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A simple fiberoptic sensor to detect the penetration of microsensors into sediments and other biogeochemical systems

Abstract—We have developed a simple and mechanically robust fiberoptic microsensor that enables optical detection of the sediment–water interface at a spatial resolution of <50 μm. The sensor measures with a tapered optical fiber the increased backscatter of near-infrared light near the sediment surface. To determine the sediment surface position independent of ambient light conditions, we developed a miniaturized opto-electronic system with an intensity-modulated laser diode (780 nm) as the light source and a photodiode as the detector. Laboratory tests of our system were done with artificial as well as with various natural sediments and biofilms. Fiberoptic microsensors for surface detection can be combined easily with both electrochemical and optical microsensors for oxygen or other reactive species.

Microsensors are frequently used tools for fine-scale measurements of chemical and physical variables in sediments and biofilms. Such microsensors have been developed to measure concentration profiles of oxygen, pH, sulfide, carbon dioxide, nutrients such as nitrate and ammonia, light intensity, and temperature (Revsbech and Jørgensen 1986; de Beer and Sweerts 1989; Cai and Reimers 1993; Kühl et al. 1994; Klimant et al. 1995, 1997; de Beer et al. 1997). An important prerequisite for obtaining useful information from microsensor measurements (e.g. calculations of diffusive boundary layer thickness, reaction rates and fluxes) is the ability to determine the exact position of the sensor tip relative to the interface between the biogeochemical system and the overlaying water (e.g. the sediment–water interface).

Penetration of the microsensor tip into a sediment or biofilm has previously been determined mainly by visual inspection of the sediment–water interface through a dissection microscope. Visual observation of the sensor tip at high magnification, however, is limited to laboratory experimental setups, whereas this is not generally possible for in situ work. Furthermore, visual determination of the penetration point can be imprecise or even impossible in systems with pronounced surface topography or with turbid overlaying water.

Most benthic landers with profiling microelectrodes are equipped with miniature resistivity probes that can also be used to detect the sediment–water interface (cf. Tengberg et al. 1995 and references therein). However, resistivity probes measure on a relatively large scale and are usually placed at some distance (cm) from the microsensor tips. It is therefore impossible to be certain that the surface position obtained by the resistivity probes is precisely relevant for each laterally displaced microsensor-measured profile.

For microsensor-determined O_2 profiles it is sometimes possible to infer the surface position from a shift in the slope of these profiles at the sediment–water interface (Reimers et al. 1986; Sweerts et al. 1989; Gundersen and Jørgensen

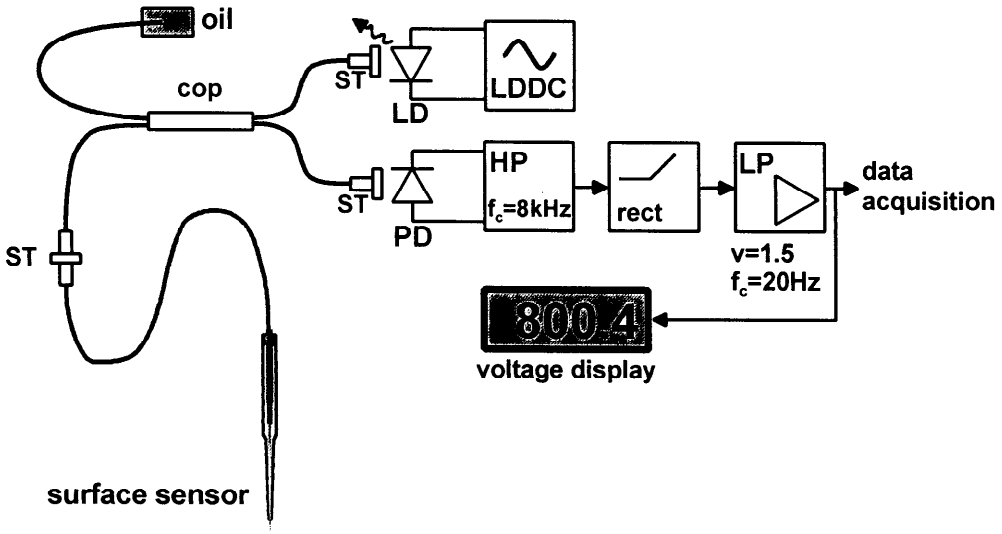

Fig. 1. Schematic drawing of the opto-electronic measuring system: ST, standard optical fiber connectors and receptacles; cop, 2×2 fiberoptic coupler; oil, immersion oil covering one branch of the fiber-coupler; LD, laser diode; PD, photodiode; LDDC, laser diode driving circuit and modulation signal generation; HP, electronic high-pass filter; f_c, cutoff frequency; rect, precision rectifier; LP, electronic low-pass filter; v, amplification.

1990; Epping and Helder 1997). This shift results from the different diffusive properties of the diffusive boundary layer (DBL) above the sediment (i.e. molecular diffusion in water) and the sediment itself, where diffusion is reduced by porosity and tortuosity. It is our experience, however, that a clear difference in slope is not present in every O_2 profile. Furthermore, in permeable sediments with significant advective transport, the DBL is often not detectable (Forster et al. 1996), and the slope method cannot be used. A simple, accurate, and versatile method to detect the sediment–water interface is still lacking.

Sediments and biofilms are dense light-scattering media with optical properties that differ significantly from those of the overlaying water (Kühl and Jørgensen 1994). The sediment–water interface is characterized by dramatic changes in both transmittance and reflectance of incident light, as measured with fiberoptic microprobes (Kühl and Jørgensen 1994; Kühl et al. 1994). A fiberoptic surface-detection microsensor based on the attenuation of external illumination in a biofilm was reported by Lewandowski et al. (1991). This sensing scheme, however, is not applicable to dark-incubated sediments or for in situ measurements at the sea floor, as the fiber tip must be directed toward the external light source. Furthermore, any fluctuation in the external illumination or the optical properties of the biofilm would affect the measurement and lead to uncertainty in the surface position. This paper presents a novel microsensor for precise determination of the sediment–water interface. The sensor detects the increasing quantities of near-infrared (NIR) light that can be measured by an optical fiber on approaching an interface, and the system is sufficiently small and robust to be easily mounted in conjunction with other microsensors. Reflection-based optical methods for surface detection and positioning have found many industrial applications, but to our knowledge no application of these methods to microscale and biogeochemical systems has been reported.

A single-strand optical fiber cable (140/100-μm graded-index glass fiber, Corning), terminated at one end with a standard (ST) fiber connector, was used to fabricate the microsensors. The protective polymer coating was stripped off one end of the fiber cable, and this bare fiber was tapered to a tip diameter of ~20 μm in a small flame (Kühl and Jørgensen 1992; Klimant et al. 1995). The tapered light-collecting end of the fiber was then mounted in a thin glass capillary or glued to an oxygen microsensor with fast-curing epoxy resin. Tapered optical fibers are mechanically robust and do not break easily.

The sensor fiber was attached to the optical system via an ST connector. The optical system is a simplified version of a previously described setup for optical oxygen microsensors (Fig. 1) (Klimant et al. 1995). A 780-nm NIR laser diode (ADVA) mounted in an ST-connector receptacle was used as a small cost-effective light source with low power consumption and heat production; this allows efficient and easy coupling of light into optical fibers. An NIR-emitting laser diode (LD) was chosen to avoid stimulation of photosynthesis in phototrophic communities. Furthermore, earlier studies have shown that 700–800-nm NIR light is not absorbed significantly in the upper layers of most sediments and biofilms and that the relative amount of backscattering is maximal in this spectral range (Kühl and Jørgensen 1994; Kühl et al. 1994). The efficient coupling of laser light into the optical fiber and the high backscattering of NIR light from the sediment made it possible to use a simple silicon PIN photodiode (iC-Haus) for detection of the backscattered light, with a signal-to-noise ratio of >1,000 : 1.

To guide both illumination and backscattered light via the same sensor fiber, a 2×2 fiber-coupler (E-Tek) was inserted

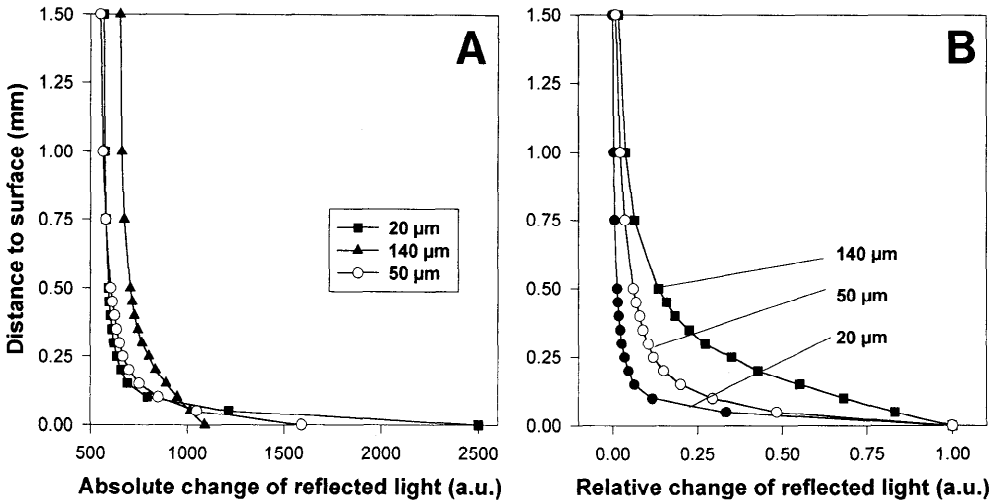

Fig. 2. Profiles of backscattered NIR light (780 nm) measured with tapered fibers of different outer-tip diameters over a flat-scattering surface of immobilized titanium dioxide. A. Absolute signal intensity (arbitratry units [a.u.]) B. Relative change in signal. Numbers on curves denote fiber-tip diameter in micrometers.

between the light source and the photodiode detector (Fig. 1). This coupler is a fiberoptic analogue to a beamsplitter and has the advantages of high directivity, simple adjustment, and robust mechanical properties. The LD and the photodiode were connected to one side of the coupler. Half of the light from the LD was guided into each of the fibers on the other side of the coupler. Only one of these fibers was attached to the sensor fiber via an ST connector. The tapered tip of the sensor fiber emits NIR light. In the absence of a scattering surface (i.e. far from the sediment), almost no light will be scattered back into the fiber from the surroundings; thus, a constant background signal will be measured, which is caused by backscattered light in the optical system itself. To minimize this background signal, it was necessary to use immersion oil in the fiber connectors. Immersion oil has a refractive index similar to the refractive index of the glass fibers, thereby minimizing back-reflection from the fiber ends.

The operating principle for our surface-detection sensor is simple. When the light-emitting fiber tip is moved toward the water–sediment interface, an increasing amount of diffuse backscattered light from the sediment surface is directed back into the fiber. The highest relative signal change occurs when the fiber tip reaches the sediment–water interface, which results from the significantly different scattering properties of sediment and water.

The opto-electronic system is based on an intensity modulation scheme in which the central electronic elements were adapted from a laser-beam barrier system (Fig. 1; iC-Haus). The LD intensity is directly modulated (rectangular signal form), and light power is controlled at a frequency of f_{mod} = 10 kHz by a special integrated circuit. The backscattered light is detected by a PIN photodiode with an integrated bandpass transimpedance amplifier that is adapted to the light modulation frequency and signal form. The DC offset of the photodiode is cut off by a high-pass filter (f_c = 8 kHz). The rectangular measuring signal is then rectified, sent through a low-pass filter (f_c = 20 Hz), and amplified. This DC voltage signal is displayed on a voltmeter display (Datel) and is available via an instrument amplifier to any analogue data-sampling instrument (e.g. strip-chart recorder or computer A/D acquisition). By using this amplitude-modulated opto-electronic system, it is possible to make surface detection measurements that are independent of the ambient light level (i.e. in full daylight). The complete measuring system, including the optical setup, can be mounted in a small compact unit for field applications.

The performance of the surface-detection system was tested in an artificial system with high scattering, but a well-defined surface (i.e. a planar plastic foil with dispersed titanium dioxide crystals, grain size 1–5 µm), all of which was glued to the bottom of a water-filled Petri dish. Measurements were done with tapered fibers with tip diameters ranging from 20 to 140 µm. The microsensors were mounted vertically in a manually operated micromanipulator (Märtzhäuser). The fiber tip and its position relative to the surface of the foil were observed through a dissection microscope; consequently, the surface position could be determined easily. The measured signal exhibited a hyperbolic increase as the fiber tip was moved toward the surface (Fig. 2A). Depending on the size of the sensor, the amount of measured backscattered light was relatively constant until the fiber tip was within 100–400 µm of the scattering surface. Closer to the surface, an increasing amount of backscattered light was detected, and the highest relative signal change was found when the tapered fiber touched the surface (Fig. 2B). The spatial resolution of the surface determination depended on the dimensions of the fiber tip, and the precision of surface detection increased with decreasing sensor size. Miniaturization of the sensor tip not only improved the spatial resolution of the measurement, but it also resulted in a strong increase in the total amount of light reflected back and detected by the optical fiber. This latter effect was caused by the higher effective numerical aperture of a tapered fiber as

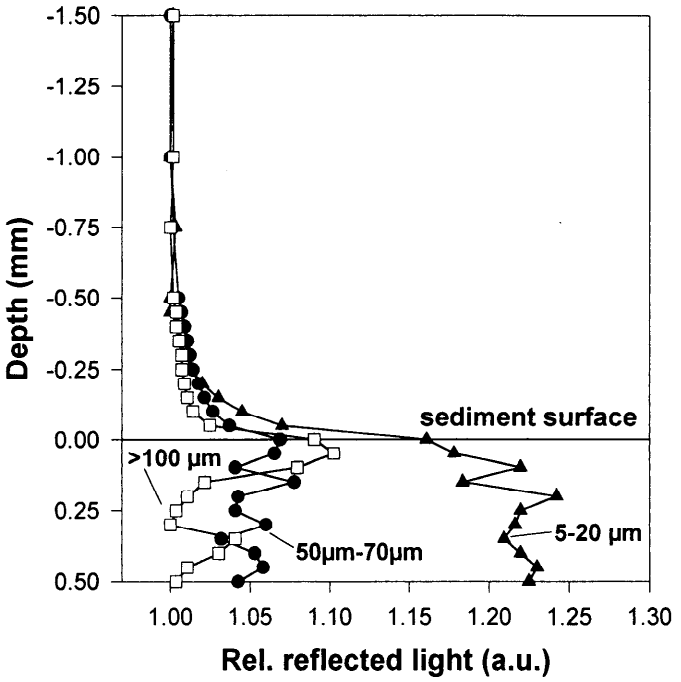

Fig. 3. Depth profiles of backscattered NIR light (780 nm) measured in sandy sediments with different grain sizes (indicated as numbers on curves). The outer-tip diameter of the tapered fiber was ~20 µm.

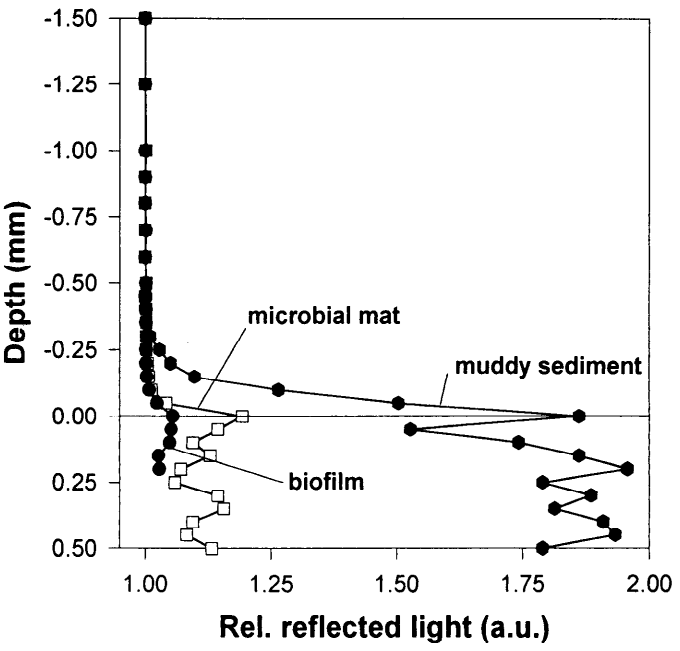

Fig. 4. Depth profiles of backscattered NIR light (780 nm) measured in various sediments and biofilms. The outer-tip diameter of the tapered fiber was ~20 µm.

compared to an untapered fiber. Best results were obtained by tapering optical fibers to 20–30-µm tip diameter (i.e. the same dimensions as previously described oxygen micro-optrodes; Klimant et al. 1995). A further miniaturization of the sensor tip to <10 µm is possible, but results in significantly lower absolute signal changes due to a strong loss of evanescent light at the tapered sides of such fibers. The shape of the fiber taper might also affect the light-emitting and collecting properties of the tip (Gao et al. 1995), but we have so far not done a systematic investigation of such effects.

The surface-detection system was also used in artificial sandy sediments with different grain size distributions (Fig. 3). When compared to the measurements in the TiO_2 layers, the measurements in sand exhibited an increase in signal at larger distances from the sediment surface. This difference can be explained by the fact that the coherent light from the LD was in part diffusely scattered back and in part directly reflected from sand grains at the sediment surface (mirror effects). Such mixed optical conditions complicate a theoretical description of the response curve for a surface-detection measurement because the shape of the response curve depends both on the fiber tip geometry and the sediment material itself (i.e. refractive index, size and shape of the sediment particles).

Best results were obtained in fine or silty sediments, whereas the use of the surface-detection microsensor in sandy sediments with larger grains (>100–200 µm) was problematic (Fig. 3). In such coarse sediments, it is only possible to detect the first collision of the fiber tip with a sand grain, and if the microsensor penetrates a pore in the sand, no significant increase in the signal is measured. Moreover, it is inherently impossible to define a surface position with a precision better than the average grain-size diameter of the sediment in such heterogeneous systems. Once the fiber tip penetrates into the sand, the signal intensity exhibites significant fluctuations, possibly due to slight variations in the density of scattering sediment particles around the fiber tip.

We have tested the surface-detection system in various natural systems with different scattering-to-absorption ratios (Fig. 4). Biofilm aggregates and cyanobacterial mats exhibited less relative signal changes in backscattered light at the biofilm–water interface than at various sediments. The signal change, however, was large enough to identify the surface position with <50-µm spatial resolution. Because the measurement is done via a thin optical fiber, our measurements are of course point measurements that will (like any other microsensor measurement) be affected by the heterogeneity of the interface (e.g. the surface topography of sediments and biofilms).

The detection principle relies on the change in NIR light-scattering properties between the water and the sediment or biofilm matrix. Even in the case of optically dense and almost black biofilms (such as the aggregate or the photosynthetic mat investigated in this study), there is a detectable change in scattering properties at the interface, although the absolute signal levels are smaller than over a white reflecting surface. The general shape of numerous measured curves was the same. The variability of the absolute signal depended on the heterogeneity of the system investigated, but this is not important for the surface determination as such. As long as there is a refractive index difference between the water and the matrix, which results in changes in NIR reflection and backscatter at the interface, the presented sur-

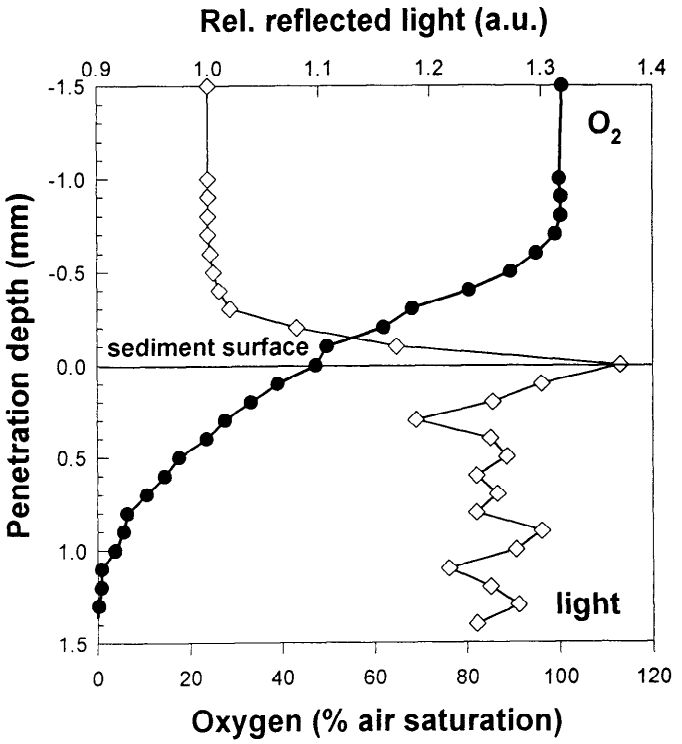

Fig. 5. Parallel measurements of oxygen and backscattered NIR light (780 nm) in a silty North Sea sediment. The combined sensor consisted of an oxygen micro-optrode glued together with a tapered optical fiber for measuring backscattered light. The surface-detection fiber was positioned ~50 μm horizontally displaced from the O_2 micro-optrode tip.

face detection system will work. In very loose and gelatinous biofilms without significant pigmentation and no significant difference in refractive index between the biofilm and surrounding water, the present measuring system might have its limitations, and further investigations and optimizations will be necessary.

The surface-detection system can be combined easily with chemical microsensors. We have made a combined surface-detection–oxygen microsensor by gluing the tapered fiber to an oxygen micro-optrode (Klimant et al. 1995) or to an oxygen micro-electrode. The distance between the two sensing tips of the combined sensor was ~50 μm. The oxygen micro-optrode was constructed and used as previously described (Klimant et al. 1995); however, the oxygen-dependent fluorescence lifetime was monitored instead of the fluorescence intensity (Holst et al. 1995, 1997). We used the combined sensor in a dark-incubated coastal North Sea sediment (Fig. 5). The results show a relatively good correspondence of the optically determined surface position and the surface position one would infer from the oxygen measurements. A possible increased compression effect of such a combined oxygen and surface-detection sensor on the DBL (Glud et al. 1994) cannot be ruled out, however, and a quantification of such an effect needs further detailed investigations.

It should also be possible to map the subsurface heterogeneity of sediments with the present system with combined position detection and measurements of chemical microprofiles. For example, it should be possible to determine if the sensor tip penetrates a worm burrow or a gas bubble during profile measurements. Also, in studies of the rhizosphere around plant roots, such combined position and chemical determinations might be useful in relating chemical gradients to the position of the root surface.

Probably one of the most important applications of the surface-detection system, in combination with chemical microsensors, is for in situ measurements (e.g. deep-sea measurements with benthic profiling landers). Owing to the small size of the optical system, it is relatively easy to adapt the surface-detection system to benthic landers, provided a good waterproof and hydrostatic pressure-stable penetrator for fiberoptics is used. We are currently investigating the possibility for in situ measurements of surface detection and oxygen concentration with combined fiberoptic microsensors mounted on a benthic-profiling lander.

In conclusion, the present detection system has proven useful for detection of the sediment–water interface in various natural systems. Parallel surface detection and measurements of chemical variables can now be realized at the same microscale, allowing for a more precise calculation of DBL thickness and reaction rates from microprofiles. Fast positioning of microsensors becomes possible without the need of a dissection microscope or other means of visual inspection.

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Comparison of the phytoplankton species composition and structure in the Climax area (1973–1985) with that of station ALOHA (1994)

Abstract—In August 1994, water samples were collected from seven depths on each of two casts at the Hawaiian Ocean Time-series station (ALOHA; 22°45′N, 158°W). These samples allowed a comparison between the larger phytoplankton taxa at ALOHA and those in the Climax area (near 28°N, 155°W) that were collected during summers between 1973 and 1985. Of the 142 species found at ALOHA, all but 6 have been seen in the Climax area. The two-layered structure that is typical of the Climax area was also found at station ALOHA, where the break between shallow and deep associations occurred between 100- and 135-m depth. However, abundances of the deep species at ALOHA were lower than in the Climax area. The correlations between the rank order of abundances of phytoplankton from Sta. ALOHA and from the Climax area fell within the spectrum of correlations between pairs of stations from the Climax area. These results indicate that in August 1994 the phytoplankton at Sta. ALOHA was indistinguishable from that in the Climax area between 1973 and 1985. Nevertheless, many additional studies are needed before results from the Climax area or results from the Hawaiian Ocean Time-series program can be unconditionally generalized.

In 1988 the Joint Global Ocean Flux Study established the Hawaiian Ocean Time-series program (HOT) to characterize processes of biogeochemical cycling at a site representative of the oligotrophic central North Pacific Ocean (Karl and Lukas 1996). The HOT program is located at Sta. ALOHA (22°45′N, 158°W), 100 km north of the island of Oahu. Observations from ALOHA consist of a series of approximately monthly cruises, each with 72–96 h on station. Because these studies have limited spatial coverage, extrapolation of data from the HOT program will depend on quantifying the spatial generality of the data. Recent analyses of remotely sensed data suggest that this generality may be limited (McGowan 1995; Karl 1995; Esaias pers. comm.). These studies, however, are restricted to a few near-surface parameters and cannot address the fundamental ecological property of species composition and its spatial variability. The ideal study regarding the latter must involve extensive commitment of resources and may not occur in the immediate future. Thus, complete comparison with existing data is important.

One available dataset is that collected between 1964 and 1985 from the vicinity of 28°N, 155°W (the Climax area; Venrick 1982, 1990), ~650 km northeast of ALOHA. However, several conceptual and methodological improvements have occurred during the intervening years, and these, together with the different objectives of the two programs, have resulted in different measurements. Thus, few of the