to cooling time. This suggests that the problem of excess radiogenic Ar^{40} may be less severe for coarse-grained rocks because they cool more slowly than fine-grained or glassy rocks.

Reliable potassium-argon ages probably will be obtained eventually from deep-sea samples, but criteria for recognition of reliable samples must first be developed. Meanwhile, ages from submarine volcanic rocks clearly cannot be used for testing of spreading of the sea floor without supporting evidence, such as determinations on two minerals from the same rock.

Much of the juvenile volatile content may still be present in volcanic rocks quenched on the ocean floor. This finding both supports earlier determinations that Hawaiian tholeiitic magma from depth contains H_2O at 0.45 percent by weight (*9*), and provides a new technique for study of volcanic gases—systematic analysis of the volatiles contained in the outer margins of fresh pillows collected from great depth.

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High-Resolution Electron Microscopy of Muscovite

Abstract. *Images of the (001) planes of muscovite were observed by electron microscopy after embedment of clay-size flakes in epoxy resin and sectioning by ultramicrotome. Dislocation or growth planes were indicated. Incomplete exchange of potassium for barium produced zones with 20-angstrom periodicity, suggesting regular interstratification.*

The interlamellar regions of phyllosilicate clays are the major sites of cation-exchange reactions occurring in the mineral fractions of soils and sediments. We have been concerned with determining the spatial arrangement of the individual molecular layers of weathered micas, and with relating this to cation selectivity (*1*). High-resolution electron microscopy can resolve lattice images of certain crystal planes. Since lattice images were first observed (*2*) in organic crystals containing heavy metal ions, they have been observed in other fine-particle materials with suitable crystal habit. Small crystals, lying horizontally on a microscopy substrate, must have the desired planes within 1 or 2 deg of the optical axis for successful imaging. Only recently have crystalline particles been oriented and microtomed for the purpose of seeing a particular set of lattice planes (see *3*).

In order to observe the lattice images of the (001) phyllosilicate planes associated with the interlamellar spacing, one must orient the flakes perpendicular to their normal position on a flat surface, and cut sections thin enough for the transmission of the electron beam. This was done as follows.

A layer of Araldite epoxy resin was cast on a glass microscope slide coated with a thin layer of silicone grease. After curing of the resin, a distilled-water suspension of mica particles was dried on the resin surface, and a second layer of resin was applied and cured. The cast "sandwich" was then removed from the slide and sawed into 1-mm cubes containing the sediment layer. The cubes were glued to larger blocks and mounted in the ultramicrotome. Thin sections approximately 300-μ square were cut at 90 deg to the sediment plane with a Leitz ultramicrotome using a diamond knife; they were estimated to be 300 to 600 Å in thickness as gauged by their reflected color (*4*).

Three samples all 0.2 to 2 μ in size were examined: (i) muscovite, (ii) barium-treated muscovite, and (iii) a weathered muscovite from a soil. Large flakes of a specimen muscovite from Amelia, Virginia, were broken apart in water with an Omni-Mixer, and the fraction between 0.2 and 2 μ was separated by centrifugation. An oriented sample of the fraction gave a sharp 10-Å x-ray diffraction peak as well as integral higher orders of this (001) reflection. Much of the potassium (76 percent) was exchanged from a portion of the fraction by repeated treatments with 0.1*N* $BaCl_2$ in an autoclave (*5*). The resultant sample gave a 12.1-Å (001) reflection. Higher orders are integral and indicative of a barium-dioctahedral vermiculite. In addition, interstratification of muscovite layers with vermiculite was confirmed by the asymmetry of the (001) peak.

X-ray diffraction also gave a 9.9-Å spacing for barium-saturated muscovite dried over P_2O_5. The resin-curing treatment or high vacuum in the electron microscope, or both, would remove the layer of water molecules associated with the 12.1-Å barium-vermiculite. The weathered muscovite was obtained by centrifugation of a sample of the B2 horizon of a Nason soil from Orange County, Virginia.

Electron micrographs (× 83,000) were taken on Eastman-P426 35-mm film with a Philips EM-200; they show parallel fringes that are lattice images of the (001) planes in the crystal and are formed by combination of the zero-order beam with a limited number of diffraction spectra from the periodic structure. The micrographs were made under bright-field conditions with a 50-μ back-focal-plane aperture; thus the image structure is formed from Bragg reflections of spacings greater than 4 Å.

The unweathered muscovite (Fig. 1A) shows fringes that are of uniform darkness when limited areas are considered. In contrast, in sections of the barium-treated sample (Fig. 1B) the dark fringes frequently alternate in intensity; this alternation may indicate zones where Ba has been substituted for K ions in alternate layers. Interstratification, indicated by x-ray diffraction, is apparently regular in these zones. Replace-

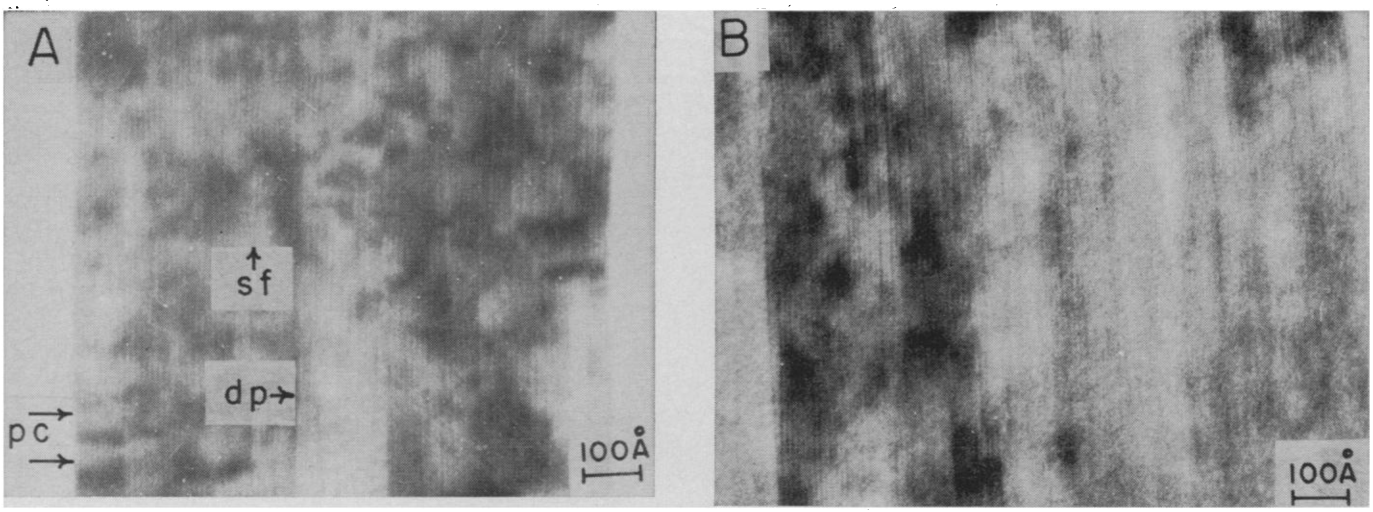

Fig. 1. Section of untreated (A) and barium-treated (B) muscovite cut normal to the (001) planes, showing 10-Å periodicity of fringes, possible dislocation or growth-step planes (*dp*), possible stacking fault (*sf*), and phase change (*pc*) at an extinction contour.

ment of two K^+ ions by one Ba^{2+} alters diffraction from such planes, and, when exchange occurs in alternate layers, 20-Å diffraction maxima are produced.

The dark spotty areas in the micrographs are extinction contours and represent slight and irregular bends in the crystal that diffract electrons outside the objective aperture and reduce their contribution to the final image.

The appearance of the lattice fringes may be modified by the interaction of diffracted beams within the crystal in accordance with the dynamical theory of electron diffraction. One important consequence is the periodic variation in intensity of diffracted beams with crystal thickness. Thus the projection of the atomic planes does not have 1 : 1 correspondence with a dark or light fringe in the image, but the phase can change at a proper thickness of specimen, as indicated in Fig. 1A (*pc*). Furthermore, effects of dynamical diffraction within the

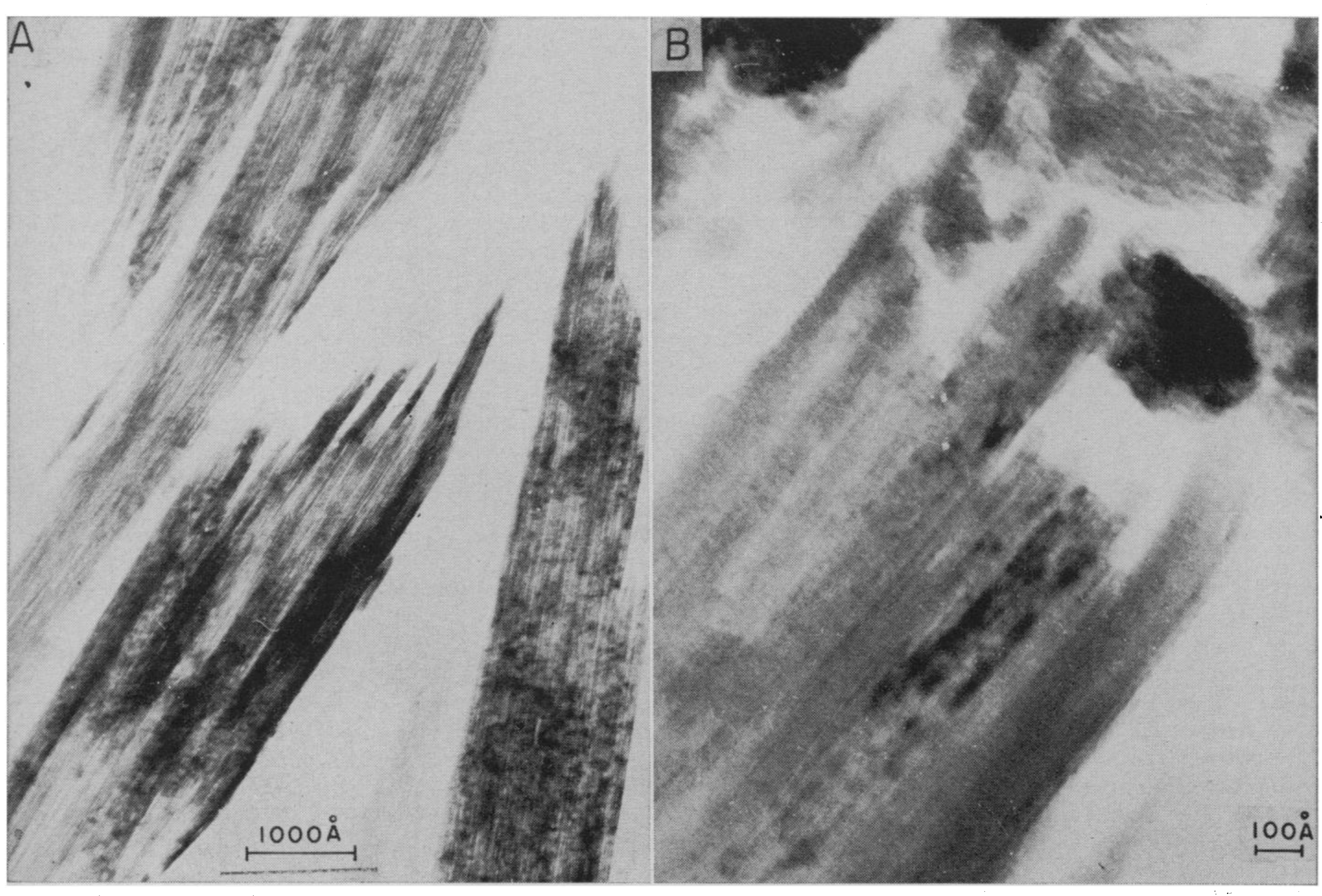

Fig. 2. (A) Frayed edges of barium-treated muscovite and (B) a naturally weathered clay-size muscovite from a soil. Sections were cut normal to (001) planes.

lattice will be modified by the presence of heavy atoms, since the phase shift on scattering, from a heavy atom, is different from that for a light atom (*6*).

Some variation in fringe intensity is seen for both the normal and the barium-treated samples; generally every fifth or tenth fringe appears darker (Fig. 1A, *dp*), and in some areas the extinction contours follow this variation, in a stepwise grouping; the effect is more obvious with the barium-treated sample. These larger groupings may represent screw dislocations (*7*) or growth steps. Dislocations due to stacking faults were not observed except as indicated in Fig. 1A (*sf*).

All the micrographs were selected from a through-focus series of six to eight exposures, each made with a focal change of 0.05 μ. When a pair of negatives separated by a focal change of 0.2 to 0.3 μ is examined in a stereoscopic viewer, one has a three-dimensional view of Bragg diffraction effects within the muscovite (*8*); the grouping of lattice planes previously mentioned is more apparent, and there appears to be some vertical shifting of groups. The extinction contours are not the effects of simple bending but are distributed through the depth of the section.

Lower magnification (Fig. 2A) shows the frayed edges of some of the particles. Fraying results not only from breakage of the larger crystals but also from dissolution of the crystal edges by the autoclave treatments. Individual layers, although separated at the ends, tend to cling together; this factor probably results from drying during the embedment procedure.

The edges of the naturally weathered clay-size muscovite from the Nason soil (Fig. 2B) are not nearly as frayed as flakes weathered in the autoclave. Groups of 5 to 20 10-Å layers protrude from the edge; these stacks appear to be related to planes extending into the particle.

Clay-size mica flakes apparently can be sectioned with little deformation of the lamellae by stress; an observation with crysotile fibers was similar (*3*). Our attempts to cut 20- to 5-μ particles failed, however. Embedment and exposure to high vacuum alters the layer spacings and edges of weathered mica flakes. Nevertheless, significant morphological features of individual molecular sheets can be distinguished by ultramicrotomy and electron microscopy.

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Paleo-Indian Remains from Laguna de Tagua Tagua, Central Chile

Abstract. *Bone and stone tools associated with extinct fauna (horse and mastodon) place man's occupation in central Chile at 11,380 ± 320 years ago.*

The area known as Laguna de Tagua Tagua, in the valley of the Rio Cachapoal (Province of O'Higgins), has been renowned for more than a century as a locale containing remains of Pleistocene fauna. In 1967, a group of Chilean scientists (*1*) organized a program for its systematic excavation. During the 1st week of work, a stone flake was encountered in association with the lower mandible of a mastodon, with the result that the primary focus of the investigation was transferred from paleontology to archeology.

Laguna de Tagua Tagua occupies a basin surrounded by the Cordillera de la Costa which rises to an elevation of 1000 m. The only natural outlet is the corridor of Cochipuy, a relatively narrow pass opening to the northeast. The area selected for excavation is an approach to the lake that is surrounded by low hills, making it a favorable region for hunting of animals as they came to drink. The region was seasonally if not permanently inundated, and probably was swampy (this will be clarified after completion of sediment analyses).

During the first 4 months of fieldwork, about 120 m^2 were excavated to a depth of 2.3 to 2.4 m. Artifacts were encountered in two levels. The most recent, at a depth of 1 m below the surface, corresponds to a group of hunters and gatherers or incipient agriculturalists. Characteristic artifacts are grinding implements and projectile points of types known from the adjacent coast. Fauna consists of modern species. This horizon is estimated to date about 3000 years ago.

At a depth of 2.40 m, chipped tools are associated with remains of extinct fauna. Bones of horse (*Hippidion*?) and mastodon (probably a new form), deer, and canid occur in a fragmentary and dispersed condition that reflects human activity; there is no evidence of natural disturbance to account for dismembering of skeletons. Much more abundant are bones of birds, frogs, fish, and rodents. Horse skeletal parts represent a single individual and include caudal vertebrae, ribs, and portions of the extremities. An astragalus and calcaneum show prominent cuts by a tool, probably produced during severing of a ligament. These cut marks continue across both bones. Many bones have been splintered by blows, and the absence of anatomical arrangement also implies butchering. The mastodon remains, also scattered and incomplete, include the basal portion of a skull lacking incisors, a mandible, two vertebrae, portions of femur, tibia and fibula, scapula, and both halves of the pelvis (which were found 1.5 m apart with the basal portion of the skull between them).

Fifty artifacts were encountered during the first 4 months of excavation. They not only belong to the same stratigraphic level as the fauna, but frequently are found as close as 2 to 5 cm from the bones. Most abundant are flakes, some of lamellar type. Some constitute chipping residue, whereas others are artifacts (Fig. 1, a–d). The artifacts include flake scrapers, one of which resembles a type described from the Domebo site in New Mexico. One flake is retouched at the distal ends, another on the proximal end and sides. Material is principally chalcedony and basalt. Obsidian flakes and an obsidian knife also occur. A prepared platform