# Nondestructive measurement of the composition of advanced armor materials by ion beam analysis.

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## ABSTRACT

Lightweight, state-of-the-art armors rely on ceramics for their enhanced performance. One goal of the United States Army is to expand the industrial base of companies that provide the armors. A systematic study of armor performance as a function of ceramic stoichiometry will result in a better understanding of the fundamental relations between composition and mechanical performance. One ceramic of interest is aluminum oxynitride (AlON). The stoichiometries of representative samples of AlON were investigated with the nondestructive techniques of Rutherford backscattering spectrometry and resonant nuclear reaction analysis. Future tests of the performance of the armor samples are to be correlated with the stoichiometries and hence, will lead to optimum, standardized processes for the manufacture of each of the ceramics investigated.

## 1. INTRODUCTION

The U.S. Army seeks advanced materials for next-generation armor systems that will provide increased survivability of soldiers and combat vehicles. Specifically, hard ceramic materials can dramatically improve performance over current metal armor at reduced weights. Ceramics also have the advantage of being transparent through much of the electromagnetic spectrum [1].

One new material being considered for optically transparent windows is aluminum oxynitride, AlON, a fully dense polycrystalline material that transmits in the visible. AlON offers advantages over glass and sapphire armor windows because it can be used in thinner and shaped panels. The Army is pursuing ways to produce less expensive raw materials and ways to manufacture large panels. Changes in starting materials and processes can and do change the resultant properties [2].

The ballistic behavior of ceramics used in armor increasingly relies on understanding the fundamental relations between the composition and structure of the ceramic and the performance under dynamic loading at strain rates of ~$10^5$. Ballistic experiments are expensive in cost and time. Thus, insight gained through a fundamental study of compositional change vs. properties can improve understanding and reduce development time.

AlON samples were formed using the hot isostatic press method over the range of 30-40 mol% aluminum nitride (AlN) in alumina ($Al_2O_3$). Small variations of a few atomic percent in the elements in the AlON can dramatically affect its performance. Systematic study of the composition is the first step to understanding the effects of those variations on both processing the AlON and its armor performance [2, 3].

## 2. APPLICATION OF THE ION BEAM TECHNIQUES

Rutherford backscattering spectrometry (RBS) is the primary ion beam technique employed at the Army Research Laboratory to nondestructively determine elemental concentrations. As shown in Figure 1 for Rutherford backscattering spectrometry (RBS), ions incident on a sample elastically scatter from nuclei via the Coulomb force, emerge from the sample, and are energy-analyzed to determine elemental concentrations [4,5].

RBS was performed in the IBM geometry on the AlON samples with 2-MeV He+ ions. The backscattered ions were detected with a 100-% efficient silicon-surface-barrier detector that was located at 170° and that subtended a solid angle of 5.2 msr. The RBS spectrum in Figure 2 indicates the presence of nitrogen in the AlON sample; however, quantitative analysis is difficult. Rutherford Universal Manipulation Program (RUMP) simulations for nitrogen concentrations from 0-10% are essentially indistinguishable [6]. The shape of the RBS spectrum does indicate that the sample is of uniform composition.

Resonant nuclear reaction analysis (RNRA) provides an alternative to RBS for measuring elemental concentrations. As shown in Figure 3 for RNRA, ions enter a sample and react with sample nuclei to form product nuclei that de-excite through the emission of characteristic γ-photons. An enhancement in the reaction cross section, a resonance, occurs when the ion energy plus the mass excess (the mass of the reactants minus the mass of the products in energy units) match an energy level in the product nucleus. Cross sections for these resonant reactions are significantly larger, often by orders of magnitude, than the cross sections for non-resonant reactions [4, 5, 7].

The number of characteristic γ-photons emitted is directly proportional to the concentration of the reaction nuclei in the sample at the depth corresponding to the resonant energy. For a sample of uniform composition, such as the AlON, the concentration of an element in that sample is independent of depth into the sample and, therefore, of ion energy above the resonant energy. The $^{27}Al(p,\gamma)^{28}Si$ and the $^{15}N(p,\alpha\gamma)^{12}C$ reactions were used to measure concentrations of the aluminum and of the nitrogen with a 1-MeV proton beam. Photon spectra were collected with a 2.75-inch diameter by 3.50-inch long cylindrical BGO detector surrounded by a 7-inch long by 7-inch outer-diameter NaI(Tl) annulus operating in anti-coincidence mode [8, 9]. The face of the BGO/ NaI(Tl) detector combination was located 4 inches from the samples at 90° relative to the incident proton beam. As shown in Figure 4, reactions involving oxygen

isotopes are not discernable in the AlON. This is due to a small reaction cross section ( $^{16}O$) or to low isotopic abundance ($^{18}O$) [10].

## 3. DATA REDUCTION AND ANALYSIS

Individually, RBS and RNRA do not yield the complete compositional information for the AlON samples; however, the relative compositions can be determined by combining the results from the two techniques. RNRA is used to fix the ratio of aluminum to nitrogen. The RNRA results are used in RUMP simulations of the RBS data to determine the atomic percentages of all three elements.

Photon spectra were collected for an aluminum standard, an aluminum nitride standard, and the AlON samples. The net counts in the aluminum peaks were determined by standard background subtraction methods [9]. The net counts in the nitrogen peaks were determined by performing the standard background subtraction and then subtracting the charge-normalized aluminum standard spectrum from the sample spectra. Typical spectra obtained by this method are shown in Figure 5. The ratio of aluminum to nitrogen in the AlON samples is given by

$$\frac{n_N}{n_{Al}} = \left( \frac{x_N / x_{Al}}{y_N / y_{Al}} \right)$$

where $n_N$ is the number of nitrogen atoms in the sample, $n_{Al}$ is the number of aluminum atoms in the sample, $x_N$ is the net counts in the nitrogen peak of the sample, $x_{Al}$ is the net counts in the aluminum peak of the sample, $y_N$ is the net counts in nitrogen peak of the AlN, and $y_{Al}$ is the net counts in the aluminum peak of the AlN.

With the RNRA-determined values for the Al-to-N ratio in AlON samples, the atomic percents of Al, N, and O in the samples can be determined from the RBS data. For each sample, the atomic percents of the Al, O and N are varied in RUMP while maintaining the Al-to-N ratio determined by RNRA until a best fit to the RBS data is achieved.

## 4. RESULTS, SUMMARY, AND PREDICTIONS

Individually, RBS and RNRA did not yield the complete compositional information for samples of AlON, a possible next-generation armor material. We have successfully combined RBS and RNRA data to extract that composition for the samples. As shown in table 1, the atomic percentage of nitrogen in AlON can vary greatly depending on the starting materials and conditions and on the processing of those materials.

The further systematic investigation of the relation between composition and performance of AlON and related materials will contribute to the development of improved armor systems needed by the Army and to the reduction in both the cost and time required for their development. Ion beam techniques, such as RBS and RNRA, will continue to play a role in those investigations.

**References**


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**Figures**

1. Schematic of Rutherford backscattering spectrometry.

2. Typical RBS spectrum of AlON using a 2-MeV He+ beam. The small atomic percentage of nitrogen makes it difficult to distinguish the nitrogen and, thus makes quantifying the nitrogen also difficult.

3. Schematic of resonant nuclear reaction analysis.

4. Typical RNRA spectrum of AlON. Oxygen is not visible due to the small reaction cross section ($^{16}O$) for or the low isotopic abundance ($^{18}O$) of the oxygen isotopes.

5. Background-subtracted, charge-normalized RNRA spectra for (a) Al, (b) $Al_2O_3$, (c) fully dense AlON , and (d) AlN.

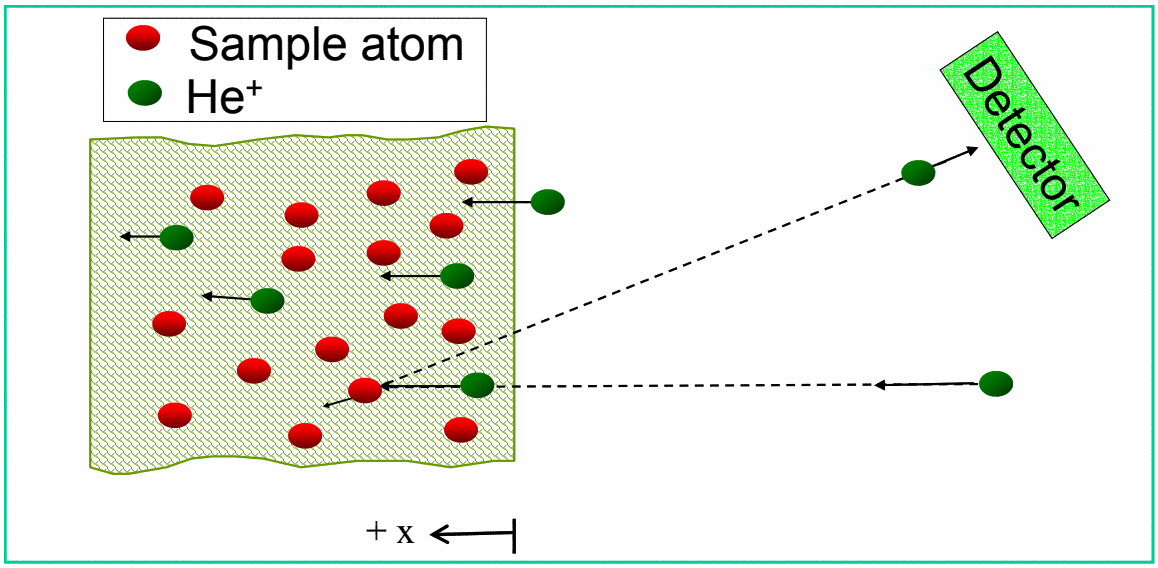


Figure 1. Schematic of Rutherford backscattering spectrometry.

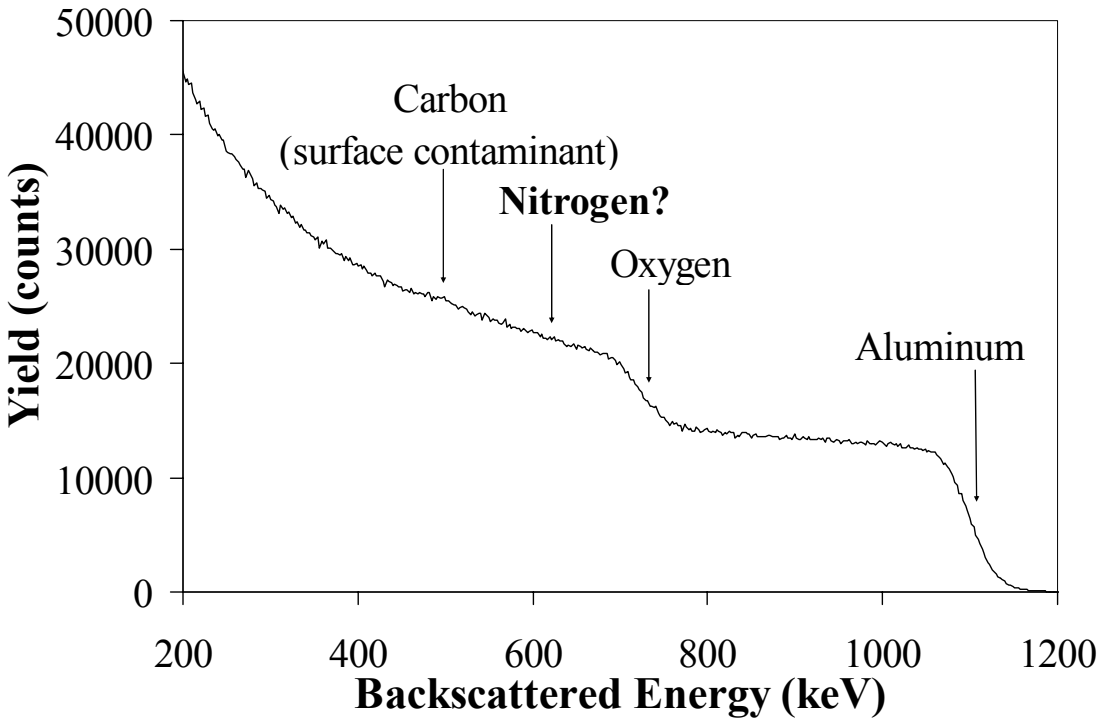


Figure 2. Typical RBS spectrum of AlON using a 2-MeV He+ beam. The small atomic percentage of nitrogen makes it difficult to distinguish the nitrogen and, thus makes quantifying the nitrogen also difficult.

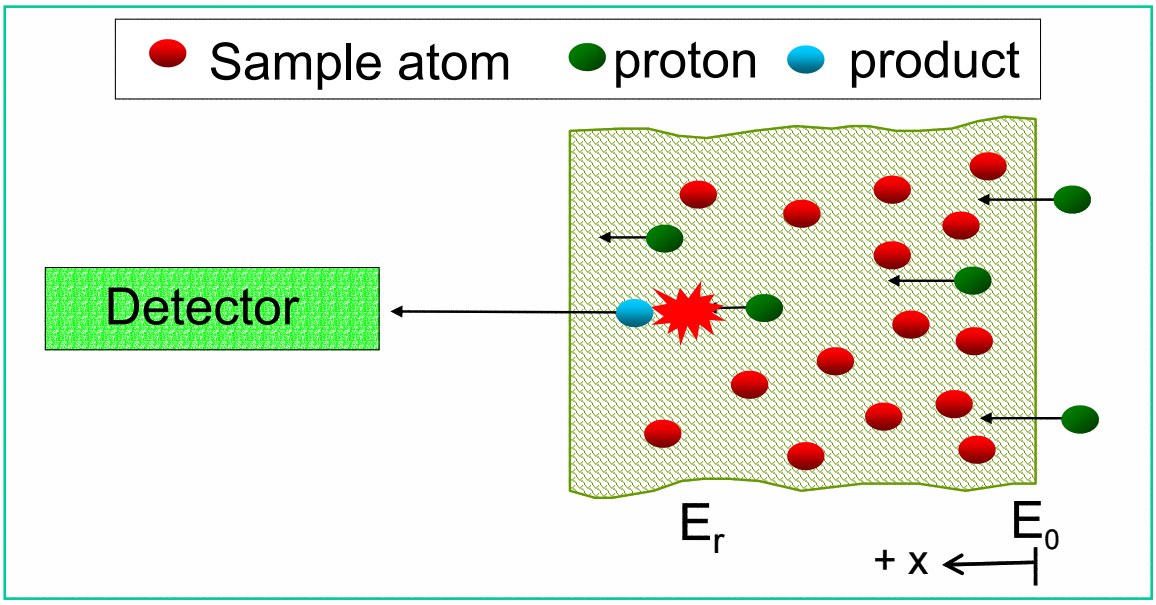


Figure 3. Schematic of resonant nuclear reaction analysis.

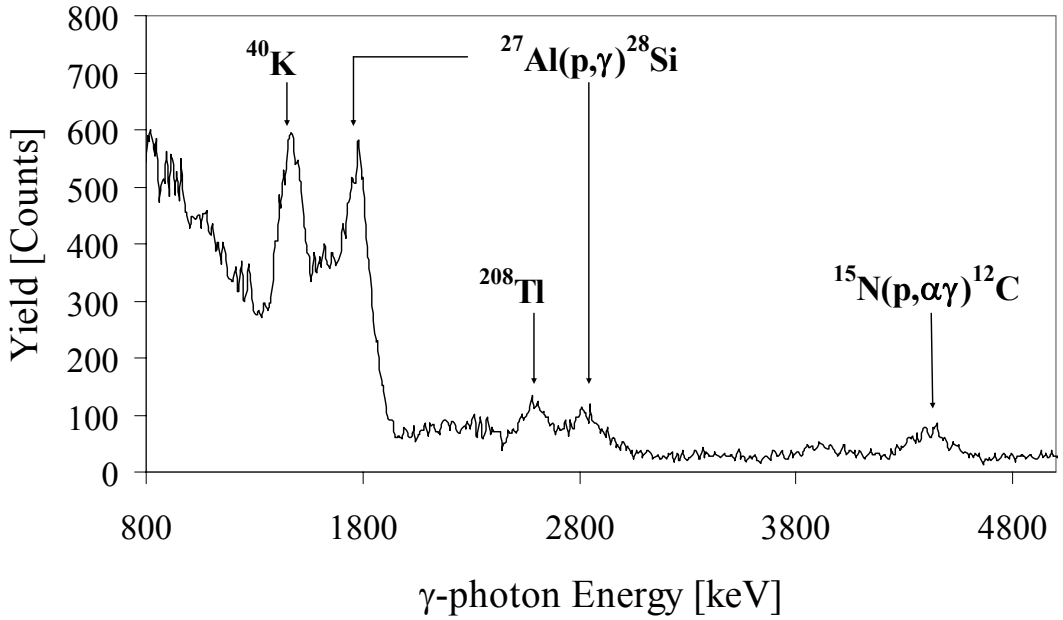


Figure 4. Typical RNRA spectrum of AlON. Oxygen is not visible due to the small reaction cross section ($^{16}$O) for or the low isotopic abundance ($^{18}$O) of the oxygen isotopes.

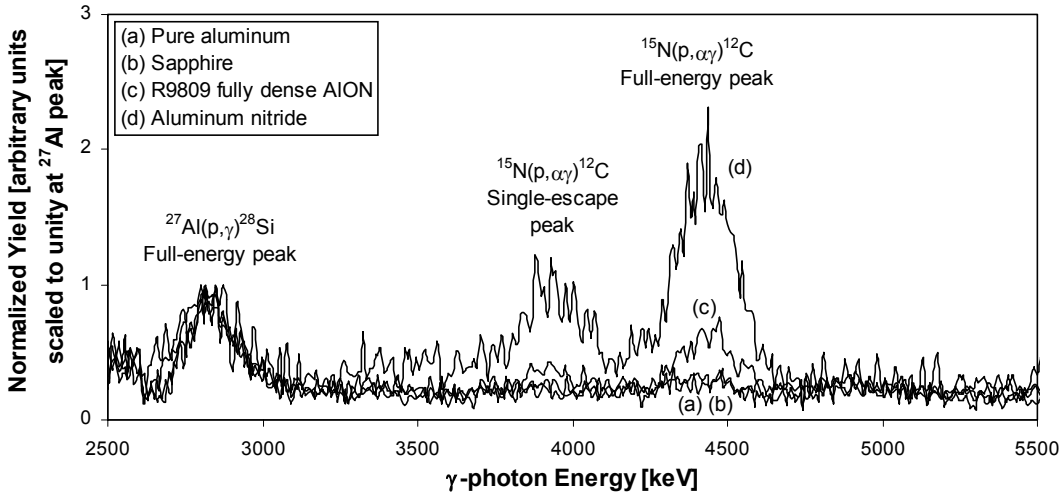


Figure 5. Background-subtracted, charge-normalized RNRA spectra for (a) Al, (b) $Al_2O_3$, (c) fully dense AlON , and (d) AlN.

Table 1. Atomic percentages [at%] of the elements in the five AlON samples investigated. R9809 is the fully dense, commercial sample. Of the laboratory samples, the conditions used to prepare AlON36 have successfully incorporated nitrogen.

| | R9809 | AlON38 | AlON36 | AlON30 | AlON28 |
|---|---|---|---|---|---|
| Al [at%] | 35.7 | 39 | 35.6 | 37 | 40 |
| O [at%] | 55.3 | 60 | 56.8 | 62 | 60 |
| N [at%] | 9 | <1 | 7.6 | <1 | <1 |