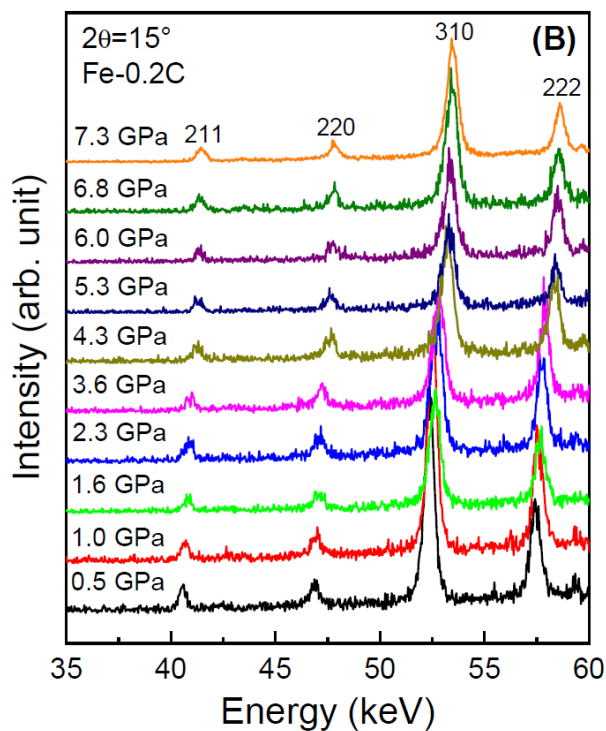
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388 **Fig. 1. The acoustic velocity and X-ray diffraction characterizations of Fe and Fe-C alloys at**
389 **room temperature.** (A) Acoustic velocities of bcc-Fe²⁸, bcc-Fe-0.2C, and bcc-Fe-1.7C. De. is short
390 for decompression. The open black circles represent V_P of Fe-C mixture with 2.2wt.% C⁴¹. The black,
391 red, and blue curves are hand-drawn relations of acoustic velocities and pressure for bcc-Fe, bcc-Fe-
392 0.2C, and bcc&fcc-Fe-1.7C, respectively. (B) X-ray diffraction of Fe-0.2C. The intensity and full-

393 width at half maximum of each peak are nearly same, indicating the deviatoric stress and preferred
394 orientation of the sample are minimal; (C) P-V relations of bcc-Fe and bcc-Fe-0.2C. The volumes of
395 bcc-Fe and bcc-Fe-0.2C are calculated according to peaks of (211), (220), (310), and (222).

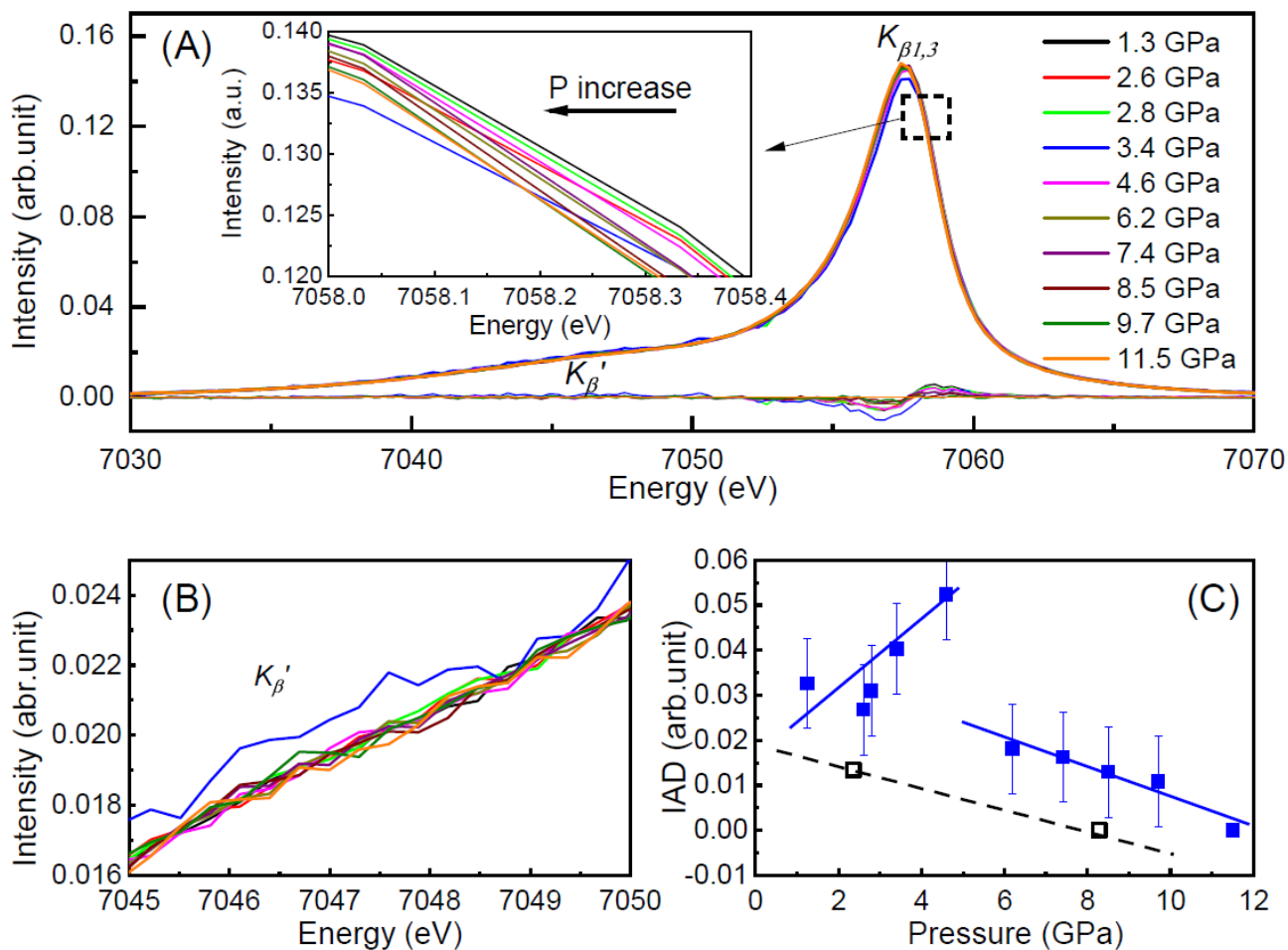


Fig. 2. X-ray emission spectra of Fe-0.2C at 1.3-11.5 GPa and room temperature. The bottom curves in (A) are the differences of spectra compared with the reference spectrum at 11.5 GPa. The inset in (A) is magnitude of the spectra labeled in the dash rectangle. (B) Magnitude of the satellite peaks (K_{β}'). (C) Integrals of the absolute values of the difference spectra for Fe-0.2C and Fe. The closed and open squares are for Fe-0.2C and Fe⁴⁷, respectively. The solid and dash lines are guides to the eyes.

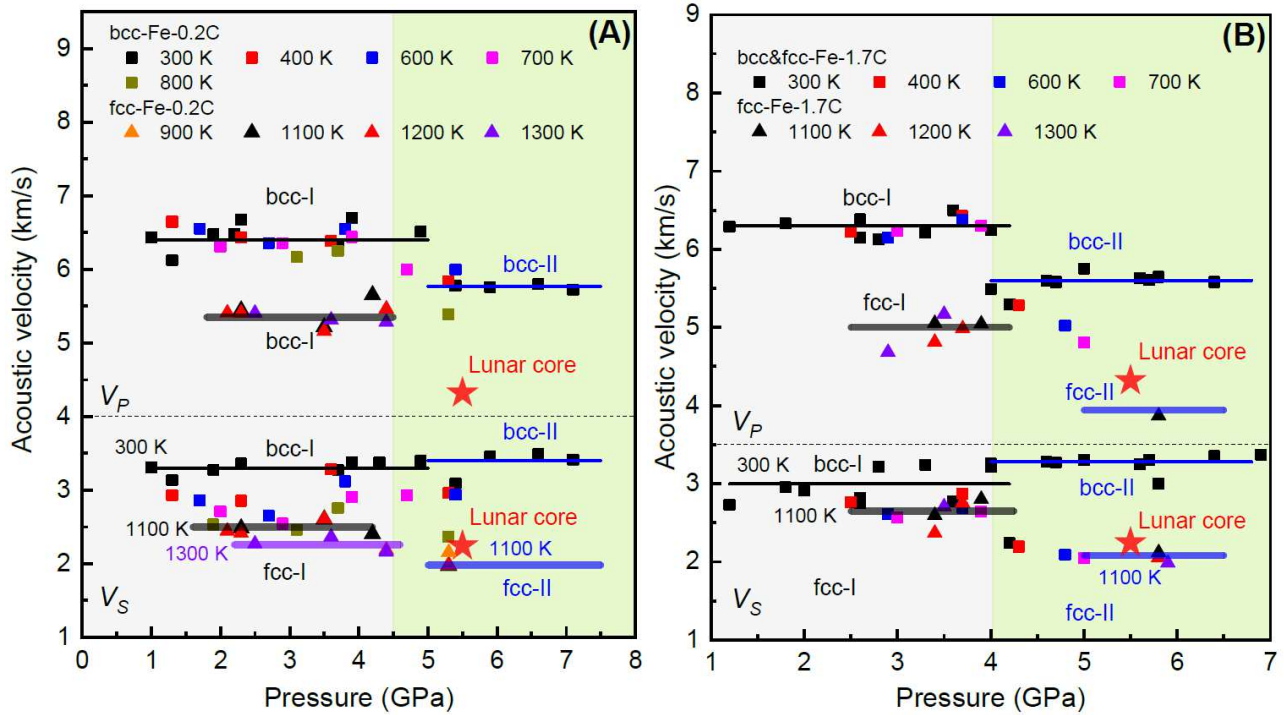


Fig. 3. Acoustic velocities of Fe-0.2C (A) and Fe-1.7C (B) at elevated temperatures. The black, blue, bold gray, bold blue curves (solid and dash) are hand-drawn variations of bcc-I, bcc-II, fcc-I, and fcc-II Fe-C alloys, respectively. The red star represents the V_P and V_S of the lunar core ⁵.

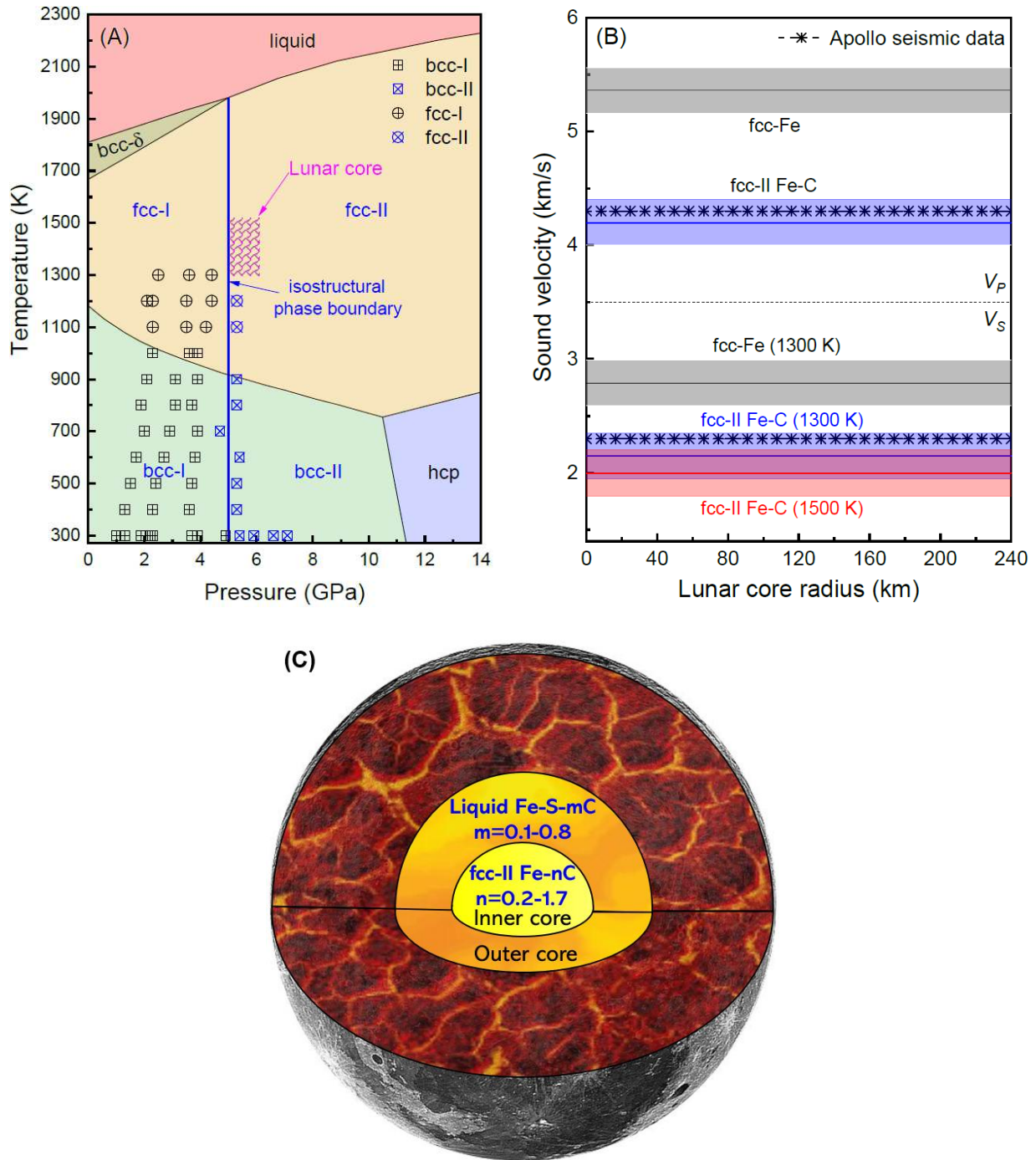


Fig. 4. The revised phase diagram of Fe (A), comparison of sound velocities of fcc-Fe, fcc-II Fe-C, and the lunar core (B), and the structure and chemistry of the lunar core (C). The phase diagram of iron is from ref. ⁵⁰. The circles and rectangles with crosses are phases of the sample at corresponding P - T conditions. Blue vertical lines are phase boundaries of bcc-I/bcc-II and fcc-I/fcc-II. Lunar core P - T conditions are represented by the pink shaded area in (A). The shaded areas in (B) are uncertainties of sound velocity from fitting error caused by data scattering. The phase diagram for Fe-1.7 C can be found in the supplementary materials. No visible temperature-dependence for V_P of fcc-

418 II Fe-C is observed in supplementary Fig. S7. We assume V_P is independent on temperature in **(B)**.

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