Reports

Nickel-Iron Spherules from Aouelloul Glass

Abstract. *Nickel-iron spherules, ranging from less than 0.2 to 50 microns in diameter and containing 1.7 to 9.0 percent Ni by weight, occur in glass associated with the Aouelloul crater. They occur in discrete bands of siliceous glass enriched in dissolved iron. Their discovery is significant tangible evidence that both crater and glass originated from terrestrial impact.*

The origin of Aouelloul glass is of current interest to investigators studying glasses derived from terrestrial impact of meteorites (impactites) and tektites possibly of lunar origin. The Aouelloul crater, 350 m in diameter, in Ordovician sandstone, is located at 20°15′N, 12°41′W in Mauritania, northwest Africa *(1)*. The glass found around the crater has been described (*2*). None of the later studies (*3, 4*), mostly of specific aspects of the Aouelloul glass, was very revealing regarding the origin of the glass.

Although the glass is associated with a well-developed and well-preserved crater, its origin is uncertain. The problem is threefold: (i) Is the crater a genuine meteorite crater? (ii) How was the glass formed? (iii) Is the glass derived from the bedrock underlying the crater?

Among recently established useful and diagnostic mineralogic and petrographic criteria for the identification of meteorite craters (*5*), those pertinent to the study of the origin of Aouelloul glass are: (i) the microstructures characteristic of minerals formed by shock at extremely high strain rates, (ii) occurrence of high-presure polymorphs, (iii) occurrence of nickel-iron spherules, and (iv) occurrence of breakdown or molten products of refractory accessory minerals. So far only baddeleyite, derived from decomposed zircon, has been reported from Aouelloul glass (*4*). We now report the presence of nickel-iron spherules in the glass.

In February 1964 we collected more than 2000 pieces of Aouelloul glass from the crater for examination in detail. Nearly 100 of the medium to large (2 to 5 cm long) glass samples were sliced into wafers 0.5 to 1 mm thick, which were examined under binocular and petrographic microscopes in search of nickel-iron spherules and inclusions and structures of special interest. As none of the samples of sandstone and quartzite collected at the crater showed macroscopic evidence of shock or impact metamorphism, it was hoped that some of the inclusions deeply embedded in the glass might contain high-pressure phases or bear evidence of shock.

Approximately 600 wafers of glass were so examined. Thin sections were made of 20 selected wafers, and polished sections were made of 30 others. Nickel-iron spherules were found in several wafers from a single glass specimen, which also contained a partly fused sandstone inclusion 4 to 5 mm long.

Figure 1 shows the occurrence and shape of the spherules; they range from about 0.2 μ, the limit of resolution of the optical microscope, to 50 μ in diameter; those larger than 20 μ are only partly spherical, with many protuberances; those smaller than 20 μ are almost perfect spheres. Each of two of the largest spherules yet recovered has a jagged side (Fig. 1A), giving the impression that it was torn from a larger molten globule and then quenched.

The spherules are clearly concentrated in narrow lenses or layers of bluish-gray to gray glass. The gray glass ranges from wisps or stretched lenses, a few microns in width, to bands up to 1 mm that extend throughout the length of the specimen. This discrete distribution of the spherules is the main reason why they were so difficult to find. Each gray lens or band is in turn embedded in a thin sheath of colorless glass within the normally brown glass (Fig. 2A). Under × 1000 magnification, a band of gray glass shows literally thousands of spherules, most of which are less than 0.5 μ in diameter; under incident light they sparkle like the stars of the Milky Way.

Of more than 50 spherules studied optically, ten were larger than 20 μ and more than half exceeded 5 μ. Unetched polished sections of them are pale yellowish-white in reflected light and are isotropic—properties consistent with nickel-iron. Nital etching of two large spherules revealed circular and slightly polygonal grain boundaries of kamacite within the spherule; no other phases were detected.

Several spherules extracted from the glass for x-ray study were strongly magnetic, almost perfectly spherical, silvery white, and bright and shiny. One spherule, measuring about 35 μ in diameter, was mounted on the tip of a blank x-ray spindle and x-rayed in a

Table 1. Nickel contents (within 5 percent) of 11 Ni-Fe spherules from Aouelloul glass; not corrected for absorption and fluorescence. Excitation voltage, 25 kv; specimen current, 0.01 μa.

Spherule No.	Spherule Diameter (μ)	Ni (wt %)
1	39	1.9
2	50	2.2
3	26	2.6
4	10	7.0
5	6	2.6
6	18	3.1
7	13	4.8
8	21	2.9
9	25	2.2
10	9	1.7
11	10	9.0

Table 2. Iron contents (within 5 percent) of gray glass (containing Ni-Fe spherules), clear glass, and brown matrix glass from an Aouelloul sample; not corrected for absorption and fluorescence. Excitation voltage, 25 kv; specimen current, 0.01 μa.

Spot position analyzed	Fe (%)
In gray glass	
20 μ from spherule 8	2.8
Between spherules 8 and 9	4.0
20 μ from spherule 9	3.2
5 μ*	2.6
30 μ*	2.0
In clear glass	
70 μ*	< 0.2
95 μ*	< .2
In brown glass	
125 μ*	1.9
155 μ*	2.0
195 μ*	1.8
260 μ*	1.8
325 μ*	1.8

* Traverse distance from spherule 9 toward viewer and normal to the boundaries of the gray, clear, and brown glasses.

Debye-Scherrer camera; the pattern showed a few rather sharp spots in both the front and back reflecting regions, indicating that the spherule contained a few crystals. The pattern however can easily be identified as kamacite or α-iron with a unit cell of $a = 2.865 \pm 0.005$ Å.

Eleven spherules, all larger than 5 μ, were analyzed with an electron microprobe, analyzed kamacite from the Canyon Diablo iron meteorite, with 7.0 percent Ni, being used as a standard. The results were not corrected for absorption and fluorescence because the matrix effect is similar to that of the standard used. The analyses show that the spherules consist essentially of iron, with a nickel content ranging from 1.7 to 9.0 percent (Table 1). These results resemble those for nickel-iron spheroids recovered from the Meteor Crater of Arizona [from 2 to 24 percent Ni (*6*)] and are consistent with a meteoritic origin for the spherules. We therefore conclude that the presence of nickel-iron in Aouelloul glass is proof that the crater is genuinely meteoritic.

Can we be sure that Aouelloul glass is not from a tektite, possibly of lunar origin, as tektites also contain nickel-iron spherules of comparable nickel content (*7*)? Study of nickel-iron spherules and of the variation of iron content in tektites (*8*) indicated that iron must be oxidized before it can be dissolved in a silicate melt. Brett (*9, 10*) has further shown that the matrix glass immediately surrounding nickel-enriched nickel-iron spherules in terrestrial-impact glasses often shows pronounced enrichment by iron, probably caused by oxidation in the atmosphere and diffusion before the glass was completely cooled. Although the lack of iron enrichment in the glass does not prove conditions of vacuum during its formation (which would be required for a lunar origin), the presence of such enrichment is strong evidence of formation in an oxygen-rich environment (*9*).

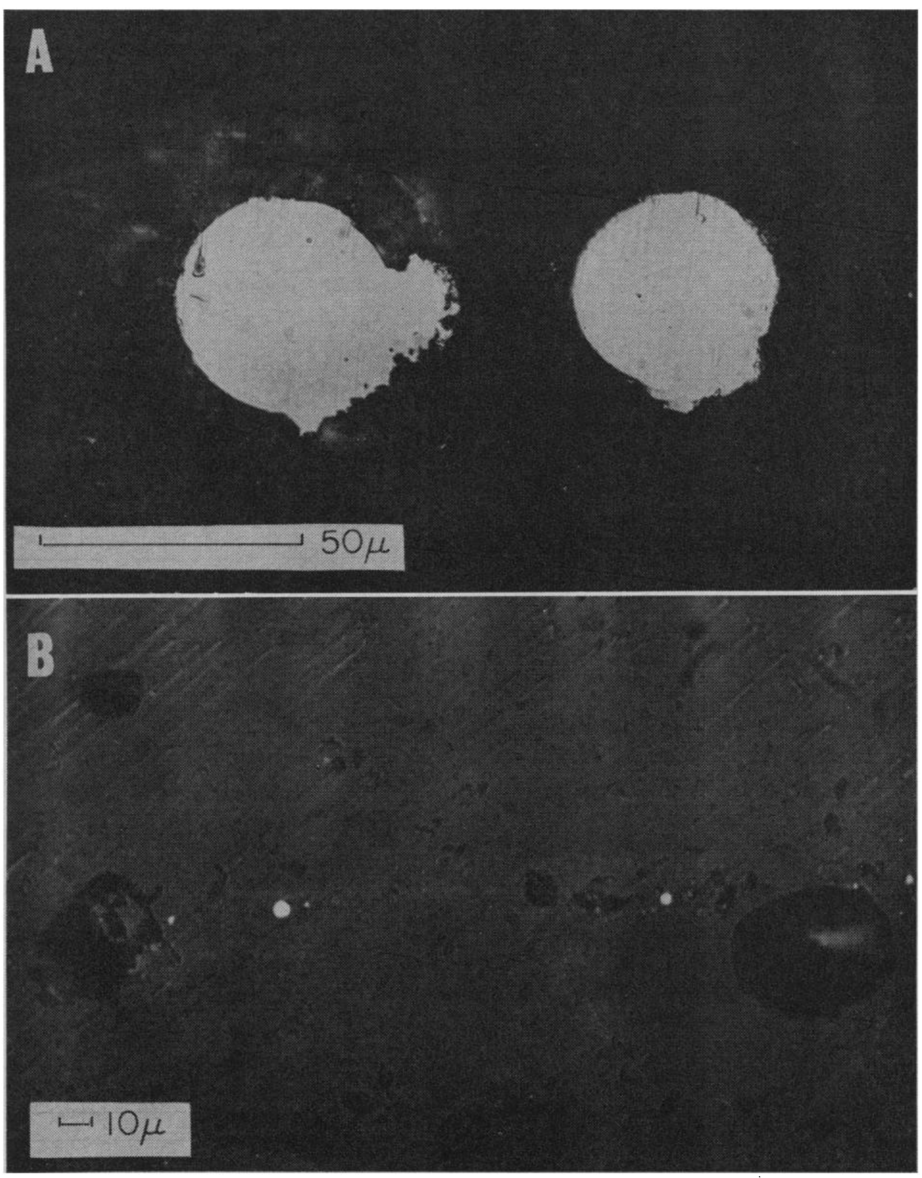

Fig. 1. (A) A polished wafer shows two partly spherical nickel-iron spherules; incident light. (B) Small nickel-iron spherules concentrated in a narrow band of glass; incident light, polished wafer.

Electron-microprobe analyses of the iron content along a traverse across the gray glass, clear glass, and brown matrix glass were made; the standards used were an analyzed moldavite with 1.35 percent Fe, an analyzed australite with 2.73 percent Fe, and an Ivory Coast tektite with 5.06 percent Fe. Because of the comparable amount of iron and similarity in matrix, no correction was made for such factors as absorption and fluorescence.

Table 2 shows clearly that various parts of the gray glass in which the spherules were embedded are distinctly richer in iron than is the brown matrix glass; in one spot, the gray glass contained twice as much iron. The iron content of the brown matrix glass, ranging from 1.8 to 2.0 percent, agrees well with the results of chemical analyses of four samples of Aouelloul glass done by x-ray fluorescence (*11*). The iron content of the sheath of clear glass around the gray glass is below the limit of detection.

Optical and x-ray studies were made to determine whether the enrichment of iron in the spherule-bearing gray glass is due to discrete, disseminated, nickel-iron spherules or to dissolved iron. Several bands of the spherule-bearing gray glass, with a sheath of colorless glass and attached brown matrix glass, were carefully removed and crushed to small fragments. Chips of gray glass bearing no visible nickel-iron spherules ($\times$ 150 magnification) and gray glass bearing visible spherules were mounted atop separate blank spindles and x-rayed for 24 hours with a microfocus x-ray diffraction unit. The latter specimen yielded a film having a weak but clear reflection at $d = 2.02$ Å for kamacite; the former showed no reflections.

Under the microscope, the boundaries between the gray glass, the colorless glass, and the brown matrix glass are very sharp. Two types of colorless glass have indexes of refraction of 1.465 ± 0.002 (almost pure silica) and 1.473

± 0.001; the brown matrix glass has an index of 1.479 ± 0.001. Some of the gray glass tends to splinter into elongated lenses and rods that show notable birefringence, thus indicating compression—a feature consistent with impact origin. Other gray glass fragments are completely isotropic and show no strain. Discrete particles of nickel-iron are disseminated irregularly in the gray glass, certain areas being essentially free of them. The gray glass has a much higher index of refraction (1.504 ± 0.001; Fig. 2B). This rise in index cannot be due to the discrete nickel-iron particles present; the rise must result from incorporation of dissolved oxidized iron from the meteorite source. This fact and the greater nickel content in one of the spherules (9 percent Ni)—relative to normal kamacite of iron meteorites—indicate that the fusion event took place in an oxidizing environment such as Earth's atmosphere.

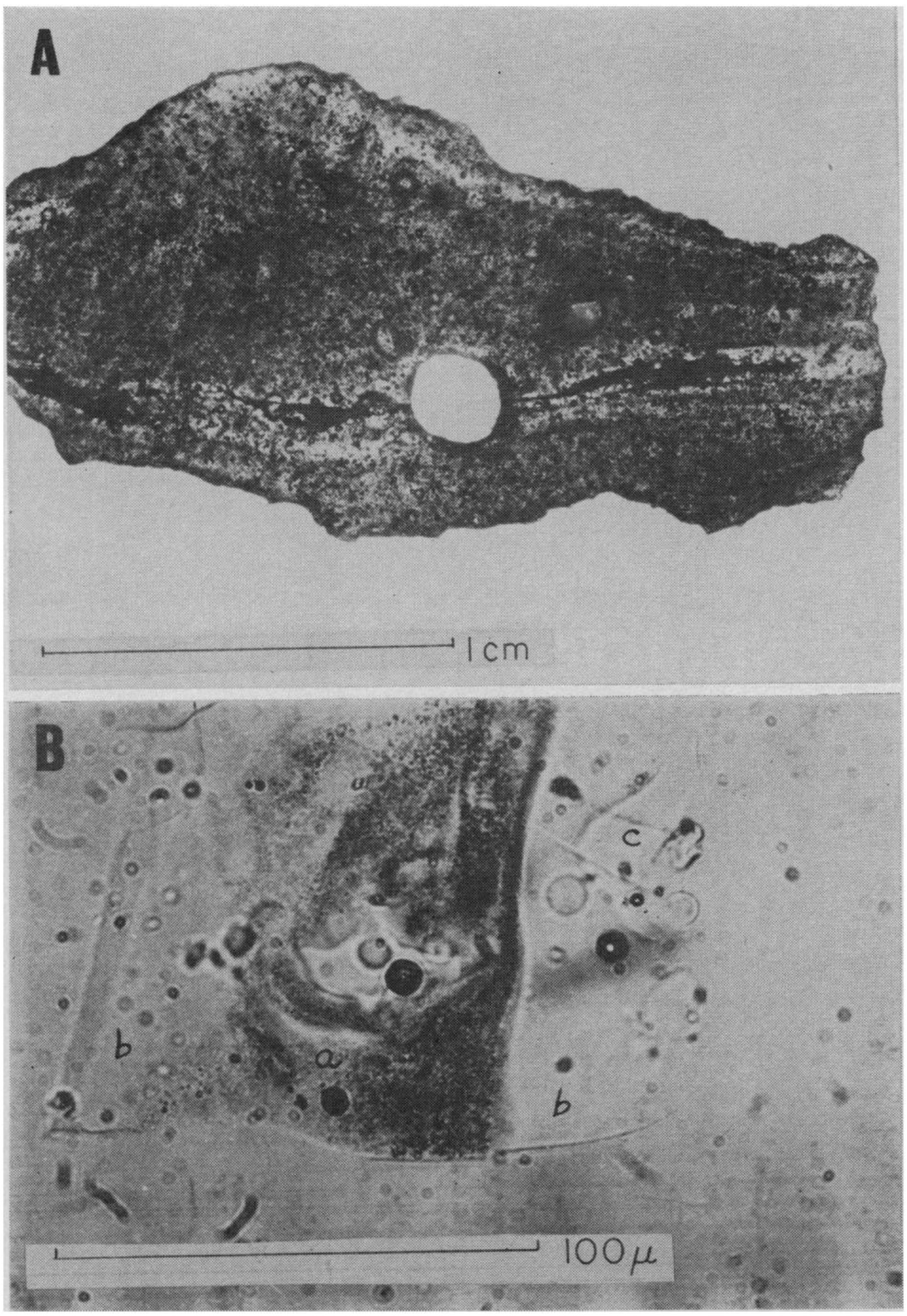

Fig. 2. (A) An Aouelloul glass wafer with a hole in it, showing the discrete occurrence of the nickel-iron spherules in three lenses (black areas) within a sheath of clear glass (white areas bordering the black) in the brown matrix glass (gray). Each dark lenticular area contains thousands of spherules. Transmitted light. (B) A fragment of spherule-bearing gray glass (a) (with an index of refraction, N, of 1.504), surrounded by clear glass (b) ($N = 1.473$) and clear glass (c) ($N = 1.465$). Black spots in the gray glass are nickel-iron spherules. Transmitted polarized light; grain immersed in oil ($N = 1.491$).

The brown Aouelloul glass contains many partially digested and undigested inclusions of sandstone; the glass must have been molten during the cratering event. As its gross chemical composition is nearly identical with that of the underlying Zli sandstone *(11)*, we believe that this glass derives from the sandstone and that it was fused by a meteoritic impact, so that much of the iron content in Aouelloul glass is of meteoritic origin. The gray glass is derived from addition of nickel-iron to a silica glass fused perhaps from either the Zli sandstone or the Oujeft quartzite *(11)*. These discrete lenses of silica-rich glass were engulfed by the more abundant brown glass during the violent event of the formation of the Aouelloul crater.

Aouelloul glass is similar in texture to some tektites; like them it contains numerous lechatelierite inclusions, but its composition is almost identical with that of the Zli sandstone underlying the crater. Its geographic distribution is restricted to the immediate vicinity of the crater. We conclude from the evidence presented that Aouelloul glass is an impactite of terrestrial origin and is not a tektite.

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