m. This increase is a reflection of the destruction of the fragile juvenile tests in the sediments at this depth by solution. Berger (*8*) has reported a similar selective solution of juvenile specimens under experimental conditions in the Pacific at depths greater than 1000 m. The basinward change from predominantly nonthickened to thick-walled populations is remarkably abrupt, taking place over a very short areal distance (Fig. 2, A–D). Each of the four species exhibits the change from thin to thick-walled populations at a different depth from the other species. For each species, the depth at which the secondary thickening takes place is constant over the entire area of the northwest Gulf of Mexico.

Bé and Ericson (see *4*) have given 300 to 500 m as the depth below which secondary thickening takes place in living specimens of *Globorotalia truncatulinoides* (d'Orbigny). The upper limit of this 300- to 500-m zone is manifest in populations of this species from sediments in the northwest Gulf. The depths recorded at which thickening takes place in the remaining three species in this study undoubtedly represent the upper limit of the thickening zone in the bathymetric column for living specimens: *G. crassaformis* (Galloway and Wissler), 700 m; *G. cultrata* (d'Orbigny), 120 m; and *G. tumida* (Brady), 200 m. The destructive solution of juvenile specimens in deep water is further reflected in the distribution of specimens with the thickened test. Because secondary thickening is primarily an adult characteristic, the loss of juvenile nonthickened specimens at mesopelagic depths causes a corresponding increase in the percentage in the specimens with the secondary crust at the same depth (Fig. 2, A–D).

W. N. ORR
University of Oregon, Eugene

References and Notes

1. D. B. Ericson, M. Ewing, G. Wolin, B. Heezen, *Geol. Soc. Amer. Bull.* **72**, 193 (1961).
2. A. W. H. Bé, *Micropaleontology* **11**, 81 (1965).
3. ———, *Science* **145**, 823 (1964).
4. ——— and D. B. Ericson, *Ann. N.Y. Acad. Sci.* **109**, 65 (1963).
5. The tops of some 700 marine sediment samples (mostly shallow cores) were supplied by Scripps Institute, Lamont Observatory, Humble Oil Company, Shell Oil Company, Standard Oil Company, and Texas A & M.
6. In all samples, except those from shallow water with limited planktonic populations, the first 200 globorotalid specimens encountered in a random strew were counted. All specimens counted were measured.
7. A. W. H. Bé, *Micropaleontology* **6**, 373 (1960).
8. W. H. Berger, *Science* **156**, 383 (1967).

11 July 1967

Isotopic Analysis of Rare Gases with a Laser Microprobe

Abstract. *A ruby-pulsed laser and high-sensitivity mass spectrometer are used to analyze isotopic abundances of rare gases from microgram samples of polished sections. The feasibility of the technique is demonstrated by the analysis of primordial helium and neon from the Kapoeta and Fayetteville meteorites.*

The variation in the abundance of primordial rare gases from meteorites is extremely important in understanding the early history of meteorites, the solar system, and the earth. Excellent discussions of the subject have been presented recently by Signer and Suess (*1*), by Suess, Wänke, and Wlotzka (*2*), and by Pepin and Signer (*3*). Of particular importance is the extreme variation of the primordial rare-gas abundances between different portions of the meteorite. Consequently, a system was developed that now extends isotopic analysis of rare gases to the microgram range and allows a direct comparison between isotopic abundance of rare gases and other chemical, mineralogical, and petrographic data.

A ruby-pulsed laser (*4*) was used to volatilize, in vacuum, approximately 1 μg per pulse of material from polished sections of meteorite. The extracted gases were separated cryogenically and were then analyzed under static conditions with a high-sensitivity 60°-sector mass spectrometer.

Details of the experimental arrangement are shown in Fig. 1. Polished sections of the meteorites are set on a stainless-steel shelf mounted inside a Varian vacuum window. The vacuum window is connected to a Grenville Philips vacuum valve by flexible stainless steel tubing. The other side of the valve goes directly to the ionization region of the mass spectrometer. Pumping and baking the system at 200°C for 10 hours, with the samples removed from the system, resulted in a static pressure of 2×10^{-7} torr.

The Varian window is readily adaptable to a microscope stage mounted on the table rather than on the laser unit. The flexible stainless-steel tubing readily permits 3 degrees of freedom for focusing and alignment of the meteorite samples.

The ruby-pulsed laser is shown with a microscope attachment. Low magnification of the sample at $\sim 50\times$ and one pulse of the laser resulted in a crater diameter of ~ 100 μ and a crater depth of ~ 30 μ.

In order to test the feasibility of the technique and the possibility that ionized rare gases may be lost to the vacuum walls, ten pulses were fired and roughly 10^{-5} g of material was extracted from various dark portions of a polished section of the Fayetteville and Kapoeta meteorites. Primordial He^3, He^4, Ne^{20}, and Ne^{22} were measured; the results are given in Table 1 with previously published data that were measured from 0.1- to 1-g samples.

Table 1. Abundances of primordial helium and neon from the Fayetteville and Kapoeta meteorites (abundance $\times 10^{-6}$ cm^3 per gram at standard temperature and pressure).

Source	He^3	He^4	Ne^{20}	He^4/Ne^{20}	Ne^{20}/Ne^{22}
		Fayetteville			
Dark vein	3	20,000	130	150	11.2
Dark vein	4.7	22,000	160	140	11.9
Dark border	< 0.4	< 170	< 5		
Chondrule	< .3	< 170	< 5		
Literature					
(*5*)			307		10.6
(*5*)			164		10.8
(*6*)	7.2	20,300	50.4	402	12.6
(*3*)	6.65	22,500	62.4	360	12.3
(*3*)	7.05	21,000	55.0	380	12.1
(*3*)	2.0	7,000			12.0
		Kapoeta			
Dark vein	<0.4	1,300	23	57	12.0
Literature					
(*7*)	.38	1,360	24	56.5	14.0
(*8*)	.50	2,040	22.2	91.0	13.5
(*9*)	.52	2,220	23	96.5	12.8
(*1*)	.39	1,320	19.3	68.5	12.8
(*6*)	.53	1,560	17.2	91.0	13.0

Primordial He^4 and Ne^{22} abundances from a dark vein of Fayetteville are reproducible to within 12 percent. The poorer reproducibility of He^3 and Ne^{20} is attributed to the background of the mass spectrometer and sample line. The peak at mass-to-charge ratio (m/e) 3 was corrected 60 percent because of a large hydrogen background, and the peak at m/e 20 was corrected 30 percent because of a significant H_2O background.

The error in the absolute abundances of primordial helium and neon from Fayetteville and Kapoeta is currently difficult to estimate, primarily because of the uncertainty in the quantity of material volatilized by the laser. This uncertainty is estimated to be within a factor of 2. Furthermore, a large and variable hydrogen background produces an uncertainty of approximately 40 percent in the He^3 abundances. However, a comparison of the He^4 abundance of Fayetteville and the He^4 and Ne^{20} abundances of Kapoeta with previously published values is quite encouraging. In particular, the He^4/Ne^{20} ratio of Kapoeta is consistent within the experimental error (15 percent) to values previously reported. This experimental error is estimated from the reproducibility of the 70 percent correction applied to the Ne^{20} from the machine and sample blank. Likewise, the error of Ne^{20}/Ne^{22} ratio of Kapoeta is estimated to be 15 percent.

The discrepancy between He^4/Ne^{20} of Fayetteville and the previously published values is difficult at present to reconcile, especially in light of the generally good agreement demonstrated by the data from the Kapoeta meteorite. Several mechanisms could account for the discrepancy, but I would not like to speculate about these mechanisms until more measurements are obtained by this technique from other parts of the meteorite.

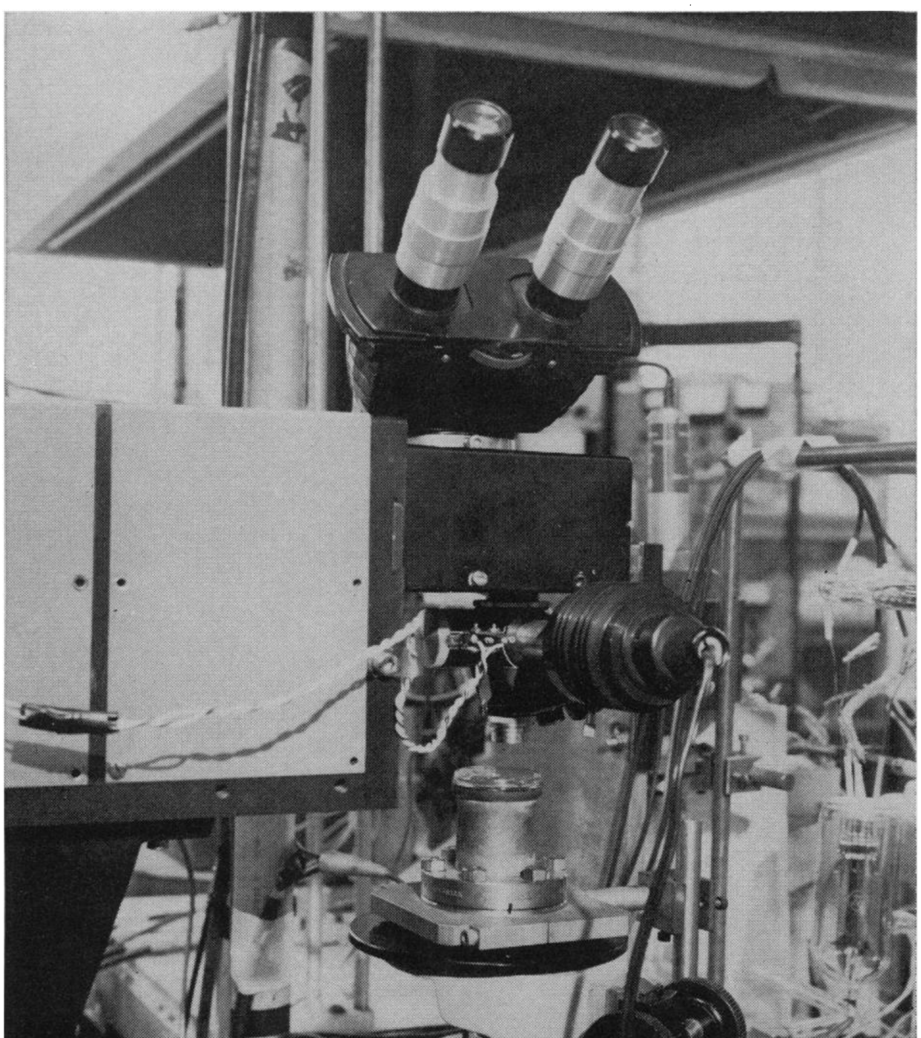

Fig. 1. The vacuum window is shown resting on a microscope stage and is connected by flexible stainless steel tubing to a vacuum valve (not shown). A Jarrell-Ash ruby-pulsed laser and microscope is focused on a meteorite sample inside the vacuum window.

From 10^{-5} g of a different dark phase and from 2×10^{-5} g of a chondrule from Fayetteville, no primordial helium and neon were detected above background. Consequently, not all dark phases of Fayetteville contain the same abundance of primordial rare gas. In fact, the highest abundances of primordial helium and neon in Fayetteville and Kapoeta appear to be confined to fine-grained matrix material. The variation of primordial helium and neon in different parts of the meteorite might be strictly a function of the amount of fine-grained matrix material in the dark phase. Future measurements of the isotopic abundance of primordial gas with a laser microprobe from other dark phases of the gas-rich meteorites, coupled with electron-microprobe, mineralogic, and petrographic data, are being carried out to explain more quantitatively the history and evolution of these meteoritic constituents.

With the improved sensitivity of mass spectrometers and sophistication of ultrahigh vacuum technology, the laser microprobe and mass spectrometer offer a powerful technique for exploring isotopic variations on a micron scale. Preferential loss of rare gases from the sample or to the vacuum surface does not appear to be a serious problem. Undoubtedly, this technique can be extended to the isotopic analysis of gases from minute samples of polished or thin sections from other extraterrestrial and terrestrial materials.

G. H. MEGRUE

Smithsonian Astrophysical Observatory and Harvard College Observatory, Cambridge, Massachusetts 02138

References and Notes

1. P. Signer and H. E. Suess, *Earth Sciences and Meteoritics* (North-Holland, Amsterdam, 1963), p. 241.
2. H. E. Suess, H. Wänke, F. Wlotzka, *Geochim. Cosmochim. Acta* **28**, 595 (1964).
3. R. O. Pepin and P. Signer, *Science* **149**, 253 (1965).
4. Jarrell-Ash Co., Waltham, Mass.
5. O. K. Manuel and P. K. Kuroda, *J. Geophys. Res.* **69**, 1413 (1964).
6. H. Hintenberger, H. König, L. Schultz, H. Wänke, *Z. Naturforsch.* **19a**, 327 (1964).
7. J. Zähringer and W. Gentner, *ibid.* **15a**, 600 (1960).
8. J. Zähringer, *ibid.* **17a**, 460 (1962).
9. T. Kirsten, D. Krankowsky, J. Zähringer, *Geochim. Cosmochim. Acta* **27**, 13 (1963).
10. I thank Dr. R. Pepin for his helpful criticism of the paper, Dr. R. Davis, Jr., for his discussion of the technique, Dr. E. Fireman for the sample of Kapoeta and his encouragement, and Dr. J. Wood for the Fayetteville sample.

14 August 1967