CONF-970201--10

# Monolithic Micro-Spectrometer For Low-Cost Sensing In Materials Processing

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Abstract

We have developed a monolithic micro-spectrometer suitable for a variety of sensing applications including industrial process monitoring. The device consists of a solid structure with a volume less than 6 cubic centimeters. All optical components of the spectrometer including two aspheric mirrors, a diffraction grating, and entrance and exit surfaces are fabricated onto the surface of the structure. All light paths are internally contained within the structure. The result is a small, rugged spectroscopic sensor ideally suited for use as a process monitor. Due to its monolithic nature the device requires no post-fabrication alignment; nor can it be knocked out of alignment during use. Although many materials and thus wavelength regions were possible, the prototype device discussed here was produced in PMMA by precision diamond turning. However, lower cost manufacturing approaches involving injection molding are under development to produce an affordable sensor.

Due to its rugged monolithic design, small footprint, and low cost the monolithic micro-spectrometer is ideally suited for distributed monitoring applications often required for industrial processes. It is expected that modified versions of this sensor designs will be required for specific applications so as to maximize the performance and resolution over the operating range. For example, the prototype device described here operates over a wavelength range of 500 nm to 1000 nm. The performance of this device as a real-time surface monitoring sensor will be discussed in detail. Our approach of using wavelength dependant scattering resulted in the reduction to practice of a miniature, monolithic, rugged, low-cost, micro-instrument in a sensor sized package. The process sensor applications community has been faced by the cost versus performance issue. Simple go/no-go sensors are typically inexpensive but they provide very limited and sometimes dubious data. Scaled down traditional instruments offer very good measurand information but are typically cost prohibitive, fragile, and still relatively large. This work will bridge the gap between these two extreme sensing alternatives.

surface. In this design the NA of this 62.5 μm core diameter fiber was 0.275 (~$16^0$ half angle). This core diameter represents the spectrometer entrance slit width and is not variable as in most laboratory sized instruments. The collimating surface redirects the light energy toward the grating with a focus at infinity. The light impinging on the grating arrives at a specific angle and with a collimated diameter of approximately 6mm. The grating in this design is a 300 lines / mm flat surface operating in the -1 order. The grating surface disperses the incident light toward the focusing surface. The light at this point is diverging from the approximately 6mm diameter beam that impinged onto the grating. Thus the useful area of the focusing surface is the largest of the five precision surfaces at approximately a diameter of 10 mm. The focusing surface intercepts the diverging cone of light and focuses it onto the image surface. A linear detector array is then attached directly to this image surface.

## Experimental Methods

Test samples simulating various surface conditions were prepared for this study. The 6061 aluminum test coupons, measuring 1.5 cm in thickness and having a diameter of 5 cm, were first diamond turned to obtain a flat optically reflective surface. These samples were subsequently treated by one of several methods to approximately simulate potential material removal / surface modification conditions. First, aluminum oxide was deposited onto one sample to simulate the growth of an oxide film on the substrate. Three different thicknesses of aluminum oxide were applied to different sections of the sample; one quarter of the sample was not coated and had only the native oxide layer. The oxide was deposited under vacuum by an ion beam sputtering process. Thicknesses of the oxide layers, measured by a stylus profilometer after deposition, were approximately 0.1, 0.5 and 1.0 μm. A second sample was also divided into quarters and each of three regions were mechanically roughened to simulate surface modification which might occur at various stages of a grinding and polishing operation. Finally, a third sample was again divided into quarters and each quadrant was chemically etched by an aqueous sodium hydroxide solution thereby modifying the surface in a manner different from the previous techniques, yet introducing elements of both as might occur in chemo-mechanical material removal processes. The extent of simulated surface modification was determined by the length of time the surfaces were exposed to the solution. Exposure times for the three regions were: 60, 120, and 180 seconds. Reflectance as a function of wavelength over the range 0.5 to 1.0 μm was measured on these surfaces. A white light source (miniature tungsten lamp) was used to illuminate the sample through a fiber optic and a collimating lens at an angle of approximately 45° with respect to normal. A second fiber optic with a focusing lens (also oriented 45° with respect to normal) was used to collect the reflected signal which was then input into the spectrometer. An area of approximately 0.27 $cm^2$ of the sample surface was illuminated. Reflectance scans were then generated by ratioing the reflected signal from the treated surfaces to a reference signal obtained from the untreated surface [2].

## Results and Discussion

Reflectance plots as a function of wavelength are shown in Figs.3-5, for the oxide coated aluminum samples. These plots show a strong effect on reflectance in the wavelength range from

0.5 to 1.0 μm produced by an oxide layer as thin as 0.1 μm. Thicker oxide layers result in an oscillatory reflectance signal caused by optical interference within the oxide layer. These results illustrate the sensitivity of spectroscopic techniques in detecting the growth of even minute oxide films on metal surfaces. From these experiments it is estimated that oxide growths as thin as 0.02 μm could readily be detected spectroscopically. It is also important to observe that the growth of oxide on the surface does not monotonically decrease the reflectance at shorter wavelengths due to the optical interference effect.

Reflectance results for samples which were mechanically roughened are shown in Figs. 6-8 and correspond to profilometer measured roughness values of 268, 398, and 531 nanometers rms, respectively. Visually, these surfaces exhibited various streaks and scratches of random lengths and orientations. Here a strong, monotonic decrease in reflectance is observed with decreasing wavelength, even for the most lightly abraded surface. The moderately and heavily roughened regions showed a more pronounced reduction in reflectance eventually extending throughout the entire wavelength region investigated. This behavior is produced by scatter of the light from the roughened surface. Scattering is especially strong for shorter wavelengths resulting in the observed decrease in reflectance at shorter wavelengths [3,4].

It is interesting to observe here the difference in the wavelength dependence of reflectance from the scratched surface compared to a surface with oxide growth. The scratched surface shows a strong monotonic decrease in reflectance with decreasing wavelength while the oxidized surface exhibits an oscillatory behavior without decreased reflectance at shorter wavelengths. This difference suggests the possibility of using optical spectroscopy to distinguish between different types of corrosion which produce these changes in the surface. To explore this possibility further a third set of experiments were performed on aluminum surfaces which had been chemically etched. The chemical attack produced a layer of corrosion product while also preferentially etching different grains exposed at the surface. The severity of the chemical attack depended on the length of time each surface was etched. The result was a series of chemically modified surfaces whose reflectance plots are shown in Figs. 9-11 and correspond to profilometer measured roughness values of 16, 49, and 361 nanometers rms, respectively. These plots show roughening of the surface (Fig. 9) and the eventual growth of a layer of corrosion product indicated by the oscillatory behavior apparent in Figs. 10 and 11. These preliminary results therefore suggest that it may be possible to use spectroscopic techniques to distinguish and control some types of chemical and mechanical processing on metal surfaces.

## Conclusions

These results indicate that optical spectroscopy can be used as an extremely sensitive probe to monitor the condition of aluminum surfaces. The growth of an oxide layer was readily detected as an oscillatory signal while roughening of the surface produced by scratching the surface exhibited a monotonic decrease in reflectance with decreasing wavelength. Since many materials such as uranium can show similar effects even during the initial material processing phase of manufacturing, this technology could prove useful in monitoring and controlling material processing in real time. Furthermore, it may also be possible to use spectroscopy to distinguish

between different types of chemical attack and determine the extent of attack. Many mechanical material removal processes would also benefit from such a monitoring capability. All of this would be extremely useful information in real-time monitoring of many industrial processes. The development of an affordable, monolithic micro-spectrometer makes this detection and monitoring possible without relying on traditional bulky, expensive, and fragile laboratory type equipment to perform these measurements in an industrial setting.

## Acknowledgments

This work was sponsored by the laboratory directors' research and development (LDRD) fund within the Oak Ridge National Laboratory (ORNL). ORNL is managed by Lockheed Martin Energy Research for the U.S. Department of Energy under contract no. DE-AC05-84OR21400. Support was also provided by the Oak Ridge Centers for Manufacturing Technology (ORCMT).

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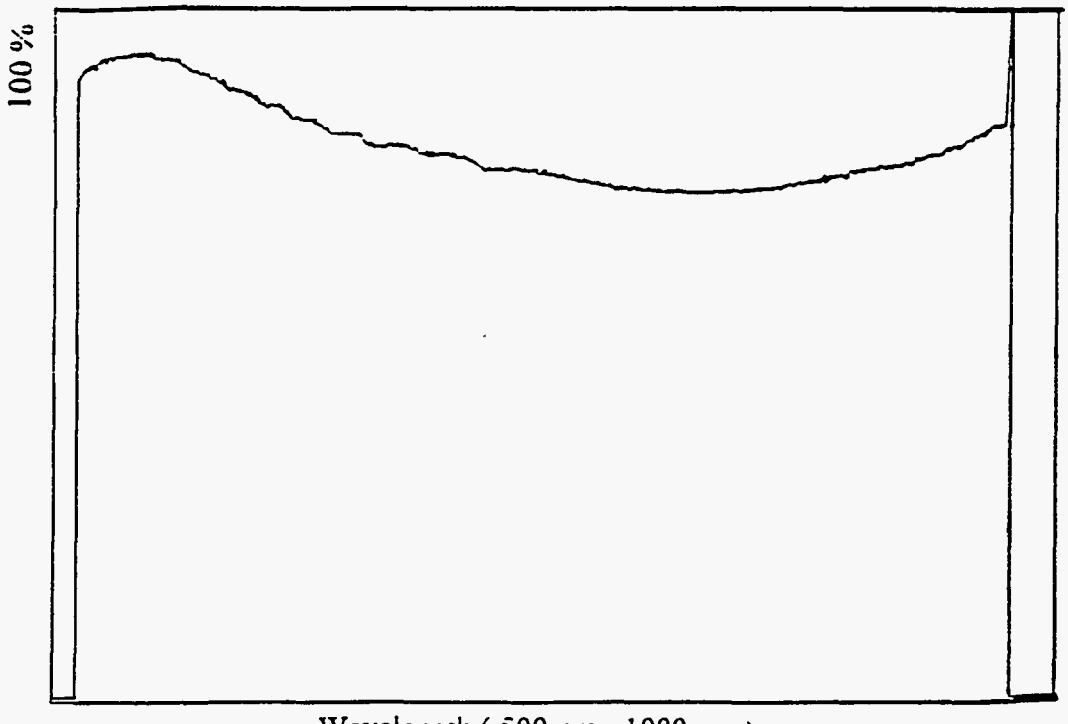


Fig. 3 Reflectance plot of ~0.1 μm thick oxide layer on aluminum.

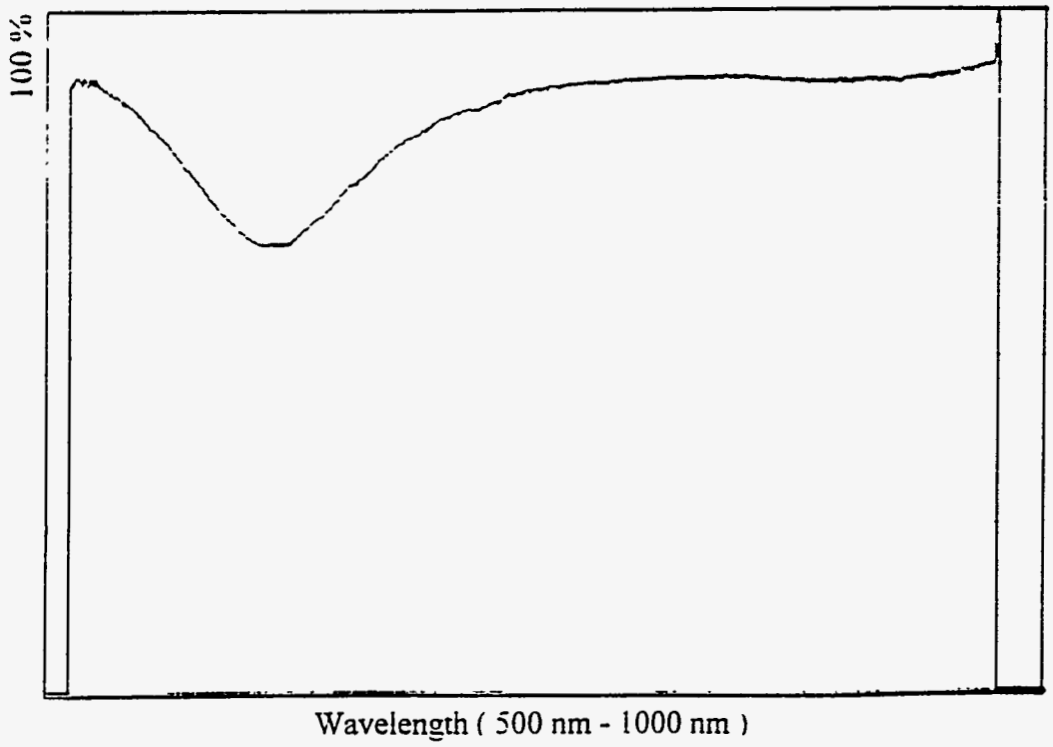


Fig. 4 Reflectance plot of ~0.5 μm thick oxide layer on aluminum.

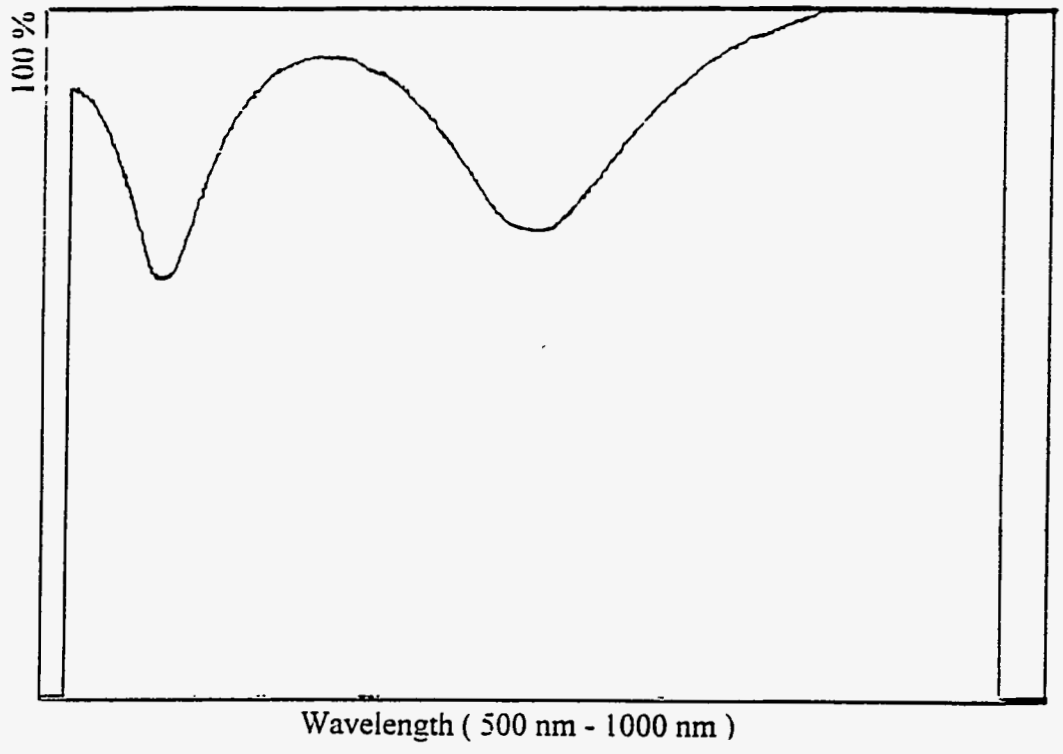


Fig. 5 Reflectance plot of ~1 μm thick oxide layer on aluminum.

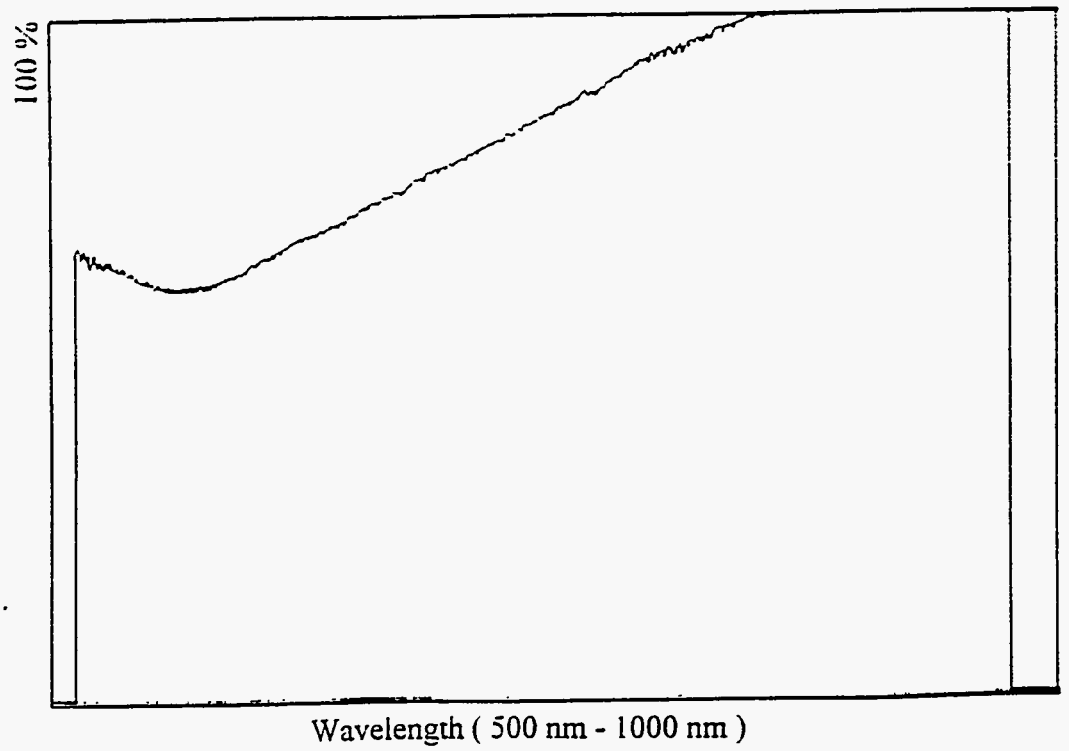


Fig. 6 Reflectance plot of lightly scratched aluminum surface.

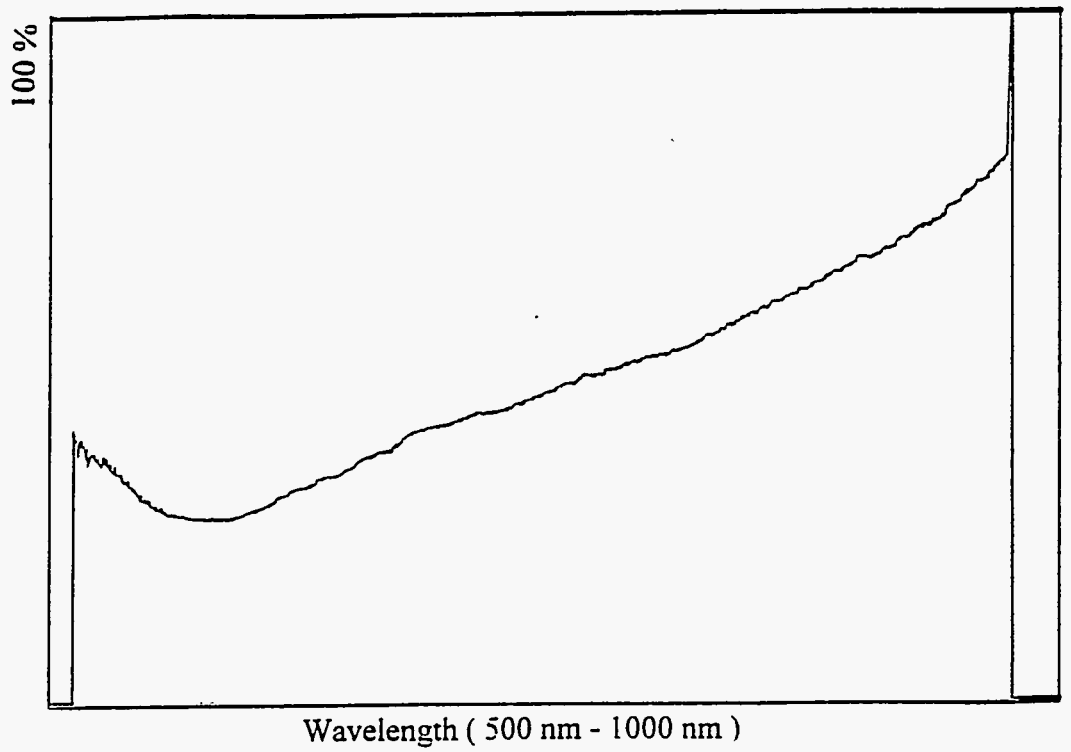


Fig. 7 Reflectance plot of moderately scratched aluminum surface.

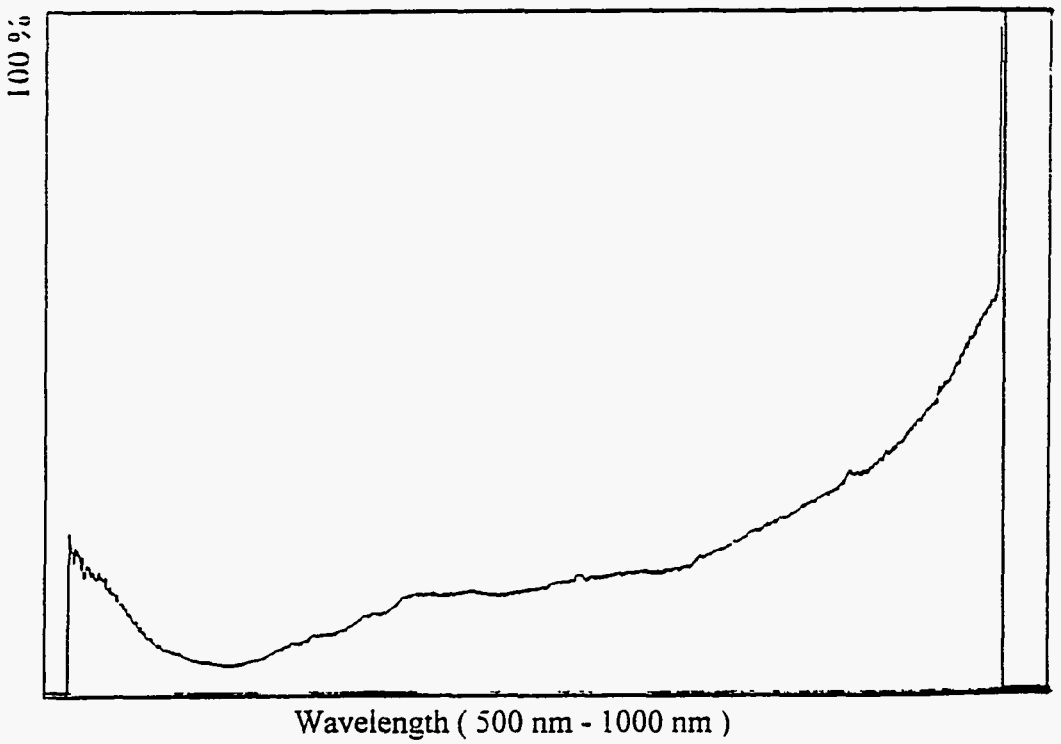


Fig. 8 Reflectance plot of heavily scratched aluminum surface.

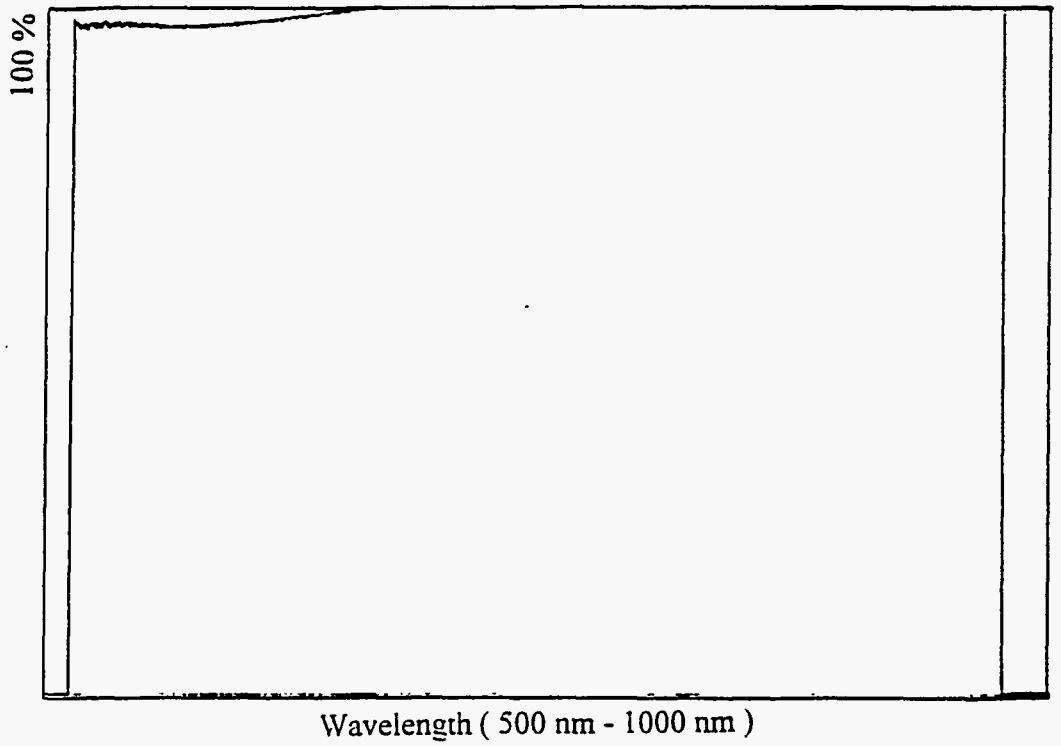


Fig. 9 Reflectance plot of a chemically etched aluminum surface (etch time = 60 sec ).

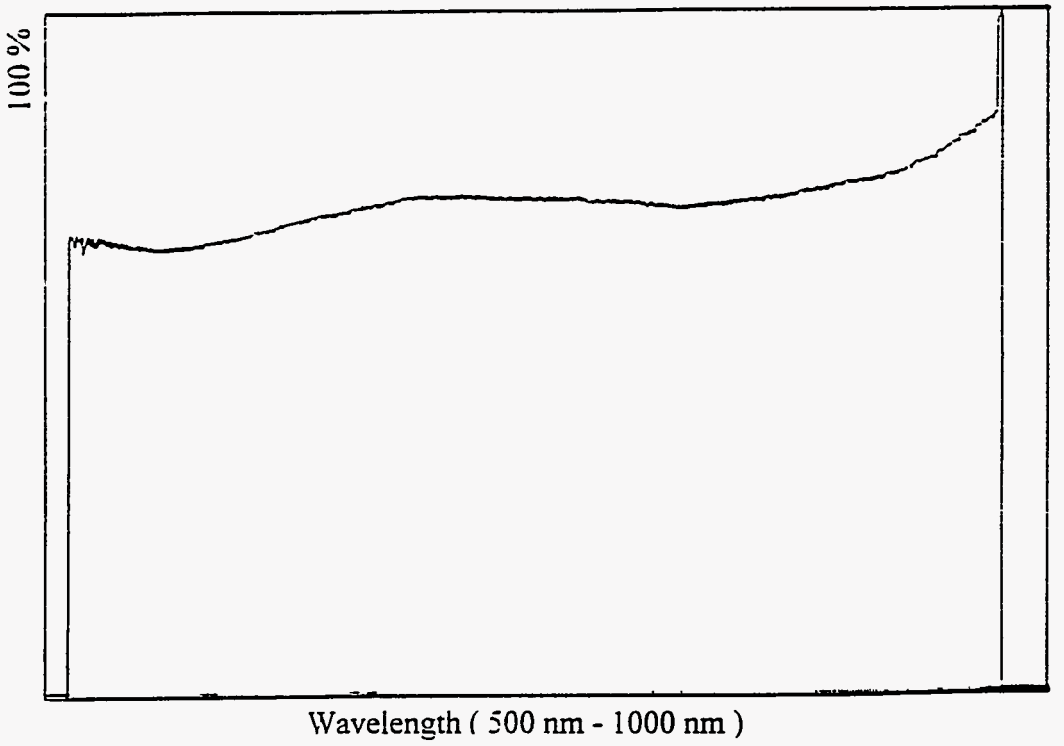


Fig. 10 Reflectance plot of a chemically etched aluminum surface (etch time = 120 sec ).

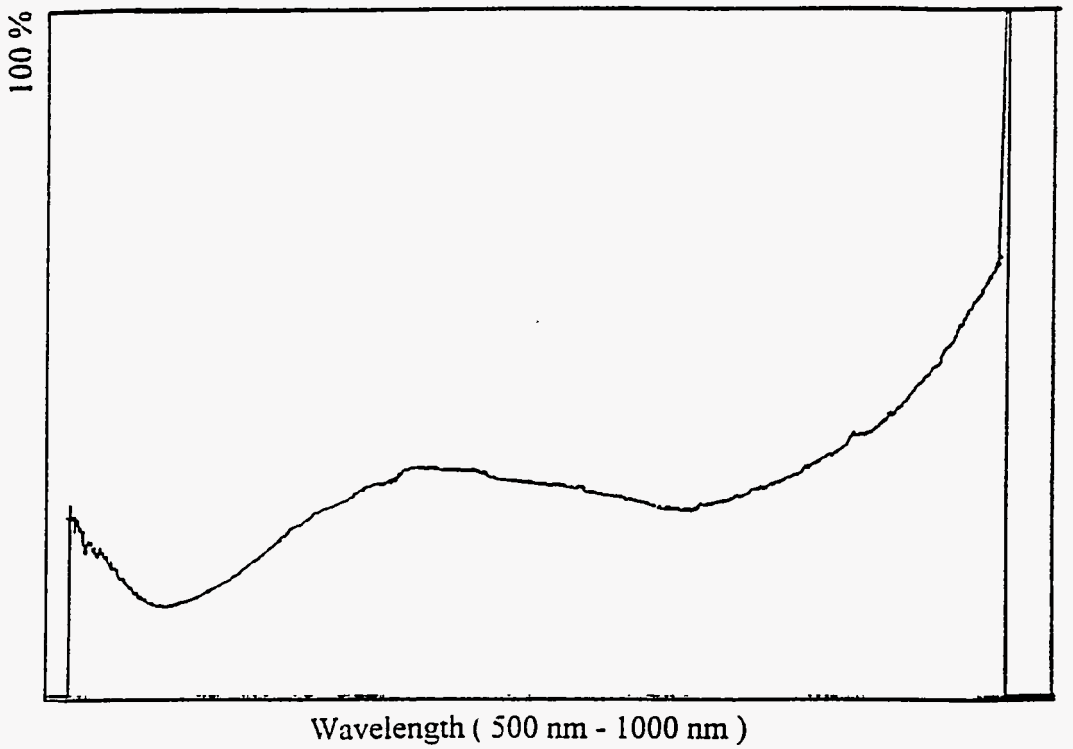


Fig. 11 Reflectance plot of a chemically etched aluminum surface (etch time = 180 sec ).