REPORTS

Anomalous Behavior of Sound Velocity and Attenuation in Liquid Fe-Ni-S

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The compressional wave velocity in molten iron containing 5 percent nickel and 10 percent sulfur increased with increasing temperature. This anomalous behavior was determined to be attributable to the presence of sulfur, which conditions the formation of large molecular units in the liquid. Temperature-induced breakup of macromolecular units resulted in greater packing efficiency and an increased velocity. Ten percent sulfur increased attenuation by one to two orders of magnitude compared with liquid iron. Such behavior at outer core pressures and temperatures would constrain the velocity gradient in the outer core and would enable the discrimination of potential light alloying elements.

An iron-nickel sulfide alloy (Fe-Ni-S) is believed to be a possible component of the cores of planetary bodies such as Earth, Mars, and Io (1). Since the first evidence of a metallic liquid outer core for Earth was found from seismological studies (2), the properties of liquid Fe and Fe-Ni alloys containing plausible light elements such as S, Si, and O have been studied at high pressures and temperatures (3) to characterize the physical and chemical properties of the outer core. In particular, the density, coefficient of thermal expansion, sound velocity and attenuation, and related thermodynamic properties of these melts are all basic inputs for modeling the thermal state and dynamics of planetary cores. We conducted an ultrasonic interferometry investigation of molten Fe-5%Ni-10%S at ambient pressure, P, in the temperature, T, range of 1673 to 1973 K. The interferometric method measures simultaneously the compressional wave velocity, c, in molten materials (4, 5) and the attenuation (or conversely, the quality factor, Q_p) (6, 7). Acoustic interferometry is based on the detection of a standing wave formed within a sample by interference of internal reflections of the wave train (8). The method is described in detail elsewhere (7, 9, 10).

About 80 g of Fe-5%Ni-10%S (11) was loaded into the high-temperature chamber of the interferometer (12). The chamber was evacuated to 10^{-6} torr, and the sample was slowly heated at a rate of 5 K min⁻¹. At 1473 K, the chamber was isolated from the pumping system, and Ar gas was slowly introduced to a final P of 1 to 1.2 bar. The T was then increased to 1773 K. The upper buffer rod was lowered into the liquid and allowed to stand for at least 1 hour before beginning the measurements. Experimental corrections for changes in sample thickness as a result of thermal expansion and contraction of the rods were made (9). Data were acquired every 25 K from 1673 to 1973 K on both increasing and decreasing T paths. An equilibration time of about 1 hour was allowed at each new T setting before proceeding with the next measurement. The T stability throughout a run was better than ± 1 K.

Interferometric measurements on liquid Fe-5%Ni-10%S at frequencies of 9.66 MHz and 18.13 MHz (Fig. 1) yielded values of c that were then fitted by least squares with a second-order polynomial in the T range 1673 to 1973 K:

$$c(T) = 3133 \left[1 + 0.395 \left(\frac{T}{T_{\rm m}} - 1 \right) - 0.154 \left(\frac{T}{T_{\rm m}} - 1 \right)^2 \right] {\rm m \ s^{-1}} \qquad (1)$$

where $T_{\rm m} = 1650$ K is the melting T (13). Unlike *c* for Fe or Fe-Ni (9, 14), *c* for Fe-5%Ni-10%S behaves anomalously in the sense that *c* increases with increasing *T*. This anomalous effect we attributed to the presence of S. It is known that liquid semimetals or semiconducting elements such as Si, Ge, Sb, and Te display an increase in *c* with increasing *T* (15, 16). It is also known that a maximum in *c* exists in H₂O at 74.2°C (17). All these materials trend toward closer packing on melting, with a concomitant decrease in volume (for example, H₂O, Si, Ge, and Sb) or an anomalously small change in volume (for example, Te) with increasing T. In addition, some semiconductors, such as Te, acquire a metallic conductivity upon melting, and the number of free electrons increases with increasing T. Such an electronic delocalization process can increase the bulk modulus, K, of the liquid and increase c, because $c = (K/\rho)^{1/2}$ (where ρ is density) (4, 15). In liquid S at 1 atm, c decreases nonlinearly with increasing T, which has been associated with structural rearrangements through the breakup of polymolecular formations (18). At P as low as 12 GPa, liquid S undergoes discontinuous changes in volume and electrical conductivity that have been attributed to liquidliquid polymorphism (19). This anomalous behavior of S may control phase relations and physical properties of S-bearing Fe liquids and solids. At high P, a densification resulting from an electronic transition (without structural change) was reported in FeS at about 6 GPa and 600 K (20). Furthermore, a continuous semiconducting-tometallic transition in monosulfide solid solution in the Fe-Ni-S system was observed under high P and T (21).

We considered the potential for immiscibility (22) or two-liquid segregation as a cause of the anomalous behavior in c. Immiscibility would cause stratification of the phases. With such a two-layer liquid, a trend of gradual change in peak spacing (that is, wavelength) on an interferogram as the upper buffer rod passes through the acoustic boundary between the layers should have been detectable with our interferometer. Such a trend could not be resolved in our data, even at the highest T of 1973 K where immiscibility should be most pronounced (22), so we conclude that immiscibility does not occur. The possibility that the anomalous behavior of *c* was related to sulfur loss was also considered (23). However, the repeatability among seven c measurements at 1673 K, taken over 3 days (Fig. 2), precludes any appreciable loss of S.

> Fig. 1. Example interferograms for Fe-5%Ni-10%S at 9.66 MHz, 1973 K (A) and at 18.13 MHz, 1673 K (B). The dots are experimental data, and the solid lines are theoretical fits.



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Using our measured value of $c = 3113 \text{ m s}^{-1}$ at $T_{\rm m} = 1650 \text{ K}$ and $6.5 \pm 0.5 \text{ g cm}^{-3}$ (24) for the 1-atm density, we obtain a value of 63 GPa for K. This is substantially lower than the ~110 GPa for K of liquid Fe at 1 atm (14), which highlights the large effect of small amounts of S on the elastic properties of S-bearing Fe melts.

In general, c of a material increases with increasing P because K, that is, the stiffness of the material, increases with P(dK/dP > 0)faster than density. In contrast, c generally decreases with increasing T because K decreases with T (dK/dT < 0) as a result of thermal expansion effects. In other words, P and T normally have opposite effects on c. However, because of the anomalous T behavior of *c* in Fe-5%Ni-10%S, both *P* and *T* in this system may act in the same direction in modifying c. Because the adiabatic T gradient with depth, dT/dz, is positive in the outer core, it is expected that a hypothetical Fe-5%Ni-10%S outer core, behaving at the high P and T in the outer core in a way similar to the liquid composition studied here at 1 atm and high T, should have a steeper velocity-depth gradient than a "normal" (in the present context) outer core. This could provide a means of discriminating between potential candidates for the light alloving element or elements.

The dimensionless compressional (or bulk) quality factor in a viscoelastic liquid, $Q_{\rm p}$, is given by

$$Q_{\rm p} = \left(\frac{\alpha}{f^2}\right)^{-1} \left(\frac{\pi}{cf}\right) \tag{2}$$

where α is the coefficient of attenuation. $Q_{\rm p}$ for Fe-5%Ni-10%S is the same for the two frequencies investigated (Fig. 3) and is in contrast to the dependence on f^{-1} commonly observed for relaxed liquid metal systems (25). In metal alloy melts, however, an additional absorption mechanism is caused by concentration fluctuations (26), which are frequency dependent and controlled by the size and mobility of the structural units. The frequency-independent $Q_{\rm p}$ values (Fig. 3) suggest that the concentra-



Fig. 2. Compressional sound wave velocity, *c*, versus T for 9.66 MHz (solid circles) and 18.13 MHz (open squares). The solid line is a second-order least squares fit (Eq. 1). The measured *c* at high T by means of ultrasonic interferometry is precise to within $\pm 0.6\%$ (9).

tion fluctuations occur at frequencies lower than those probed, that is, on time scales greater than 5×10^{-8} s. With an apparently similar effect on *c* and *K* as for other properties such as surface tension and $T_{\rm m}$, S drastically reduces the magnitude of $Q_{\rm p}$ relative to the $Q_{\rm p}$ for Fe (27). The addition of 10% S produces an increase in attenuation by about one to two orders of magnitude. A linear least squares fit for the *T* variation of $Q_{\rm p}$ in the range of 1673 to 1973 K gives

$$Q_{\rm p}(T) = 661 \left[1 - 0.195 \left(\frac{T}{T_{\rm m}} - 1 \right) \right]$$
 (3)

The decrease in Q_p with the addition of S is consistent with recent measurements of the shear viscosity of liquid Fe-27%S at *P* between 2 GPa and 5 GPa (28). The viscosity values for liquid Fe-27%S were three orders of magnitude greater than the viscosity of liquid Fe and were interpreted as due to the structural control of S to the extent that it conditions the formation of large, macromolecular viscous flow units. Temperatureinduced breakup of molecular associations would result in additional energy loss mechanisms that could account for the negative dependence of Q_p on *T*.

The velocity and quality factor in a viscoelastic medium are related to the dynamic longitudinal viscosity, η_l , through the relation (29)

$$\eta_l = \frac{\rho c^2}{2\pi f Q_p} \tag{4}$$

Because c and Q_p are independent of f (Figs. 2 and 3, respectively), η_l becomes frequency-dependent and should vary as f^{-1} . Using our measured values at $T_m = 1650$ K for c = 3113 m s⁻¹ and $Q_p = 661$, and $\rho = 6.5 \pm 0.5$ g cm⁻³ (24) for the 1-bar density, we obtain

$$\eta_l = \frac{(1.5 \pm 0.3) \times 10^7 \,\mathrm{Pa}}{f} \tag{5}$$

Because seismic frequencies are typically within an order of magnitude of 1 Hz, η_l given by Eq. 5 is within an order of magnitude of 1.5×10^7 Pars. This value is consistered



Fig. 3. Compressional quality factor, Q_p , versus *T* at 9.66 MHz (solid circles) and 18.13 MHz (open squares). The solid line is a linear least squares fit (Eq. 3). The scatter in the data is less than $\pm 10\%$, with a mean standard deviation of $\pm 4\%$.

tent with seismological studies of *P*-wave attenuation, which give upper bound viscosity values in the range 10^7 to 10^8 Pa·s (30).

Furthermore, η_l is related to the bulk viscosity η_v and the shear viscosity η_s by $\eta_l = \eta_v + 4 \eta_s/3$ (29). To our knowledge, η_s has not been measured for Fe-5%Ni-10%S; however, the experimental value of n. in liquid Fe-8.51%S at 1873 K and 1 bar is $\sim 4 \times 10^{-3}$ Pa·s (31). This value should not vary significantly from η_s in Fe-5%Ni-10%S, and because η_1 from Eq. 4 is 0.75 ± 0.15 Pa•s at 20 MHz, we conclude that $\eta_1 \approx$ η_v . This result is in marked contrast to that of Fe and most liquid metals where η_v is nearly equal to η_s (4, 29, 31). In order to account for the large η_v , a relaxation mechanism, in addition to structural rearrangements, such as possible intra-macromolecular process, slow equilibrium adjustment of molecular associations, and concentration fluctuations ought to take place in liquid Fe-5%Ni-10%S.

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- 8 In essence, a long pulse of known frequency, f, is transmitted through two vertical 20-cm-long buffer rods separated by a layer of melt. Acoustic impedance contrast between the rods and the melt generates multiple reflections within the melt layer that interfere with each other. A resonant, constructive standing wave appears as a maximum on a plot of amplitude versus melt thickness, L, and is formed for L equal to integral multiples of half the wavelength, $L_{\rm max} = n\lambda/2$, where λ is the ultrasonic wavelength in the melt. Similarly, an anti-resonant, destructive condition appears as a minimum and occurs when the rod-to-rod separation (that is, the melt thickness) corresponds to $L_{min} = (n + 1/2)\lambda/2$. When the upper buffer rod is moved up and away from the fixed lower buffer rod, the transmitted amplitude, A(L), of the ultrasonic signal traces out an alternating constructive $[A(L_{max})]$ is maximum] and destructive $[A(L_{min})]$ is minimum] interference pattern. Measured L and A(L)values are used to construct an amplitude versus melt thickness plot (interferogram). A theoretical expression for A(L) (7, 9) is then fitted to the data, and a



matrix inversion algorithm is used to extract the melt velocity and specific attenuation, Q_p^{-1} . P. M. Nasch, thesis, University of Hawaii, Honolulu

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- 12. The liquid sample was contained in an Al_2O_3 ceramic crucible penetrated by, and cemented to, a lower buffer rod made out of dense, polycrystalline Al₂O₃. The crucible was enclosed in a graphite sleeve that acted as an inductive load for the radio-frequency (rf) induction heater. A ZrO2 radiation shield provided insulation for the graphite sleeve. The sample T was monitored by an Al2O3-sheathed W-Re thermocouple located in the melt. Another protected thermocouple, also located in the melt, was connected to a temperature controller that controlled the furnace power supply. A clear-fused quartz tube was placed around the high-temperature assembly and was sealed at both ends by water-cooled flanges. The movable upper buffer rod was a 20-cm-long by 1.27cm-diameter single-crystal sapphire rod. In preparation, both ends of both rods were polished to achieve a 1-µm surface finish and flatness and a mutual perpendicularity to the rod axis of $\pm 0.01^{\circ}$ (1.2 min). Before each experiment, a trial run was undertaken at room conditions with distilled water (H₂O) or glycerol (C3H8O3) to identify misalignment of the buffer rods (asymmetry in the resonance peaks), poor transducer bonding or electronic tuning (low overall transmitted amplitude), and lack of parallelism or flatness of the rod faces (ripples in the decay pattern).
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Inducible Expression and Phosphorylation of Coactivator BOB.1/OBF.1 in T Cells

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BOB.1/OBF.1 is a transcriptional coactivator that is constitutively expressed in B cells and interacts with the Oct1 and Oct2 transcription factors. Upon activation of Jurkat T cells and primary murine thymocytes with phorbol esters and ionomycin, BOB.1/ OBF.1 expression and transactivation function were induced. BOB.1/OBF.1 was phosphorylated at Ser¹⁸⁴ both in vivo and in vitro, and this modification was required for inducible activation. Mutation of Ser¹⁸⁴ also diminished transactivation function in B cells, suggesting that the activating phosphorylation that is inducible in T cells is constitutively present in B cells. Thus, BOB.1/OBF.1 is a transcriptional coactivator that is critically regulated by posttranslational modifications to mediate cell typespecific gene expression.

The octamer motif is a critical element for constitutive B cell-specific gene regulation (1). Its function is dependent on the combinatorial activity of ubiquitous or lymphoid-specific octamer (Oct) transcription factors and B cell-restricted transcriptional coactivators (2-7). One such coactivator named BOB.1, OBF.1, or OCA-B (BOB.1/ OBF.1) interacts with the POU domains of the Oct1 and Oct2 transcription factors, even in the absence of DNA, and mediates transcriptional activation from octamer-dependent promoters (5, 6, 8). Complexes containing BOB.1/OBF.1 and Oct proteins are selective for a subset of octamer motifs (9), and this is at least in part due to direct

contacts of the NH2-terminal domain of BOB.1/OBF.1 with the major groove of the DNA (10). Mutation of the gene for BOB.1/OBF.1 results in a severe defect in terminal B cell differentiation, strongly reduced responses to both T cell-independent and T cell-dependent antigens, and a lack of germinal-center formation (11).

Several genes that show regulated expression in T cells contain functional octamer motifs within their proximal promoter regions (12-14). These elements are required for inducible transcription. In some cases, like the proximal interleukin-2 (IL-2) promoter, octamer motifs are in close proximity and functionally cooperate with bind-