# DETECTION AND DEPTH PROFILING OF <sup>19</sup>F USING RESONANCES IN THE <sup>19</sup>F(α ,p) <sup>22</sup>Ne REACTION

L.C.McIntyre Jr., J.A.Leavitt, M.D.Ashbaugh, B.Dezfouly - Arjomandy, Z.Lin, J.Oder

(University of Arizona, Tucson, AZ 85721, USA)

R.F.C.Farrow and S.S.P.Parkin

(IBM Research Division Alamaden Research Center, San Jose, CA 95120-6099, USA)
(Received November 1989)

#### ABSTRACT

Resonances in the reaction <sup>18</sup>F ( $\alpha$ , p) <sup>22</sup>Ne have been used to detect and depth profile <sup>18</sup>F in solid targets. Incident alpha particles in the range 2100—2500 keV were used and protons were detected at  $\theta=135^{\circ}$  with a large solid angle surface barrier detector covered to stop elastically scattered alpha particles. This technique is a simple, nuclide specific probe and is particularly useful in detecting <sup>18</sup>F in the presence of heavy elements such as GaAs where conventional Rutherford backscattering is ineffective. Examples using this technique on epitaxially grown thin films containing LaF<sub>3</sub> layers will be presented.

Key words: Fluorine Depth profile Nuclear reaction analysis

# I. INTRODUCTION

In the ion beam analysis of thin films or near surface layers of bulk materials, it is often desired to detect and depth profile light elements in the presence of a matrix containing heavier elements. Conventional Rutherford backscattering spectrometry (RBS) using MeV beams of He ions is usually not effective in such cases. We report here an investigation of the use of resonances in the <sup>19</sup>F ( $\alpha$ , p) <sup>22</sup>Ne nuclear reaction to detect and depth profile <sup>19</sup>F. If a single isolated narrow resonance is available, the concentration distribution of <sup>19</sup>F can be determined by observing the yield of protons as a function of incident He energy. Complications arise if the sample thickness and resonance spacing are such that more than one resonance produces protons for a given incident energy. General techniques of nuclear reaction analysis, including the resonance technique discussed here has been reviewed by Amsel et al. <sup>[1,2]</sup>

Previous work on the use of this reaction for near surface analysis of  $^{19}F$  has employed a resonance at 2443 keV incident He energy  $^{[3,4]}$ . An alternative resonance technique involving the  $^{19}F$  (p, $\alpha$   $\gamma$  )  $^{16}O$  reaction has been used by several authors to depth profile  $^{19}F^{[5-11]}$ . The ( $\alpha$  , p) reaction has also been used for analysis of  $^{31}P^{[12]}$ , including work done in this laboratory  $^{[13]}$ .

We report here a measurement of relative proton yield from the  $^{19}F$  ( $\alpha$  ,p)  $^{22}Ne$  reaction from a MgF<sub>2</sub> target at a reaction angle of 135° for incident alpha particle

energies from 2100 to 2500 keV. This information is then used to investigate the location of <sup>18</sup>F in two epitaxial thin films containing LaF<sub>3</sub> layers in addition to layers of pure rare earth or other elements. Some of these films are quite thick and more than one resonance is involved at a given beam energy. A computer simulation was used to handle such cases.

# **II. RELATIVE YIELD MEASUREMENT**

A beam of 'He<sup>+</sup> ions was obtained from the University of Arizona 5.5 MV CN (High Voltage Engineering Corp) Van de Graaff accelerator.

Protons were detected in a 450 mm<sup>2</sup> area Si surface barrier detector located 25 mm from the target at an angle of 135°. The solid angle subtended is about 0.7 sr. The detector was covered with a 25  $\mu$  m Mylar foil to stop backscattered alpha particles. The energy loss of a 3 MeV proton in this cover is approximately 360 keV. This is the same arrangement used in our work on the <sup>31</sup>P ( $\alpha$ , p) <sup>34</sup>S reaction <sup>(13)</sup>.

Fig. 1 is an example of a proton spectrum obtained with the detector described above. The target was a  $MgF_2$  film described below. Two proton peaks are visible, one  $(p_0)$  resulting from a reaction leaving <sup>22</sup>Ne in its ground state, the other  $(p_1)$  from a reaction leaving <sup>22</sup>Ne in its first excited state at 1.28 MeV<sup>[14]</sup>.

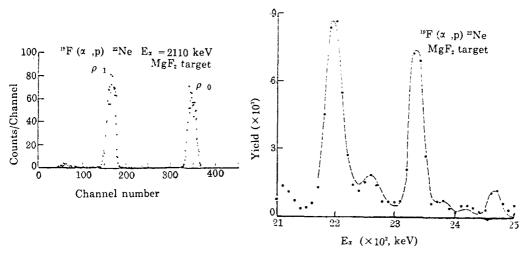


Fig.1 Proton spectrum from the

<sup>19</sup>F (α,p) <sup>22</sup>Ne reaction resulting
from bombardment of a MgF<sub>2</sub>
target with 2110 keV alpha particles
The energy dispersion is 7.5 keV/channel

Fig.2 Yield of <sup>16</sup>F (α, p) <sup>22</sup>Ne protons as a function of incident alpha- particle energy from a MgF<sub>2</sub> target with a Ta cover
The dashed line is a computer simulation described in the text. The simulation is normalized to the data

Fig. 2 shows  $p_0$  proton yield as a function of incident He<sup>+</sup> energy from 2100 to 2500 keV in 10 keV steps. The target was a 24  $\mu$  g/cm<sup>2</sup> (approximately 76 nm) MgF<sub>2</sub> film on

a carbon backing covered with a 33  $\mu$  g/cm<sup>2</sup> Ta layer (approximately 20 nm). The energy loss of the He beam was about 11 keV in the Ta layer and 26 keV in the MgF<sub>2</sub> layer. Two large and several smaller resonances are evident. This reaction has been studied by several investigators in this energy range<sup>[16-17]</sup>. Our results are in general agreement with the results of Kuperus et al.<sup>[15]</sup> taken at 120°.

Notice that the energy of the resonances is shifted by 11 keV in Fig. 2 because of the Ta cover. Also, the width of the peaks is determined by the thickness of the MgF<sub>2</sub> layer, which is greater than the reported<sup>[15]</sup> width of the resonances in every case. The resonance at 2315 keV is sufficiently isolated to be useful in depth profiling <sup>19</sup>F in layers where the incident He ion beam energy loss is less than about 25 keV. This corresponds to about 100 nm is Si. The resonance at 2445 keV has heen used previously for this purpose<sup>[3,4]</sup>, however the yield is much less than the one at 2315 keV under our experimental conditions.

## III. COMPUTER SIMULATION OF THE YIELD CURVE

We have written a computer program to simulate the proton yield from the <sup>19</sup>F (α, p) <sup>22</sup>Ne reaction from a film containing <sup>19</sup>F. This is useful if the film is thick enough so that several resonances contribute to the yield at a given incident beam energy. Eight resonances between 2100 and 2500 keV were included. Energy independent Bohr straggling was used and Bragg-rule linear additivity was assumed for both energy loss and straggling<sup>[18]</sup>. The program is similiar in spirit to that of Simpson and Earwaker<sup>[10]</sup>.

We present in the next section some examples which use this program to simulate the proton yield from He bombardment of some multilayer samples containing <sup>19</sup>F in some of the layers.

## IV. EXAMPLES OF 19F DETECTION IN MULTILAYER FILMS

Proton yield curves were obtained from He bombardment of two films containing LaF<sub>3</sub> layers. These films were epitaxially grown by MBE on GaAs substrates. Their nominal composition is given in table 1.

Table 1
Sample composition

Layer	Sample A	Sample C	
1	10 nm Ag	55nm LaF <sub>3</sub>	
2	40nm Dy	160nm Dy	
3	180nm Er	240nm LaF <sub>3</sub>	
4	10 nm LaF <sub>3</sub>	_	
5	-	<b></b>	

To obtain information about the heavy elements in these films, Rutherford

backscattering spectra were taken with 1892 keV He<sup>+</sup> ions at a scattering angle of 170°. These spectra are shown in Figs. 3(a), 4(a). Notice that the RBS spectra contain essentially no information concerning <sup>19</sup>F content. Elastic scattering from <sup>19</sup>F appears at about channel 440 in the RBS spectra shown here. The large signal due to the GaAs substrate prevents accurate measurements of the <sup>19</sup>F signal.

The results of  $^{19}$ F ( $\alpha$ ,  $p_0$ )  $^{22}$ Ne proton yield measurements are shown in Figs. 3(b) and 4(b). The measurements were taken with a 100 nA beam for a total collected charge of 20  $\mu$  C per point. No beam damage was detected on the samples reported here; however, beam damages at these current densities has been observed on other samples. Computer simulations, calculated as described in the last section, are shown

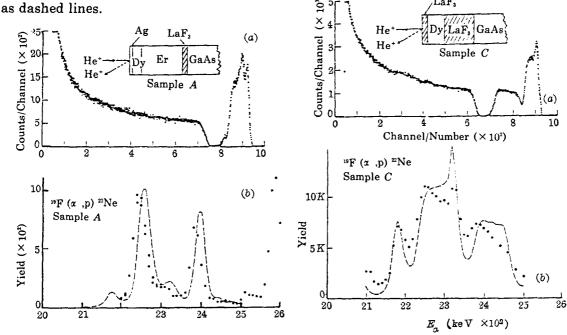


Fig.3 RBS spectrum of 1892 keV alpha particles (a) and Yield of  $^{16}$ F ( $\alpha$  , $p_0$ )  $^{22}$ Ne proton as a function of incident alphaparticle energy on sample A(b)

Fig.4 RBS spectrum of 1892 keV alpha particles (a) and Yield of "F ( $\alpha$  , $p_0$ )

"Ne proton as a function of incident alpha- particle energy on sample C(b)

The dashed line is a computer simulation described in the text. The simulation is normalized to the data.

Sample A contains one LaF<sub>3</sub> layer located underneath three layers of heavier elements. The energy loss of the incident He<sup>+</sup> beam in the top three layers is approximately 85 keV. The data and simulation are roughly consistent with the assumed composition, resulting in a resonance yield similiar to that obtained with the MgF<sub>2</sub> target but shifted by the energy loss in the top layers. The discrepancy of less than 10 keV between the data and simulation is not understood.

0//

Sample C consists of two LaF<sub>3</sub> layers separated by a layer of Dy. The two major resonances are evident in each of the LaF<sub>3</sub> layers. We hesitate to draw any detailed conclusions concerning <sup>19</sup>F distribution in such thick targets until more experimental work on this technique is done, however, the gross features appear to be correctly modeled.

## V. CONCLUSION

We have demonstrated the use of the  $^{19}F(\alpha,p)$   $^{22}Ne$  reaction in detecting and depth profiling  $^{19}F$  in multilayered thin films. The films we used were not favorable cases for accurate measurement of effects such as minor diffusion of  $^{19}F$  into adjoining layers. The gross distribution, however, was easily determined. Absolute concentrations of  $^{19}F$  in the near surface region can be measured by comparing with a standard such as  $MgF_2$ .

The lack of background in the proton spectra results in good sensitivity for <sup>19</sup>F detection. We estimate that a concentration of about 10<sup>19</sup>F atoms/cm³, which is less than 0.01 atomic percent in SiO<sub>2</sub>, could be detected in a reasonable time.

## **ACKNOWLEDGEMENT**

The authors would like to thank Peter Stoss for his excellent work in maintaining and operating the Van de Graaff accelerater. We also thank L. Lingg, C. Hwangbo, and J.Lehan for providing the MgF<sub>2</sub> film. This work was partially supported by the Optial Data Storage Center and the Center for Thin Film Studies (AFOSR-URI), both administered through the Optical Sciences Center, University of Arizona.

#### REFERENCES

- [1] G.Amsel et al., Nucl. Instr. Meth., 92 (1971),481.
- [2] G.Amsel and W.A. Lanford, Ann. Rev. Nucl. Part. Sci., 34 (1984), 435.
- [3] P.J.M. Smulders, Nucl. Instr. Meth., B14 (1986), 234.
- [4] F.G. Kuper et al., J. Appl. Phys., 60 (1986),985.
- [5] B.Maurel et al., J. Electrochem. Soc., 119 (1972), 1715.
- [6] M.Croset and D.Dieumegrad, J. Electrochem. Soc., 120 (1973), 526.
- [7] J.W. Mandler et al., Thin Solid Films, 19 (1973), 165.
- [8] D.Dieumegard et al., Nucl. Instr. Meth., 168 (1980), 93.
- [9] G.Deconninck and B.Van Oystaeyen, Nucl. Instr. Meth., 218 (1983), 165.
- [10] J.C.B. Simpson and L.G. Earwaker, Nucl. Instr. Meth., 7 B15 (1986), 502.
- [11] T.E. Derry et al., Nucl. Instr. Meth., B35 (1988), 431.
- [12] P.Mazzoldi and G.Moschini eds., Proceedings of the internation symposium on three day in depth review on the nuclear accelerator impact in the interdisciplinary fields, Laboratori Nationali di Legnaro (Padova) Italy, May 30-June 1, 1984, p.66.

- [13] L.C. McIntyre Jr. et al., Nucl. Instr. Meth., B35 (1988), 446.
- [14] F.Ajzenberg Selove and T.Lauritsen, Nucl. Phys., 11 (1959), 1.
- [15] J.Kuperus, Physica, 31 (1965), 1603.
- [16] W.A. Schier et al., Nucl. Phys., A266 (1976), 16.
- [17] J.Cseh et al., Nucl. Phys., A413 (1984), 311.
- [18] W.K. Chu et al., Backscattering spectrometry, Academic Press, New York, 1978, Chapt 2.