THE STUDY OF IRREGULAR SURFACES USING THE SCANNING ELECTRON MICROSCOPE

by

Robert J. Anstead
Quality Assurance Division
Systems Reliability Directorate
Goddard Space Flight Center

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INTRODUCTION

The absence of an optical instrument capable of resolving fine details of irregular surfaces at magnifications greater than 100X has long been a significant deterrent to definitive studies of these objects. The optical microscope has a very shallow depth of field, a resolution of the order of 3500Å, and is limited in practical magnification to less than 2000X. Furthermore, its depth of field, while never ideal, decreases markedly with increasing magnification. As a result, the optical microscope is of very limited value at magnifications greater than 200X if the surface under examination is not planar. Transmission electron microscopes, on the other hand, have exceptional resolving power (<10Å), are capable of very high magnifications (>150,000X), and have reasonably good depth of field. However, this instrument cannot be used to examine all specimens directly. A replica of the surface must be made and it is this replica that is examined, not the original sample. While the art of replica-making has reached a high level of sophistication, it is not always practical or even possible to make a replica.[1] Consequently, a definite gap has existed in analytical instruments available in this area.

These limitations have, to a great extent, been overcome with the development of the scanning electron microscope (SEM) as a commercially available instrument. Since the manufacture of the first practical SEM in the early 1950's,[2] the applications of the microscope have been increasing rapidly, limited, until recently, primarily by instrument scarcity.

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The detailed theory of the SEM is covered fully in Advanced Electronics and Electron Physics;[3] however, in brief the operation can be described as follows. Electrons are thermionically emitted from a heated tungsten filament and accelerated by means of an electron gun whose potential can be varied from 1 to 20kv. These electrons (electron beam) are focused on the sample by three electromagnetic lenses (some microscopes use two lenses) and pass through three collimating operture disks (Figure 1). A scanning coil is also incorporated into the column so that the electron beam can be scanned over a given area on the target sample. The size of the area is controlled by adjusting the amplitude of the scanning voltages and this variation in scanning area determines the magnification of the microscope. The primary electrons (electron beam) are either absorbed or reflected by the specimen surface. The reflected electrons lose very little of their energy and leave the surface in a direct path dependent upon the angle of incidence. The absorbed electrons lose their energy by ionizing the atoms of the sample and forming electron-hole pairs. The depth of penetration of the primary beam is dependent upon both its initial energy and the composition of the sample material. These free energetic electrons (approximately 2000 free electrons per each single 10kv primary electron[4]) diffuse randomly within the sample. Most of the electrons recombine with free holes but some reach the surface with sufficient energy to overcome the surface barrier potential and are thus emitted as low energy (1-5 ev.) secondary electrons. This secondary emission yield is determined by the angle of incidence, density of the target, and the topography of the surface.

An electron collection apparatus is placed close to the target and is biased at + (200-250) volts. This voltage is sufficient to attract the secondary electrons emitted by the sample but too weak to influence either the primary beam or the reflected electrons. The output of this collector-amplifier system is fed into a long-persistence grey-scale oscilloscope in syncronism with the scanning primary beam. This results in a visual presentation on the cathode ray tube (CRT) screen. Each point on the screen has a brightness

proportional to the number of secondary electrons emitted by the target where the primary beam impinged. The resultant picture is a high quality representation of the target surface with considerable depth of field (electron trajectory approximately 1 degree from optic axis), high resolution (depending on the size of the focused beam, <500Å), and a wide magnification range (depending on the ratio of the CRT screen size to the area of the primary beam scan, 20X to 100,000X).

A word should be given here concerning the examination of insulating materials. As explained above, the electrons ionized by the primary beam diffuse randomly within the sample, some recombining with free holes, many others going to ground, etc. However, this presupposes a reasonable mobility for the electrons which is not the case in an insulator. As a result, excess charge cannot be drained off to ground and the surface will charge negatively or positively depending on whether the secondary electron yield is positive or negative.[5] To overcome this problem electrical insulators are either coated with an antistatic liquid prior to insertion into the vacuum chamber or are coated with a thin film (≈500Å) of aluminum or gold.

APPLICATIONS

With the availability of this instrument in the GSFC failure analysis laboratories, a considerable number of different types of samples have been examined. In many cases investigations were accomplished which hitherto were either impossible or extremely difficult. Primary among these were studies of anomalies in semiconductor devices, which included such common failure modes as electrical opens in evaporated interconnects and microcracks in wire bonds. Other investigations covered such diverse reliability problems as the surface quality of ball-bearings. However, the usefulness of the SEM has not been confined solely to areas of component reliability. On the contrary, a considerable portion of the SEM effort has been directed toward solving problems for other groups within GSFC, other NASA centers, various government laboratories, industrial concerns, and universities. Studies for these organizations have included

such diverse subjects as the study of microcraters formed in thin metal foils as a result of high energy bombardment by micron-size particles, the investigation of meteorite surfaces, the application of the SEM to the study of various paleontological specimens, and the physiological investigations of the chemoreceptive hairs of the blow fly.

Semiconductor Studies

1. Electrical Opens

Electrical discontinuities in the evaporated interconnects of transistors and integrated circuits has long been a problem to both the manufacturer and the user. These discontinuities result from a variety of causes, e.g., mechanical scratches due to mishandling, dirt particles imbedded in the photoresist, electrical overstress, and various technical problems associated with the evaporation process or the etching step. Quite often these faults are situated along the planar surface of the device, and, if they are not too small, are readily discernible with optical microscopes. More subtle is when the open occurs at a step in the silicon dioxide layer.[6] Here the failure exists at a position of nonplanarity and the lack of a useful depth of field at the necessary magnifications renders the optical microscope of little value in verifying the failure mode. For reasons explained previously this lack of planarity is of no consequence to the SEM and the point of failure is easily located. This is readily apparent from Figure 2 where both an optical and SEM photographs of an electrical open in a transistor are illustrated. Figure 2a, the optical photomicrograph, gives vivid example of the power of the optical microscope in the hands of a competent microscopist. The detail of the planar transistor and the aluminum strips are clear and of great value in most studies. However, there are no details in the aluminum film at the oxide step, and any attempt to tilt the unit to better define the step at that particular site results in blurred images of limited value. The SEM photograph (Figure 2b) demonstrates very vividly the opens around the periphery of the window in the oxide layer.

2. Bond Problems

The integrity of the wire bonds used in semiconductor devices has long been one of the more serious reliability problems associated with these devices. Consequently, much study has been directed toward the determination and the correction of the failure modes responsible for these problems. Unfortunately, these bonds, because of their size (0.001" - 0.005" in diameter), shape (three dimensional), and positioning in the device header, are not easily examined. Most previous studies have entailed cross sectioning techniques[8] which, while making considerable progress in the understanding of the metallurgical aspects, were destructive in nature and had no way of correlating results with the original visual condition of the bond.

High quality visual examination and/or photographs of these bonds are now readily available using the SEM.[6] Photographs have been taken at magnifications in the range of 200X to 2500X and significant details have been resolved which could well be contributing factors towards the failures associated with these bonds. Microcracks, intermetallic formations, and extreme ratios of bond size to wire size are some of the more common anomalies seen. This type of visual evidence, together with information from pull tests, cross sections, etc., could well prove instrumental in definitively pinpointing the cause of many bond failures. Figure 3 illustrates the difference in photographic results available using optical microscopes and the SEM.

Ball-bearing Surfaces

The importance of cleanliness in the manufacture of precision ball-bearings is evident from the elaborate "clean rooms" used by most manufacturing companies. Also of great importance is the condition of (quality of) the surface of the ball itself. Excessive scratches or irregularities must be avoided. Unfortunately, the ball, being small and spherical in shape, has posed problems similar to those experienced with semiconductor bonds. In this case, the difficulty is compounded by both the uniformity of the surface

and the continuously varying slope of the surface. This latter case causes extreme difficulty in obtaining proper illumination (Figure 4). Again, although some minor difficulty occurs due to electrons reflecting from the surface of the ball, very satisfactory SEM photographs are possible (Figure 4). As a result, visual quality control inspection at magnifications >1000X is now possible of these ball-bearings.

Microcraters

Investigations are being conducted at several laboratories on methods of measuring the size and momentum of microscopic, high-energy particles, e.g., micrometeorites. One method is to bombard a thin metal foil with these particles and measure the diameter and depth of the hole. Since these particles range in size from 1 micron to 10 microns in diameter and form holes in the metal foils which are quite deep relative to the particle diameter, some type of electron microscopy is necessary for any meaningful study. NASA CR-768,[9] covers in full attempts to replicate the surface and use transmission electron microscopy. Figure 5a shows microphotographs taken using an optical microscope while Figure 5b demonstrates the details using the SEM. It is quite obvious that not only can the diameter of the hole be measured from this type of photograph, but by using two SEM photographs, each taken at a different angle ($\delta\theta \approx 5°$) of tilt of the substrate target, the depth of the hole can be computed using the methods popular with aerial photography.

Meteorite Surface

There has been much speculation concerning the metallurgical make-up of meteorites. Investigations are being conducted at GSFC and the surface of the meteorites are being studied in some detail. In order to get high quality, high magnification surface pictures the SEM has again been used. Figure 6 is a SEM photograph of a meteorite surface showing the different metallographic phases of iron and nickel existing within the meteorite. The different phases were isolated by etching.

Paleontological Studies

Paleontologists have been studying minute specimens for centuries. Many of these have posed identical problems as the bond studies to semiconductor engineers and ball-bearings to mechanical engineers. The unique problem of the paleontologist, however, is that these fossils, etc., must be sketched and catalogued regardless of the shortcomings of their optical microscopes or difficulties associated with replicas. Here the use of the SEM should make a huge impact on the operations as they now must be performed.[8]

Where previously many illustrations of paleontological specimens had to be laboriously hand drawn while viewed through microscopes, SEM photographs can be taken easily and quickly. In addition, because of the depth of field and resolution of the SEM, these photographs contain a considerable amount of detail (Figure 7) that previously was unobservable, and this should result in new insights into the make-up and/or origin of many specimens.

CONCLUSION

The above tests and illustrations give vivid demonstrations of not only the unique abilities of the SEM but also of the wide application of the instrument. Its usefulness as a tool in failure analysis studies has already been amply verified in the failure analysis laboratories at GSFC. The enthusiasm manifested by users encompassing many disciplines has further attested to the importance and the potential impact of this instrument. The studies of semiconductor failure modes and paleontological specimens have been especially enlightening as the results so far have uncovered considerable details that hitherto were unknown. It might be said that the results achieved in the short time SEM has been in operation at GSFC has only begun to test the capability of this instrument.

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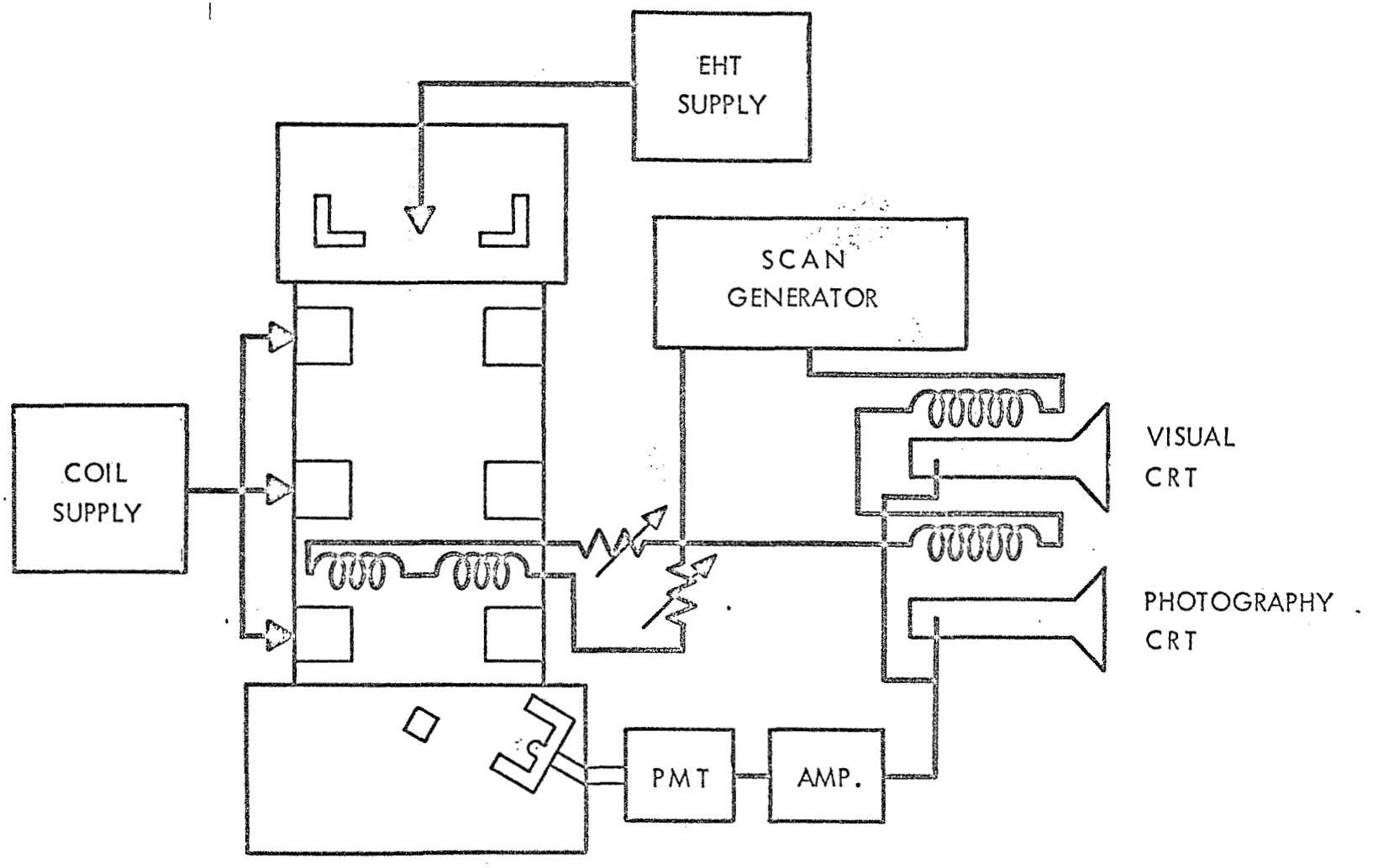


Figure 1. Schematic of the Scanning Electron Microscope

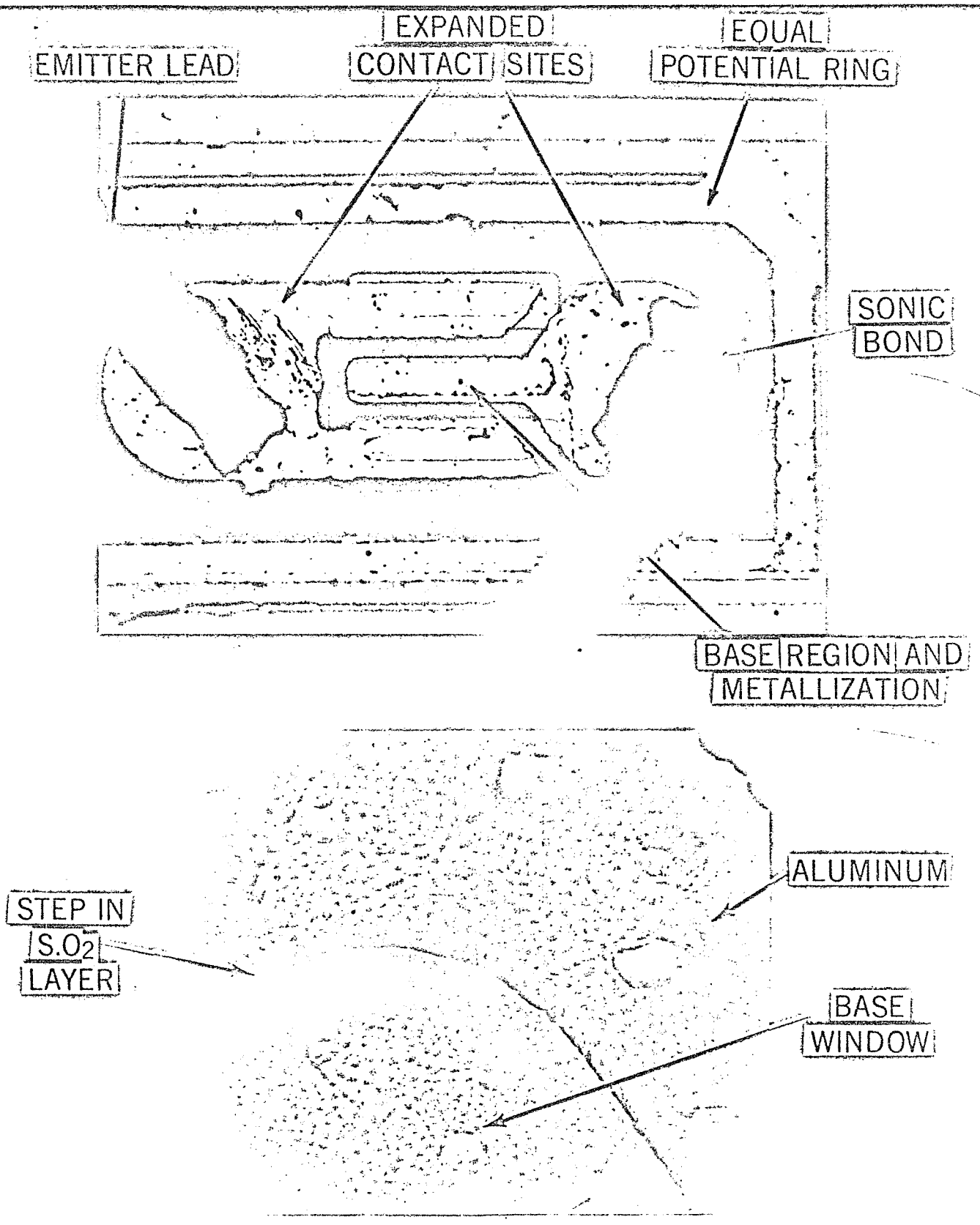
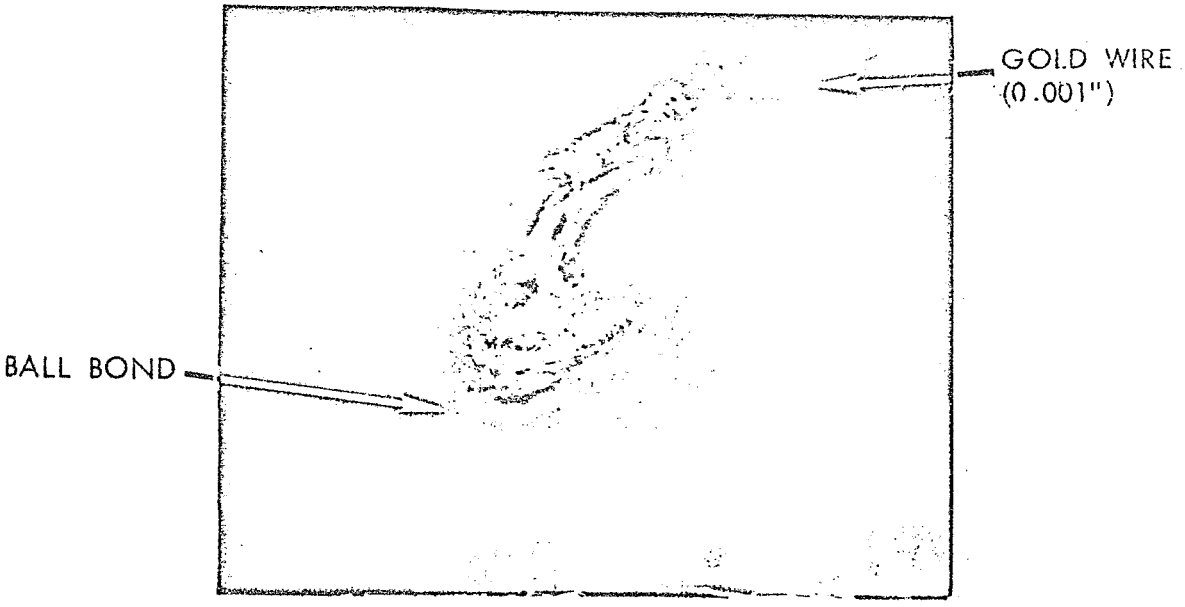
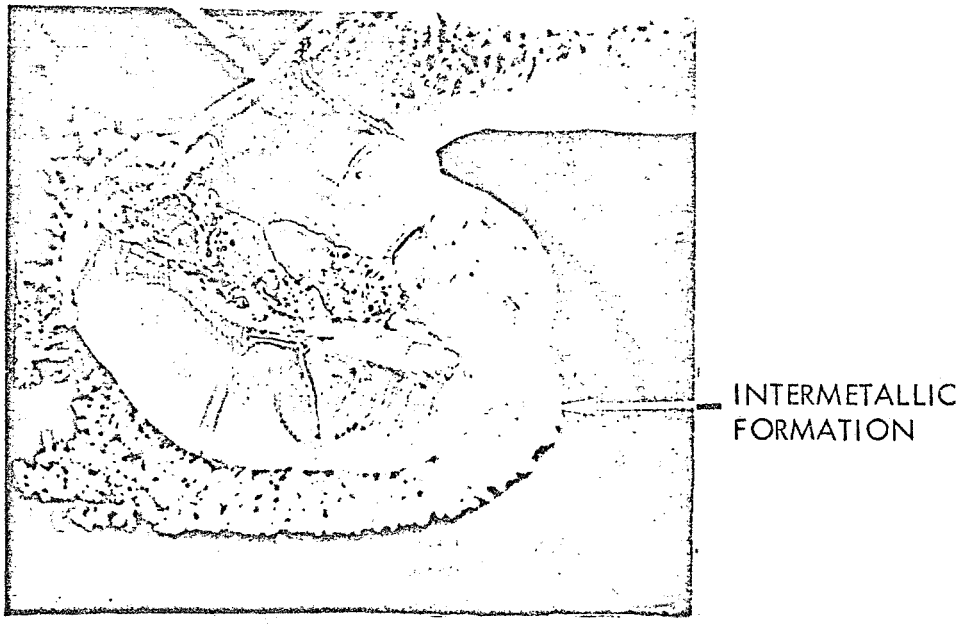


Figure 2. Comparison of Optical and Scanning Electron Microscope Photographs of Metallization Problems; Magnification (a) ≈ 500 X (b) ≈ 6500 X



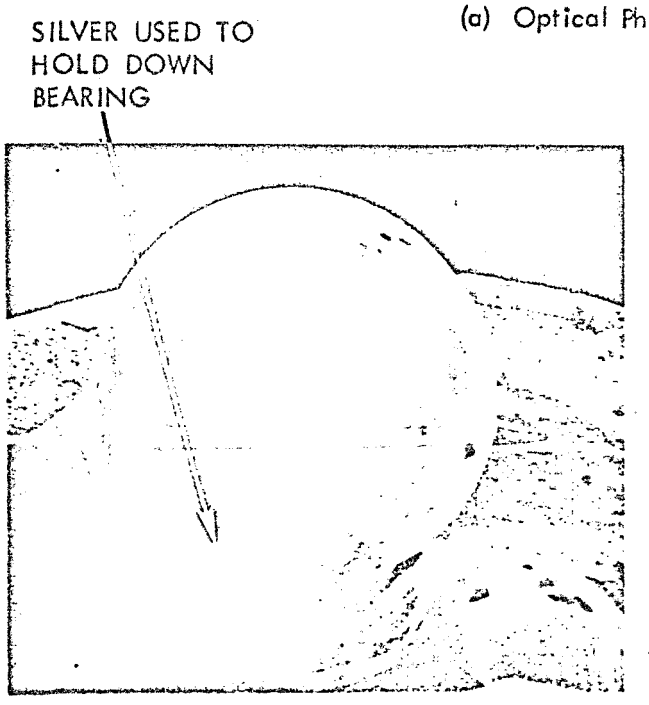
a) Optical Photograph, 500 X



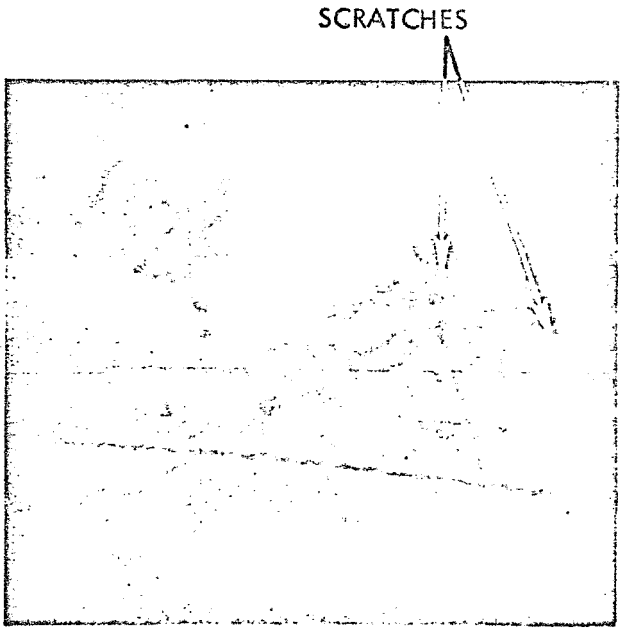
b) Photograph using Scanning Electron Microscope, 500 X

Figure 3. Comparison of Optical and Scanning Electron Microscope Photographs of a Gold to Aluminum Ball Bond

(a) Optical Photograph, 260 X

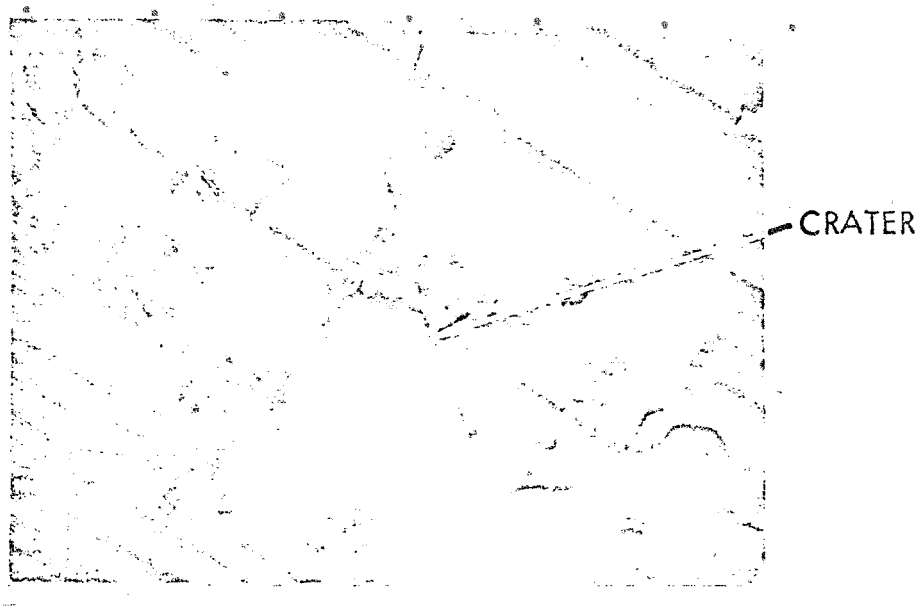


(b) SEM Photograph, 26 X

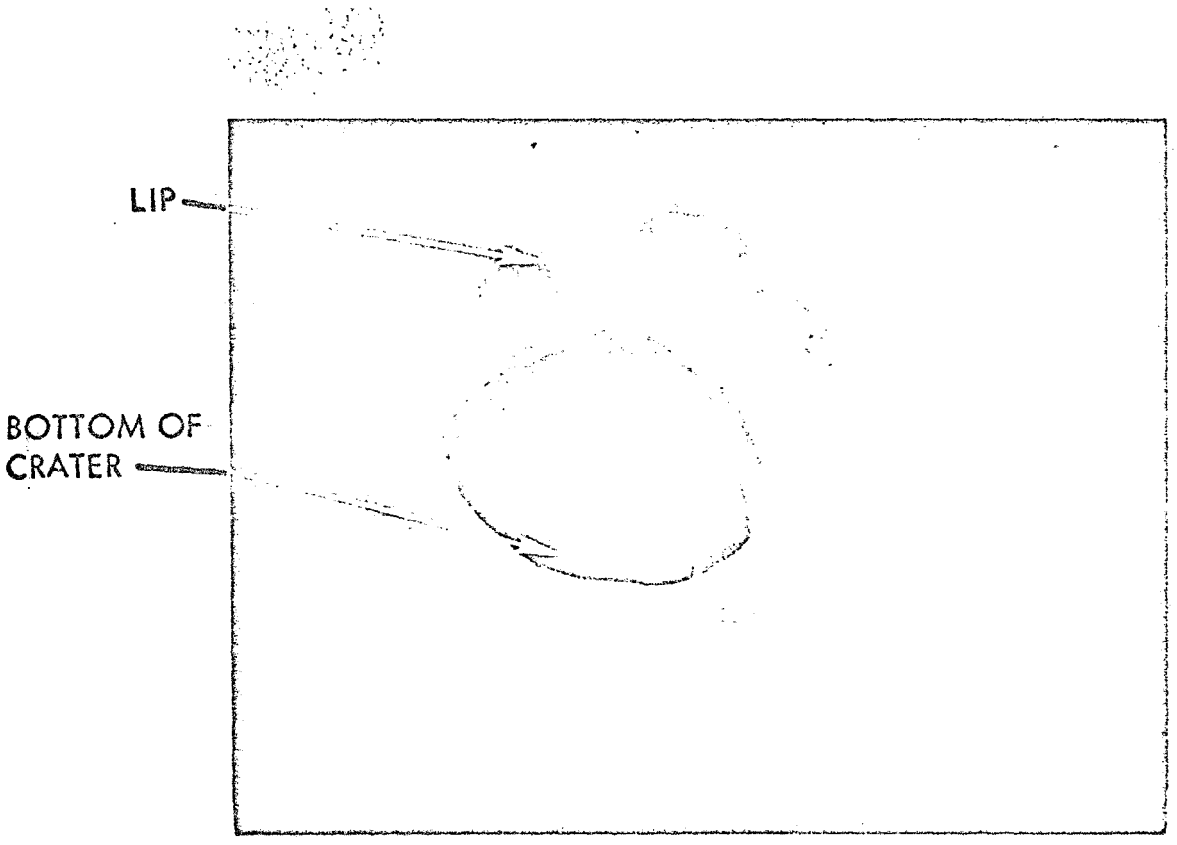


(c) SEM Photograph, 2000 X

Figure 4. Ball Bearing Studies



(a) Optical Photograph, ≈ 1000 X



(b) SEM Photograph, ≈ 6400 X

Figure 5. Optical and SEM Photographs of Microcraters in Metal Foil

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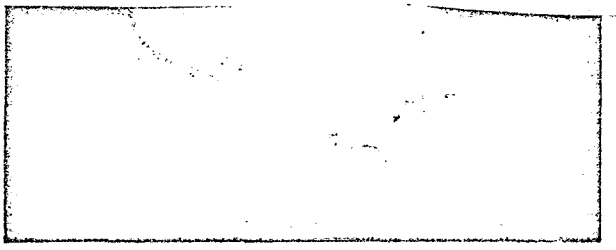
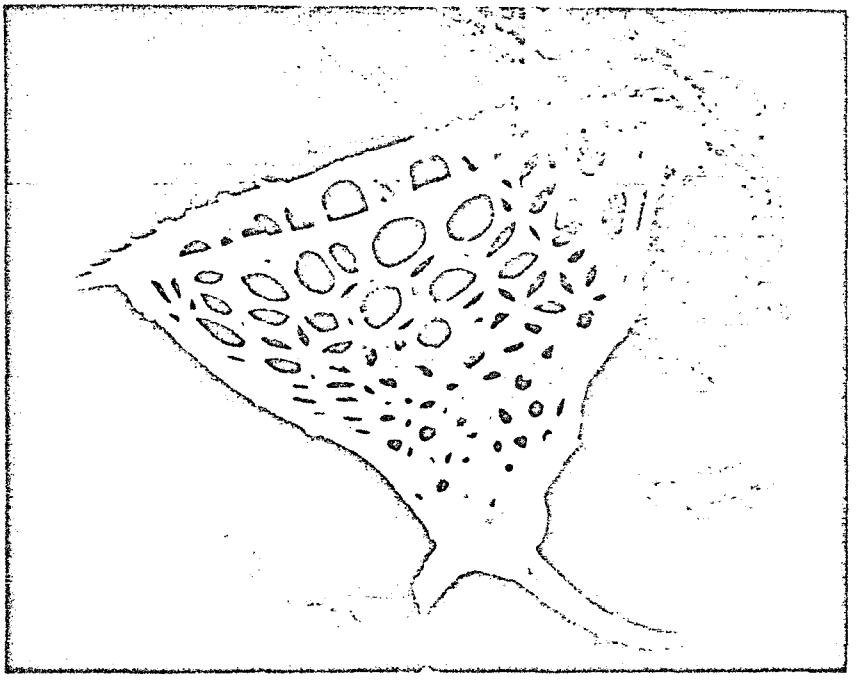


(a) Foraminifer Fossil, 250 X, (Optical)



(b) Radiolaria Fossil, 550 X, (SEM)

Figure 7. Comparison of Optical and SEM Photographs of Paleontological Specimens.