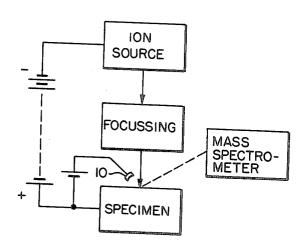
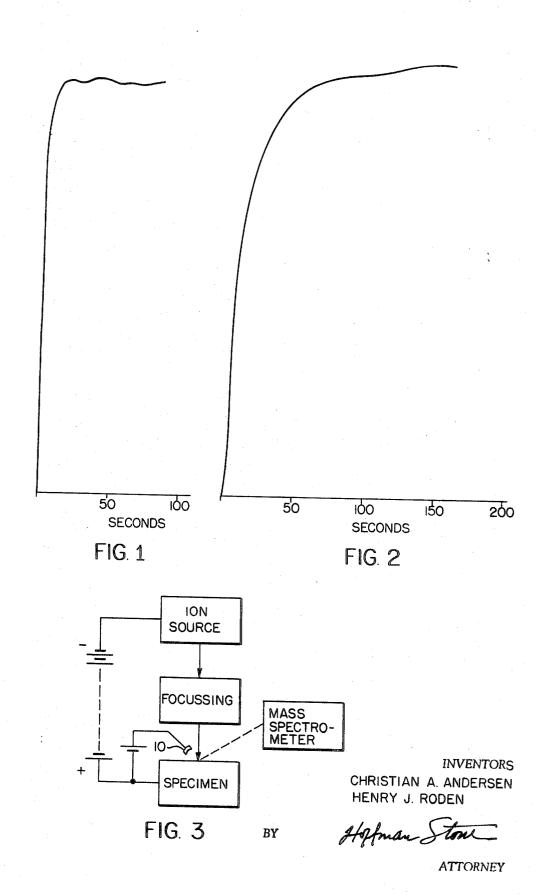
[72]	Inventors	Cristian A. Andersen Solvang;	[56] UNIT		References Cited ED STATES PATENTS	
[21] [22] [45] [73]	Appl. No. Filed Patented Assignee	Henry J. Roden, Santa Barbara, Calif. 723,026 Apr. 22, 1968 Apr. 6, 1971 Applied Research Laboratories, Inc. Sunland, Calif.	2,947,868 3,328,210 3,336,475 3,341,754 Zhurnal pgs. 63—73	8/1960 6/1967 8/1967 9/1967 OT Fekhniche	Herzog       250/49         McCaldin et al       14         Kilpatrick       2	48/1.5 50/43 /1.5X
[54]	METHOD AND APPARATUS FOR ION BOMBARDMENT USING NEGATIVE IONS 3 Claims, 3 Drawing Figs.		Primary Examiner—James W. Lawrence Assistant Examiner—A. L. Birch Attorney—Hoffman Stone			
[52]	U.S. Cl		ABSTRACT: Improved stability and control in ion bombard- ment is achieved in the case of many materials under bom- bardment by the use of negative ions. The method is especially advantageous for the analysis of insulating materials by secon-			
[51]	Int. Cl					
[50]	Field of Search				also enables improved results in ic	on im-





## METHOD AND APPARATUS FOR ION BOMBARDMENT USING NEGATIVE IONS

## **BRIEF SUMMARY**

This invention relates to a novel method of ion bombardment enabling the achievement of improved and heretofore unachievable results in processes that include the step of bombarding a material with charged particles.

Heretofore in ion bombardment work, either for spectrochemical analysis by secondary ion emission, or for other purposes such as, for example, the making of solid state electronic devices by ion implantation, the bombarding ions have been selected from among those having a positive charge. Extremely useful results have been achieved, especially in mass spectro-chemical analysis. However, it has not heretofore been possible to analyze insulating materials by this method with a reasonable degree of reliability. The surfaces of insulating and poorly conducting materials appear to become electrically charged, not only by reason of the charges delivered by the 20 impinging ions, but also by the departure of secondary electrons driven from the surfaces by the ions. After a relatively brief bombardment, the surfaces of many insulators become charged to the point where the bombarding beam is diffused, or repelled, or in some other way adversely affected so that it 25 is no longer possible even to detect the emission of secondary

It has previously been suggested that this effect could be overcome by evaporating a grid of conductive material on the surface to be bombarded so that the charges delivered to the 30 surface by the bombarding ions and created thereon by the departure of the secondary electrons could leak off, having to travel only a short distance along the insulating surface. Another previous suggestion was to place a source of electrons close to the surface of the specimen under bombardment 35 and biased negatively relative to the specimen so that electrons from the source would be drawn to the specimen to neutralize the positive charge. These expedients have been found to be of very little practical use in most instances.

Briefly, in accordance with the present invention, it has been found that the hereinabove described problems may be substantially completely overcome, and excellent results achieved if the specimen is bombarded with negative ions in place of the previously used positive ions.

The theory on which the invention is based is not understood, but is believed probably to be related to the known phenomenon that all materials release secondary electrons when subjected to ion bombardment. In the case of materials that are electrically insulating, the statistical yield of secondary electrons is greater than the number of impinging ions when the bombarding ion energies are in the range ordinarily used in microanalysis work. The electrons also greatly outnumber the yield of sputtered ions, and the charging effect on the surface of the material being bombarded seems to be primarily determined by charges delivered by the impinging ions and charges taken away by the secondary electrons. The secondary electrons seem to be usually of relatively low energy so that, as the surface under bombardment starts to charge in the positive direction due to the departure of secondary electrons in 60 greater numbers than the arriving negative ions, enough of the secondary electrons are electrostatically attracted back to the surface to establish an equilibrium condition. The small positive potential at the point of impact of the bombarding ions is insignificant compared to the acceleration imparted to the 65 rent in the ion beam was about 3×1019 amperes, the acceleratbombarding ions, which is usually in the range of about 1,000 to 20,000 electron volts.

Thus, by the use of negative ions, the heretofore disastrous effects produced by electrostatic charge accumulation on the surface of the specimen are avoided. The technique has been 70 found to be extremely useful in the operation of an ion microprobe of the type described and claimed in the copending application of Helmut J. Liebl, Ser. No. 494,388, filed Oct. 11, 1965, entitled "Ion Microprobe" and assigned to the present assignee. It is also thought that it will be found highly 75 tor.

beneficial for sputtering in general, and in other applications also, especially in connection with ion implantation. For example, a growing degree of interest is currently evident in industry in the use of ion implantation methods for making solid state electronic devices. The use of negatively charged ions in accordance with the invention is expected to facilitate the achievement of higher concentrations of implanted ions into more precisely defined regions, relative to implantation by bombardment with positive ions.

## DETAILED DESCRIPTION

The invention will now be described in greater detail in connection with the accompanying drawing, wherein:

FIG. 1 is a chart illustrating the secondary yield of Al+ ions from an aluminum specimen in response to bombardment with positive oxygen ions;

FIG. 2 is a chart on a comparable scale of the yield of Al+ ions from an alumina specimen in response to bombardment by negatively charged oxygen ions; and

FIG. 3 is a schematic block diagram of apparatus according to the invention.

Insofar as is presently known, an impinging ion produces the same effect in a material regardless of its charge in respect of sputtering of material from the specimen and the release of secondary electrons. The impinging ion is simply a submicroscopic bullet delivering energy in accordance with its velocity and mass. It appears to become electrically discharged as it approaches or meets the surface. The polarity of the charge carried by the impinging ions, therefore, appears to be immaterial in the bombardment of electrically conductive specimens. Whether the ions are electrically negative or positive, the sputtering results are substantially identical for similar beam currents and particle energies. Either positive or negative ions may be chosen on the basis of collateral considerations such as the characteristics of the ion source.

When electrically insulating specimens are to be bombarded, however, the charges carried by the bombarding ions and deposited upon the surfaces of the specimens are trapped there, and the selection of ions of the proper charge becomes a matter of vital importance.

FIG. 1 illustrates the output of secondary positive aluminum ions of atomic weight 27 sputtered from an aluminum specimen in response to bombardment by a beam of O+ ions (nascent oxygen, atomic weight 16) in an ion microprobe of the type described in the hereinabove identified copending application of Helmut J. Liebl. It is seen that within a few seconds of the start of the bombardment, the output of sputtered ions reaches a relatively high and stable value.

The use of oxygen ions in place of the more commonly used chemically inert ions such as argon ions is described and claimed in the copending patent application of Christian A. Andersen and Helmut J. Liebl, Ser. No. 678,840, filed Oct. 30, 1967, entitled "Analysis by Bombardment with Oxygen Ions," now abandoned, and the continuation-in-part thereof, Ser. No. 753,822, filed Jul. 12, 1968, entitled, "Analysis by Bombardment with Chemically Reactive Ions.'

FIG. 2 is a chart on the same scale as the chart of FIG. 1 showing the emission of secondary Al+ ions from a specimen of relatively pure alumina (Al<sub>2</sub>O<sub>3</sub>) under bombardment in the same instrument by a beam of 01 ions. In both cases, the curing potential was about 8 kilovolts, and the ion beam was defocused to cover an area of between about 20 and  $50\mu^2$ .

It is seen that the output of aluminum ions from the alumina shows a characteristic very similar to that obtained in the case of metallic aluminum. The output of sputtered ions rises rapidly to about the same relatively high and stable value. The selection of negative ions to constitute the primary beam successfully alleviates the problems heretofore encountered due to building of an electrical charge on the surface of the insula-

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The negative ions may be produced in any desired way. It is known, for example, that by properly positioning the outlet orifice of an ion source of the type known as a duoplasmatron, negative ions can be drawn from it in relatively large numbers. No claim is made in this application relative to the negative ions per se, or to the means for producing them. The invention is directed chiefly to the concept of using negative ions for bombarding materials, especially for bombarding electrically insulating materials, for any desired purpose. Also, since the nature of the matter sputtered from the bombarded specimen appears to be independent of the electrical polarity of the bombarding ions, the practice of the invention in spectrochemical work is not is any way limited in respect of the selection of sputtered ions for analysis, or of the methods used for analyzing the sputtered ions or other materials.

FIG. 3 schematically represents the ion microprobe described in the hereinabove-identified box labeled FOCUS-ING indicates a system of lenses for producing an ionic image

of the source upon the surface of the specimen.

The secondary electrons are ejected from the bombarded surface at relatively low velocities, and it is desirable to provide a collector electrode 10 (FIG. 3) fairly close to the surface to ensure against the accumulation of an excessively large negative space charge adjacent to the surface. Electrodes suitable for this purpose are normally present in all ion bombardment instruments equipped for mass spectrometric analysis of secondary ions, and instruments of this type ordinarily need no special modification for collecting the secondary electrons. In other types of ion bombardment apparatus, it may be desirable to add a simple, positively biased electrode spaced within an inch or so of the specimen to be bombarded.

The spacing and bias are not critical.

We claim:

1. Method of implanting particles of atomic dimension in a specimen of an electrically insulating material comprising the step of bombarding the specimen with negatively charged ions, imparting energy to the ions at a value selected to cause them to drive secondary electrons out of the specimen to remove negative charges at a rate to compensate fully for the negative charges carried to the surface of the specimen by the ions thereby to avoid the accumulation of an excessive negative electrical charge on the surface of the specimen.

2. Method of sputtering an electrically insulating material comprising bombarding the material with negatively charged ions to cause particles of the material to be ejected from it, imparting energy to the ions at a value selected to cause them to drive secondary electrons out of the specimen to remove negative charges carried to the surface of the specimen by the ions thereby to avoid the accumulation of an excessive nega-

tive electrical charge on the surface of the specimen.

3. Method of analyzing an electrically insulating material comprising the steps of bombarding a specimen of the material with negatively charged ions with enough energy to sputter secondary ions from the material, imparting energy to the ions at a value selected to cause them to drive secondary electrons
 out of the specimen to remove negative charges at a rate to compensate fully for the negative charges carried to the surface of the specimen by the ions thereby to avoid the accumulation of an excessive negative electrical charge on the surface of the specimen, and mass spectrometrically analyzing the
 sputtered ions.

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