

Aluminum Oxynitride Armor

Production and Modeling of Next Generation
Transparent Armor for the Global War on Terror

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ABSTRACT

The use of novel materials in conventional armor solutions has been difficult due to extant processing methods and expensive methods. This is especially apparent in the use of aluminum oxynitride (AlON), currently being researched for its application in transparent ceramic armors. Development of a reaction sintering process utilizing unique additives in the direct sintering of alumina and aluminum nitride powders can reduce costs and provide a more practical manufacturing process for the delivery of next generation armor to the military in a timely fashion. Using a liquid sintering process and aluminum phosphate as an additive it is possible to sinter AlON directly while decomposing phosphate and alumina from the sample. It was found that this approach formed the requisite γ -AlON phase and the densities of samples could approach 3.63 g/cc. Based on these results, and subsequent ballistic testing of larger scale samples, it was concluded that AlPO_4 is a viable additive in a reaction sintering process to improve density and transparency of AlON.

1. INTRODUCTION

In the spring of 2005 the Department of Defense and Department of Energy began a joint research project to study the effects of the increasingly sophisticated Improvised Explosive Devices (IEDs) being fielded in Iraq and Afghanistan. In addition to modeling the explosives and their interactions with current military armors, the study also funded a small offshoot group to research novel armor solutions that could replace existing systems with lightweight, modular designs capable of defeating both ballistic, and shockwave related threats.

With escalating conflicts in two theaters of operation, the US Military's legacy combat systems were quickly becoming obsolete as threat development outpaced the speed at which armor and other protection could be fielded to combat troops. With this in mind, the Explosives Survivability Research Center was created at the Idaho National Laboratory (INL). Their tasking was to research, develop, and produce alternative design and production answers to the problems evidenced by the growing IED, advanced ballistics and shaped-charge threat. Development followed a two stage process of laboratory production and field testing. In the summer of 2007, this led to final testing conducted on Aluminum Oxynitride (AlON), an innovative, nearly transparent ceramic alloy. To understand AlON's significance, it is first important to have an understanding of historic research and contemporary solutions.

A. Brief History of Ceramic Armor

Armor development is not a new concept. Researchers have sought to protect soldiers from harm since the dawn of organized war. However, ballistics and explosives survivability, specifically the advancement of sophisticated metallic, metallo-ceramics, and composite armor utilized on cutting edge vehicles and troop body armor is a relatively new and small field. Conventional armor dates to the early days of trench warfare in World War One and the medium

tanks fielded by the British and German Armies.¹ This early heavy steel armor became the foundation for further innovation. In the coming decades the development of increasingly devastating anti-armor weapons such as shaped-charges and kinetic energy penetrators facilitated exploration of unique metallo-ceramic materials. In the 1960s, Great Britain and the United States jointly pioneered a composite armor using a Silicon Carbide (SiC) tiling pattern embedded within a conventional steel plate matrix. This armor design and subsequent ceramic composites were dubbed “Chobham Armor” after the Research Labs at Chobham Common in Surrey, England.

The extreme hardness of ceramics, coupled with the stable, conventional metal lattice frame enabled superior resistance of both shaped-charge explosive jets and kinetic energy penetrators. This concept challenged the established armor paradigm that thick, dense metal plating was needed.² This armor technique was perfected on the American M1 Abrams and British Challenger 2 tanks still in use today. Further application of this technology has been slowly developed through introducing protective back plates for the ceramic, compressing the tiles along the vertical and horizontal axes, and finally coating the tiles with a spray-on appliqué of molten steel or titanium.³

Most recently further experimentation with different ceramic composites has enabled modular armor production. Previous to the 1990s, ceramic armor had been almost exclusively for vehicle applications. However, new research into Boron Carbide and Titanium Boride has permitted lightweight ceramic applications in body armor, and rotor wing aircraft. The Interceptor Body Armor (IBA) currently fielded by the US Military in the War on Terror is a

¹ Phillip Trewitt, *Armoured Fighting Vehicles* (Dempsey, New York, 1999) p. 20-23

² A.L. Chang and B.E. Bodt, “JTCG/AS Interlaboratory Ballistic Test Program – Final Report” (unpublished) *Army Research Laboratory – TR 1577 – December 1977* p. 12

³ H. Chu, K. M. McHugh, T.M. Lillo, “Manufacturing Encapsulated Ceramic Armor System using Spray Forming Technology” *Publications Idaho National Engineering and Environmental Laboratory*, Idaho Falls, 2001

prime example of the protection afforded by these new ceramic applications. The IBA armor system is primarily composed of the Enhanced Small Arms Protective Insert (ESAPI) ceramic plate, surrounded by a Kevlar weave vest. The ESAPI plate is a Boron Carbide tile mounted on a Dyneema backing.⁴ The mechanism relied upon is similar to the vehicle platform armor – albeit without the stabilization of the metal framework. The combination of Ceramic and framework (Dyneema is an application of ultra-high-molecular-weight polyethylene that is 40% stronger than KEVLAR⁵) backing allow for multiple-hit ballistic, blunt force, and explosive protection.

This outgrowth of research beyond heavy vehicle armoring is rapidly becoming the focus of current and future developments in the field. This is especially apparent with the lightweight, highly protective materials such as Boron Carbide and the field of Aluminum Oxides and Nitrides. These materials are not only cost prohibitive, but very sensitive to production variations. As such, the focus of the Explosives Survivability Center, as well as a number of contemporary field studies and research groups was to develop a cost effective and reliable manufacturing process to not only produce laboratory testable samples, but to generate a viable industrial base for future applications as well.

B. Previous Work with AlON

Aluminum Oxynitride is a late comer to the ceramic armor research field. Not synthesized until 1974,⁶ AlON is a unique compound in the Al_2O_3 -ALN system. Following a spearhead effort to map the phase diagram of the Al_2O_3 -AlN system by Ken Jack, at the University of New

⁴ "Hard Body Armor Insert Plates" BAE Systems. 2008.

⁵ *Lightweight Ballistic Composites for Military and Law-Enforcement Applications* ed. By Ashok Bhatnager. (Woodhead Publishing, Cambridge, 2006) p.

⁶ J. Mcauley et. Al., "AlON: A Brief History of its Emergence and Evolution *Journal of European Ceramic Society* Vol 29 p 223

Castle in the early 1970s⁷, the Army Research Lab put together a team to assess AION suitability for advanced materials projects. Under James McCauley they succeeded in reactively sintering AION to near full density.⁸ Following their reaction sintering process, Sakai and Kollemberg were able to reproduce their results using a Hot Isostatic Press (HIP) procedure and a pressureless sintering.^{9,10} Through these three procedures and a number of other duplicates it became apparent that the production of AION was very sensitive to environmental constraints. The equilibrium state was most often achieved with a high degree of micro-pores in the material. This led to a translucent material rather than a wholly transparent plate as was theoretically possible. Because of these inherent design flaws, AION production previously focused on very small quantity material runs in order to combat the defects.

It was not until Wang and Li used a similar HIP process with an AION powder rather than the precursor Al_2O_3 -AlN that fully dense, blemish free

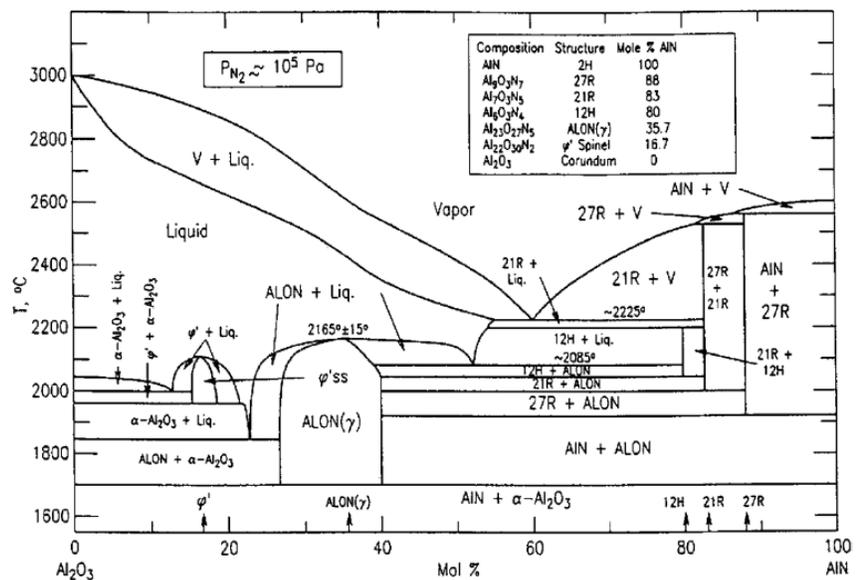


Figure 1 Phase Diagram Plotted by McCauley

⁷ K. Jack and W. Wilson, "Ceramics Based on the Si-Al-O-N and Related Systems" *National Physics Society* Vol 238 1972 p 28

⁸ J. McCauley and N. Corbin "High Temperature Reactions and Microstructures in the Al_2O_3 -AlN System" *Progress in Nitrogen Ceramics* ed. By F. Rley, Martinus Nijhoff, The Netherlands 1983 p 115

⁹ T. Sakai, "Hot Pressed Oxynitrides in the System AlN- Al_2O_3 , Sintering Theory and Practice" *Material Science Monograph* 1981 Vol 14 p 594

¹⁰ E. Kollemberg and E. Ryman Lipinska, "Sintering in Aluminum-Oxynitride (AION)" *Keramische Zeitschrift* Vol 44 Issue 8 1992 p 522

AION was produced.¹¹ However, this process proved to be very cost prohibitive. AION produced in this fashion for high grade optics proved to be around \$12-15 per square centimeter to produce. This process demonstrated the viability of AION production, and allowed further mechanical testing to be conducted, but ruled AION materials out of the question for mass production.

These subsequent high stress mechanical tests further proved the value of AION as an alternative, lightweight armor material. Testing into edge and face impacts, dynamic bar testing (similar to the metal frame casement and ceramic core apparent in Chobham Armor), and elasticity analysis have all proven that AION remains one of the greatest multipurpose ballistic and shockwave materials in current production. Most notable of these tests, in 2005 Strassburger and McCauley demonstrated the dynamic fracture pattern of AION plates when struck at ballistic speeds (~381 meters/second).¹² Its performance validated AION's performance and renewed pursuit of a cost effective means of production. Appendix A contains several high speed photographs and charts pertaining to the edge-on-impact tests conducted.

In tandem with this ballistic research and testing, Gentilman and Macquire have published – and patented new developments in AION production. Their use of Al_2O_3 -AlN powders rather than a premade AION powder has radically reduced the time and cost of AION plate production. Their new approach included the uses of a liquid phase and yttrium and lanthanum additives.¹³ Cutler expanded on this research, finding that a $LiAl_5O_8$ alloy, when introduced during the reaction sintering can be added in small amounts and is capable of eliminating much

¹¹ X. Wang, F. Wang, and W. Li, "Synthesis Microstructures and Properties of γ -Aluminum Oxynitride" *Materials Science Engineering: A* Vol 342.5 p 245

¹² E. Strassburger et. Al. "Visualization of Wave Propagation and Impact Damage in Polycrystalline Transparent Ceramic AION" *Proceedings of the 22nd Intl. Symposium on Ballistics* 2005.

¹³ R. Gentilman, E. macquire, L. DOLhert, Transparent Aluminum Oxynitride and method of manufacture, US patent 4,720,362. 1988

of the porosity.¹⁴ However, both of these methods fall short of current requirements. The addition of yttrium and lanthanum is especially detrimental to pure final products because in any amount beyond ~.01% by weight yttrium or lanthanum may be deposited in the AlON crystals formed, and disrupt the crystalline structure creating a less than ideal structure. The LiAl_5O_8 additive behaves likewise.

Based on these findings, the explosives research group at the Idaho National Laboratory endeavored to find a suitable alternative for this reactive sintering process. The production experiments in 2007 and the subsequent ballistic tests on large scale AlON plates based their methods on Gentilman and Cutler's processes while trying to discover an appropriate additive to recreate full density AlON.

C. Contemporary Fieldwork

Currently, US Military research focuses primarily on three ceramic applications, Boron Carbide, Silicon Carbide, and a variety of Aluminum Oxides and Nitrides. The vast majority of testing and research is targeted towards the current threat environment of the Global War on Terror. Additionally, there are a number of academic and commercial outlets – often tied to a number of defense projects – pursuing novel production techniques for a number of ceramics that are difficult or cost prohibitive to produce.

1. Military Applications

The Air Force Research Laboratory (AFRL) is currently funding a round of testing of AlON based transparent armors as a baseline for new vehicle window, canopy and viewport

¹⁴ R. Cutler, et. al. "Effects of LiAl_5O_8 additions on the sintering and optical transparency of LiAlON" *Journal of the European Ceramic Society* Vol 26 2006 p 1354

applications.¹⁵ By itself, this research was not revolutionary – in essence they recreated at a somewhat larger scale the ballistic testing conducted by Strassburger and McCauley in 2005. The AION plates used were to simulate the size and thickness of contemporary military vehicle windows and viewports – specifically those of an Up-Armored HMMWV “Humvee” that are currently fielded by US and Coalition Forces in Iraq and Afghanistan. What is important is that AFRL AION samples were created using an aqueous colloidal technique pioneered by Kumar and Rajeswari at ARCI in Hyderabad, India.¹⁶

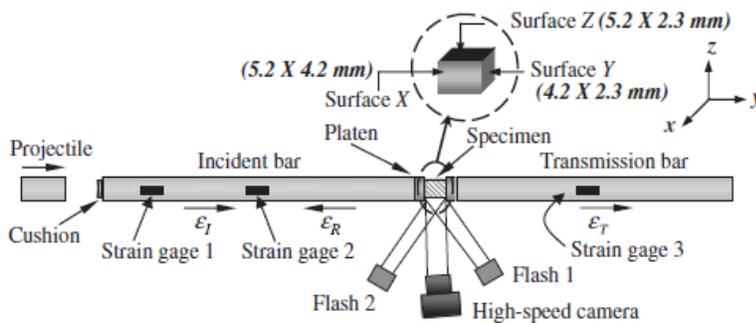


Figure 2 Paliwal, Ramesh, and McCauley's Experimental Design

This ballistic testing is a sister project to the ongoing Compressive Failure Testing being conducted by Paliwal, Ramesh, and McCauley at the Army Research Laboratory (ARL) at Aberdeen Proving

Ground. Through their testing, Paliwal, Ramesh and McCauley are testing the high-strain rate compression that small sample of AION are capable of undergoing. This testing is important to current developments because it demonstrated AION’s capacity to absorb high impact shock and trauma when utilized as a part of a metal framed armor system.¹⁷ The system design essentially models a small volume of AION as a component in a linear armor system. By finding the limits

¹⁵ L. Lundin “Air Force Research Laboratory Tests Transparent Armor” *Advanced Materials & Processes* Vol. 164 issue 11 2006, p45-46

¹⁶ R. Kumar, K. Rajeswari et. al., “Processing of Aluminum Oxynitride through Aqueous Colloidal Forming Techniques” *Journal of the American Ceramic Society* Vol 93 Iss 2 2009 p431

¹⁷ B. paliwal, K. Ramesh, and J. McCauley “Direct Observation of the Dynamic Compressive Failure of a Transparent PolyCrystalline Ceramic (AION)” *Journal of the American Ceramic Society* Vol 89 Iss 7 p 2129 2006

of shock and blunt force impact that the system can sustain, the protective qualities of AION as part of a Chobham-like system can be assessed.

2. Commercial/Academic Pursuits

At the same time as these military applications, a number of corporate and academic institutions are funding advanced research in AION production and applications. AION is not only pursued for its armor properties, but also as an important component in military grade EO/IR systems. AION has the distinction of being one of the few ceramics that is transparent not only to visible light, but mid-wavelength infrared (MWIR) and some bands of RF that are exclusive to military communications. The Surmet Corporation currently holds an exclusive license for Wang and Li's HIP process and has produced plates up to 20x30 inches.¹⁸ This technology demonstration has been put to use not only through windows and viewports, but in shaped optics such as radomes and missile targeting apertures.

Additionally, Miller and Kaplan of Technion, Haifa, Israel are currently studying alternative methods of delivery for lanthanum and yttrium additives in AION production. Their studies are of great importance to the current scope of research insofar as their goals of finding greater maximum levels for additive and dopants in the sintering process.¹⁹ Thus far, their work has followed a similar pattern to that of explosive survivability center at INL but has identified a new additive in Magnesium. The research team will pay close attention to further developments that may have an impact on our studies.

¹⁸ J. Wahl et. al., "Recent Advances in ALON™ Optical Ceramic" (Unpublished White Paper) Surmet Precision Optics, Murrietta, CA 2010

¹⁹ L. Miller, and W. Kaplan "Solubility Limits of La and Y in Aluminum Oxynitride (AION) at 1870°C" (unpublished report) Technion, Haifa, Israel, 2008

2. METHODS

AION is a crystalline material that can be produced in a transparent form. Recent interest is driven by the potential for AION to replace aging armor solutions – especially in the realm of ballistic glasses in vehicles, facilities and aircraft. As referenced above, AION production is very cost prohibitive. This is because the primary means of manufacture is through hot pressing, which is necessary for the creation of AION plates that are free of micro-pores. The goal of this research was to produce AION at a reduced cost by utilizing a liquid phase sintering process and removing the need for pressure assisted densification. These alternatively produced AION test plates would then undergo ballistic and optical testing to demonstrate their effectiveness.

A. Al₂O₃-ALN System

An alternative to using pre-fabricated AION powders by reaction sintering Al₂O₃ and AlN was evidenced by McCauley and Corbin²⁰. Due to its cost-effectiveness this process was chosen as a baseline, however it is important to note that while this method is effective at forming γ -AION, its polycrystalline structure has an intragranular porosity that is difficult to remove.²¹ A liquid phase sinter process, introduced to the Al₂O₃-AlN structure could reduce, if not completely eliminate this porosity. It is important to note that the liquid phase must be transient, as any remaining liquid during the final reaction would impair transmission. The process developed by Hartnett, Gentilman and Macquire was studied because of the unique characteristics yttrium and lanthanum have when added during the sintering process. However, as evidenced by their trial and error, only small amounts could be introduced to the samples before secondary phases developed and yttrium was deposited in the AION, reducing transparency significantly. Pursuant

²⁰ J. McCauley and N. Corbin "High Temperature Reactions and Microstructures in the Al₂O₃-AlN System" *Progress in Nitrogen Ceramics* ed. By F. Rley, Martinus Nijhoff, The Netherlands 1983

²¹ N Corbin, "Aluminum Oxynitride Spinel: A Review" *Journal of the European Ceramic Society* Vol 5 (1989) p 145

to the transparency requirement, AlPO_4 was also selected because of the formation of a liquid phase at $\sim 1850^\circ\text{C}$. At this temperature AlPO_4 decomposes to P_2O_5 gas and Al_2O_3 , allowing the Al_2O_3 to react with the parent structure and form AlON. It should be possible then to add any quantity of AlPO_4 without fear of formation of second phases.

B. Experimental Procedure

Two types of Al_2O_3 powder were used throughout the experiments – Ceralox SPA-.05 and Alcoa calcined alumina. This was to observe any reaction changes and introduce varying grades of purity into each batch. Tokuyama AlN grade E powder was employed in every experiment. The Al_2O_3 and AlN were batched according to desired AlN mol percentage (mol%) relative to the Al_2O_3 . AlPO_4 , produced by Acros Organics, was added to each batch of the Al_2O_3 -AlN mixture according to a desired weight percentage (wt%) ranging from .5-20%. The three compounds were dispersed in ethanol and mixed in a shearing mixer for a minimum of ten minutes at 6000rpm. The subsequent mixture was poured into a drying tray for 24 hours.

Following drying, the caked powder was removed from the tray and ground in an alumina pestle and mortar and sieved through a 100 mesh. The powder was divided in 5 gram samples. These were pressed into small plates with a ram pressure of $\sim 10,000$ lbs/in. At this point, a number of samples were lost to cracking so were reground and sieved. Surviving plates were Cold Isostatic Pressed (CIP) at 60ksi. Following CIPing, the samples were baked in air for 15 hours at 600°C in order to remove any binding agent or organic contaminants.

The firing process was accomplished in an Astro Industries 1000A furnace using Boron Nitride (BN) crucibles. It is important to note that the furnace used has carbon heating elements.

As Carbon can be detrimental to AlON formation,²² a protective powder was formulated to coat the plates prior to firing. This included Al₂O₃ powder AlN, and occasionally BN or yttria. These protective powders were poured into the crucible before the sample plates and then again after them to create a shielding layer from the carbon heating elements.

Following the firing and subsequent overnight cooling, samples were removed from the protective powder coat. This had to be carefully cracked off of the plates because it had a tendency to bake onto the samples and form a soft crust around the core AlON material. The sample plates then underwent a set of tests to measure density and compound presence (Al₂O₃-AlN reaction, AlPO₄ decomposition, and any contamination). Density was important for further ballistic testing – only samples with the appropriate properties would be produced at higher volume for assessment as armor candidates. Density measurements were taken using the Archimedes method in water. To assess the purity, a Bruker D8Advance X-Ray Diffraction (XRD) system was used to ascertain compounds present in the sample. Finally, samples were polished and etched for both optical and scanning electron microscopy (a Keyence DHX digital microscope was used for optical, and a Phillips XL 300 ESEM was used for SEM) to assess composition.

C. Experimental Series

Through the course of research five series of experiments were conducting utilizing various mixes of the Al₂O₃-AlN powder mix as well as different compositions of the protective settling powder. The author was present for the first three series and the subsequent ballistic testing of series two and three. For continuity purposes, and at the kind permission of Dr. Henry

²² N. Corbin, "The Influence of Carbon, Nitrogen, and Argon on Aluminum Oxynitride Spinel Formation" Masters Thesis, Department of Materials Science and Engineering, MIT, Cambridge, MA (1982)

Chu and Dr. Michael Bakas, under whose direction the research was carried out, a short summary of series four and five and their purposes is included in this paper.

The first series of experiments conducted determined the viability of AlPO_4 as an

additive for reaction sintering.

Samples fired with small amounts of the additive (.5 wt% to 1.0 wt%) had no traceable amount of

decomposed phosphates in the final AlON samples. Figure 3 shows an example of the XRD analysis of series one samples. The XRD

indicated that primarily γ - AlON was present, but an unexpected

amount of AlN was unreactive and

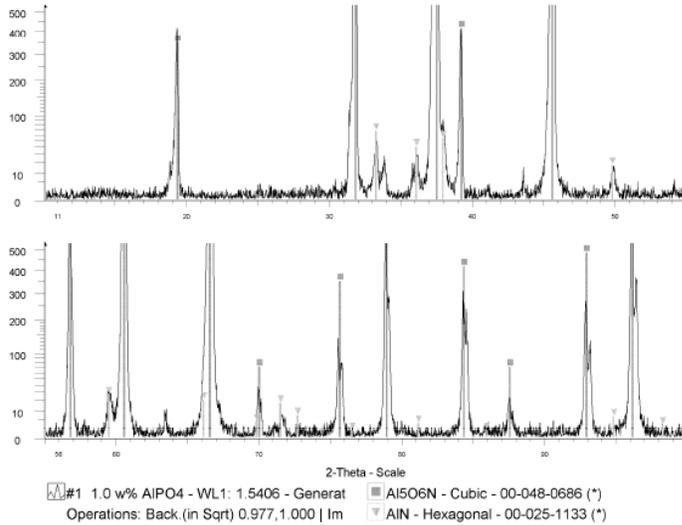


Figure 3 XRD of series 1 sample batched at 35.0 mol% AlN with 1.0 wt% AlPO_4 added

so hindered the final sample's transparency and density. During this series a problem was observed with the settling powder. When the AlPO_4 was added at a much higher amount (≥ 10.0 wt%) the settling powder used in firing had a tendency to surface bond to the sample plates.

The second series built upon the results of the first series and investigated additional interaction between the AlPO_4 additive and the settling powders. Most important to note from these series is that over the course of firing, the AlPO_4 facilitated material interaction between the settling powder and the Al_2O_3 - AlN sample. This shifted the residual mol% of the AlN in the samples beyond acceptable values and greatly decreased the transparency. The areas of the sample plates where most interaction occurred had a curious optical degradation beyond what

was expected. The AION formed had a much lower AlN mol% than desired in most cases – far beyond levels that transparency is possible. This transition area was speculatively dubbed ϕ' phase AION and will be the study of further exploration.

The third series of experiments focused on eliminating the secondary ϕ' phase, thus improving transparency and optical appearance. The interaction between the settling powder and the AlPO_4 was apparently driving the AlN out of the γ -AION region in the sample plates. This unreacted AlN allowed some of the liquid transient phase to remain. Rather than decomposing in the firing, the AlN-liquid interaction created the ϕ' phase on cooling. It was surmised that increasing the AlN mol% would compensate for this interaction. The third series samples subsequently contained 15.0 wt% AlPO_4 and increasing amounts of AlN in both the sample plates and the settling powder.

Series four and five shifted concentration to addition conventional additive yttrium (in the form of Y_2O_3 to the settling powder). The maximum density achieved during series three had been using 44.0 mol% AlN – very near to the traditional method Wang and Li pioneered. However, transparency was still not achieved. Series four explored the addition of Y_2O_3 in the powder and the optical properties of the samples that were produced. Unfortunately, while smaller in size, the ϕ' phase AION was still present, and often tinted a yellow color. Elimination of these phases remains the goal of the current (fifth) series of experiments in order to produce wholly transparent AION through this reaction process. **

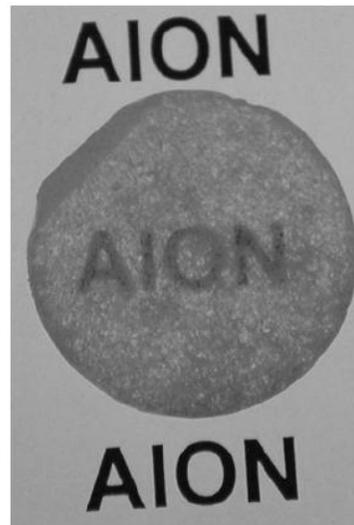


Figure 4 44.0mol% AlN sample from series 3, with ϕ' phase on plate boundaries

D. Ballistic Testing

The AION samples created during the second and third series were dense enough to warrant ballistic and explosive testing in comparison with contemporary “bullet proof” armors and materials. Ballistic testing was conducted by the author and advisors on the INL explosives test range. The explosive testing materials and methods were classified due to their sensitive nature and the specific threats to current American military interests. As such, explosive material loads, computer models, and associated testing equipment will not be discussed in this paper.



Figure 5 the Author conducting ballistic testing.

The ballistic testing was done using an Armalite AR-50 .50 caliber sniper rifle. Each sample was a 5x5 inch tile of AION. Control samples of body armor grade Boron Carbide and conventional bullet proof glass were utilized for comparison. Samples were mounted on a hydraulic platform to lift them to an appropriate

target height. Each sample was fired on from 25 meters using Hornady Armor Piercing cartridges. The bullet weight for each test was 750 grains, with an additional 285-287 grains of propellant per casing.

An additional test performed earlier was used as a benchmark comparison to AION produced during this research. Using a six inch thick plate of ALON™ produced by the Surmet Corporation a similar .50 caliber rifle ballistic test was conducted. For comparison, three separate laminate glass plates of the same dimensions were used. This test served to compare the AION produced by the research group with the commercially available, more expensive ALON™.

3. RESULTS/DISCUSSION

The first series of experiments carried out demonstrated that AlPO_4 is a viable alternative to yttrium and lanthanum additives during reaction sintering. However, the important discovery that the AlPO_4 liquid phase used could have serious detrimental effects on the sample's composition necessitated the further series to refine the amount of AlPO_4 that is usable and to quantify the interaction between it and the settling powder. The composition of AION produced during this test should have been within acceptable limits in the single phase AION region of the phase diagram devised by McCauley (see figure 1). However, the samples fired with high wt% of AlPO_4 demonstrated that this was not the case. Based on the AlPO_4 -settling powder interactions, it was imagined that interaction was more significant than first suspected. This was especially clear in later tests that had significantly lowered densities.

The second set of experiments explored this AlPO_4 -settling powder relationship and sought to find a beneficial result. What was discovered was that the AlPO_4 interaction can introduce additives from the settling powder to the sample plate. With a greater the amount of AlPO_4 that present, a corresponding greater the interaction and degree of introduced material was observed. Figure 5 shows a 500x optical micrograph of AION produced during the second series containing both the primary γ -AION and secondary ϕ' phase AION. It is important to note that this secondary phase was detrimental to overall density more so than to transparency. Microscopy confirmed the secondary phase on individual crystal grain boundaries. These secondary phases contributed to less stability in the crystal structure and a translucency when viewed at a macroscopic scale. However, further minimization of this phase would result in a near perfect sample.

The final series of experiments focus on improving appearance had an interesting development. By increasing the AlN mol% in both the sample and settling powder, it was

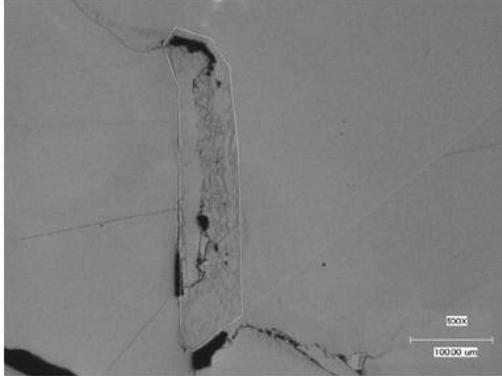


Figure 6 Optical Micrograph of 44.0 mol% AlN sample. ϕ' phase is faintly outlined

possible to minimize the ϕ' phase AlON, but with a sharp decrease the density. This trend was apparent through the XRD as well. As AlN mol% increased, the peaks associated with the ϕ' phase became less and less prominent. At 44.0 mol% and above, samples were nearly pure. As AlN mol% increased above this threshold, the density fell sharply.

The exact workings that control the interaction of AlPO₄ with the settling powder are currently unidentified. The prevailing opinion is that the AlPO₄ allows some amount of the surrounding powder to infiltrate the sample plate. However, this seems to have much longer lasting effects than expected – AlPO₄ is expected to decompose at 1750°C while firing of the

samples ranged from 1850°C-1950°C. Supplementary research into phosphate decomposition indicated that significant porosity should have been evident in the sample. The fact that it was not evident in any

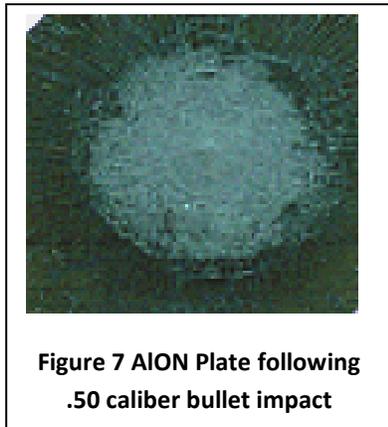
The exact workings that control the interaction

Sample AlN mol%	Density (g/cc)	AlON Phases Present
40.0%	3.632	ϕ' and γ
41.0%	3.619	ϕ' and γ
44.0%	3.637	ϕ' and γ
44.25%	3.545	γ
46.0%	3.563	γ

Table 1 Sample Density of Selected Series 3 Sample plates

sample with elevated AlPO_4 additive is indicative of settling powder infiltration. Further exploration of the settling powder, AlPO_4 and yttria (in the case of series four and five) interactions must be conducted for specific conclusions to be made.

The ballistic testing was very promising with every sample tested. The AION test plates arrested the .50 caliber armor piercing rounds with much the same fractal pattern observed by Strassburger and McCauley in 2005 and the AFRL tests in 2006. They demonstrated that AION



performs much same against small and medium caliber weapons as conventional laminate bulletproof glass that is more than twice its thickness. This testing established that AION of less than ideal density (3.637 g/cc versus the expected 3.696 g/cc) and transparency can perform at much the same level of ballistic protection as the more rigorously produced optically transparent AION. For comparison, the team referenced ballistic tests

conducted by Strassburger and McCauley and the AFRL that incorporated the same metrics and calibers and found our results to be very similar. Graphics and metrics of previous test can be found in Appendix B.

4. CONCLUSIONS

AlPO_4 is a viable alternative additive for reaction sintering of AION from Al_2O_3 -AlN precursors. However, sufficient addition of AlPO_4 can cause an adverse reaction with settling powders and environmental surroundings. While the exact nature of this method of interaction remains unidentified, it is possible that the settling powders infiltrate the sample as the AlPO_4 decomposes during the firing process. However, under positive conditions this interaction does not have adverse effects on the density and ballistic properties of the material. Further refinement of this process will lead to full transparent AION that is a viable alternative to current production capabilities.

**Author's note: Current testing with series five procedures has lead to a patented AION production process, US Patent 7833922.

5. ACKNOWLEDGMENTS

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The submitted manuscript has been authored for the express purpose of documenting research in the field of contemporary military grade ceramic armors. Any reference contained to specific commercial products, processes, by trade name, trademark or manufacturer does not constitute endorsement. The views and opinions expressed herein are those of the author and do not constitute official views of Brigham Young University, Idaho National Laboratory, or the US Department of Energy.

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APPENDIX A: List of Figures from Previous Stress Tests

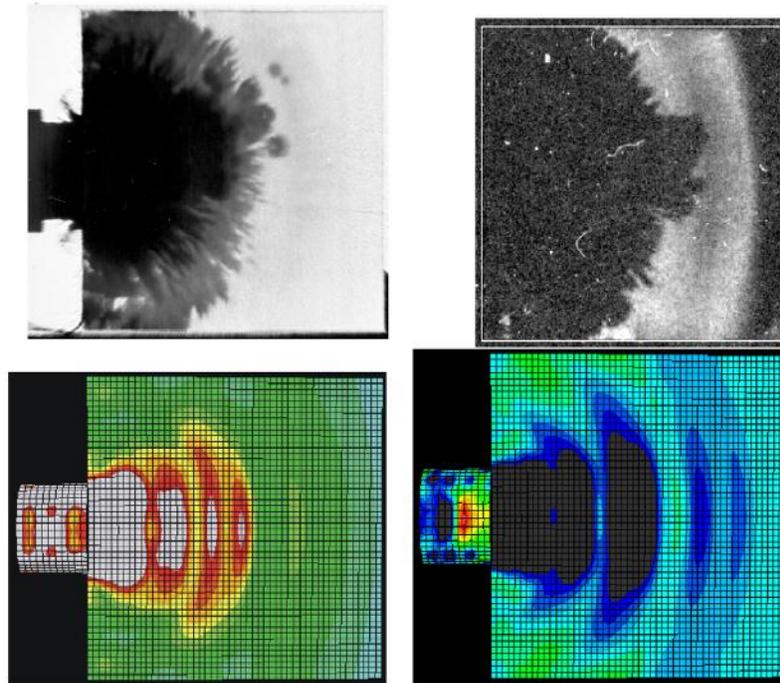


Figure 8 Comparison of high speed photographs with Finite Element Analysis (FEA) simulation from Strassburger and McCauley. The top two images are high speed EoI images taken approximately $8.7 \mu\text{s}$ after impact. The bottom images are the FEA simulation of compression and shearing stress (right and left images respectively) expected. Note the similarity of the actual damage and stress patterns to the expected models. (From Strassburger and McCauley)

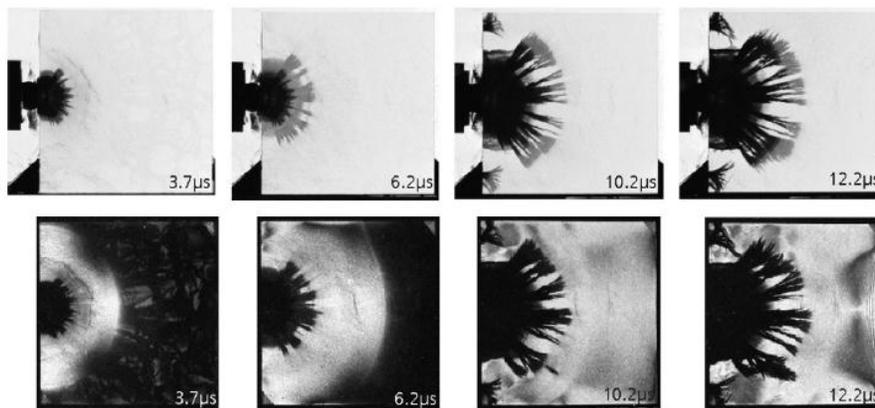


Figure 9 Edge-on impact photos in plane and crossed polarized light. Steel sphere impactor at 429 m/s. top series in plane, bottom series crossed polarized light (From Strassburger and

APPENDIX B: Ballistic Testing Graphics

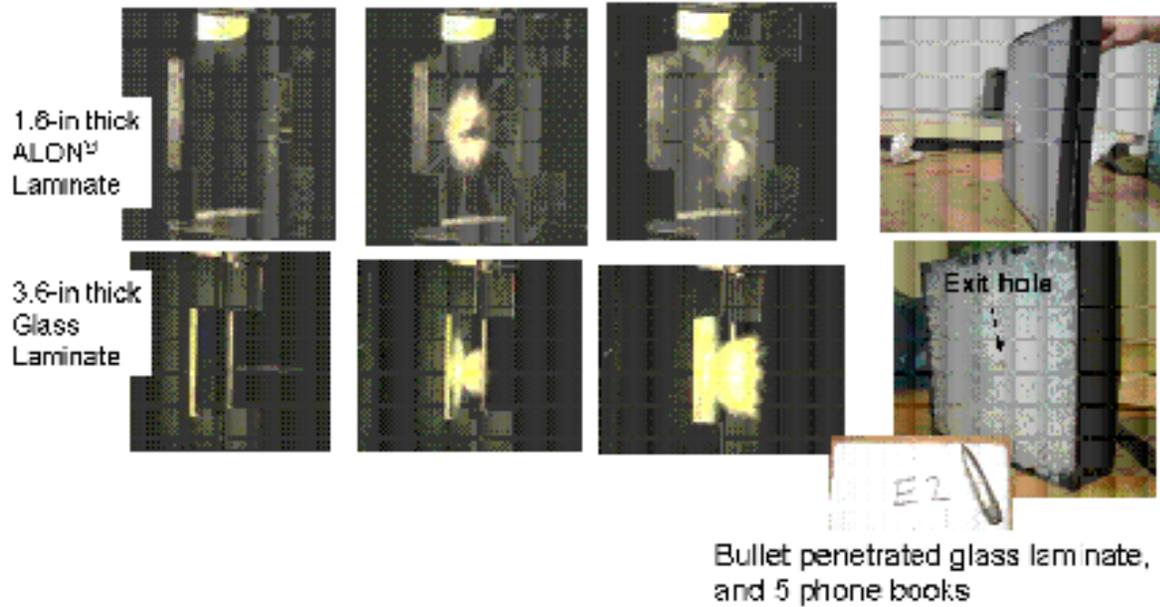


Figure 11 Experimental set up of Strassburger and McCauley. Special note should be taken that 1 6-inch plate of ALON was capable of stopping .50-cal API that 3 6-inch plates of laminate glass and 5 phone books were unable to



Figure 12 AFRL Ballistic Testing set up. This test was conducted with ballistic ALON manufactured according to the licensed process owned by the Surmet Corporation

APPENDIX C: Selected X-Ray Diffraction Charts

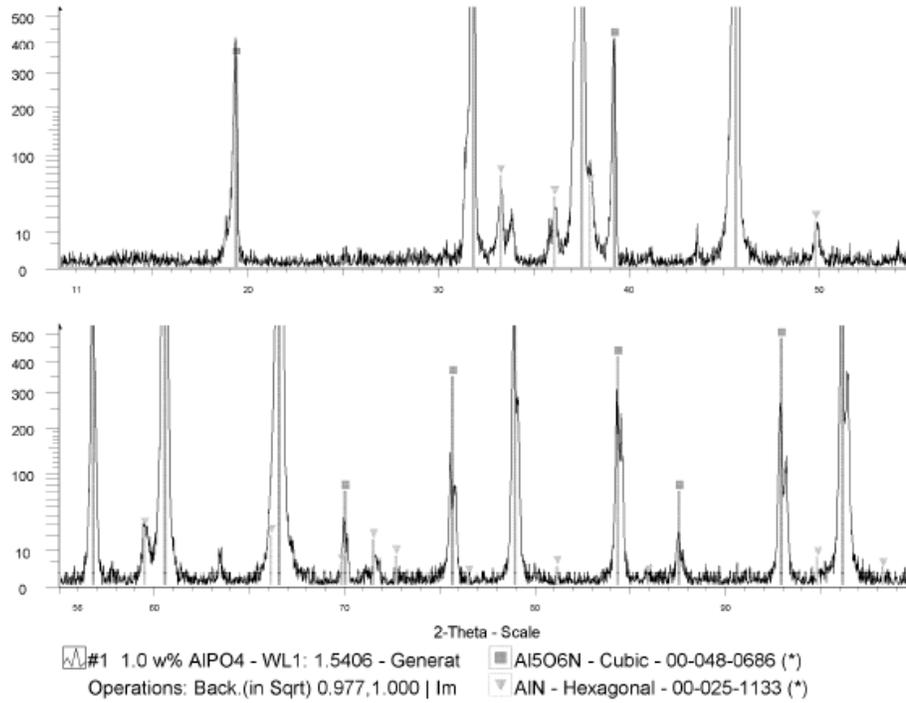


Figure 23 XRD of sample batched at 35.0 mol% AlN with 1.0 wt% AlPO₄ added, fired at 1950°C

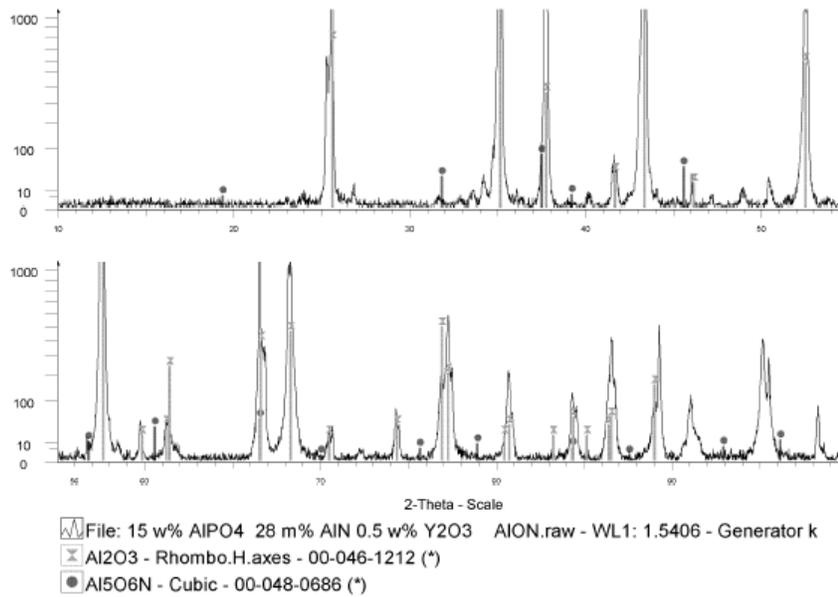


Figure 34 XRD of sample batched at 28.0 mol% AlN with 15.0 wt% AlPO₄ added, fired at 1950°C with a 28.0 mol % AlN settling powder

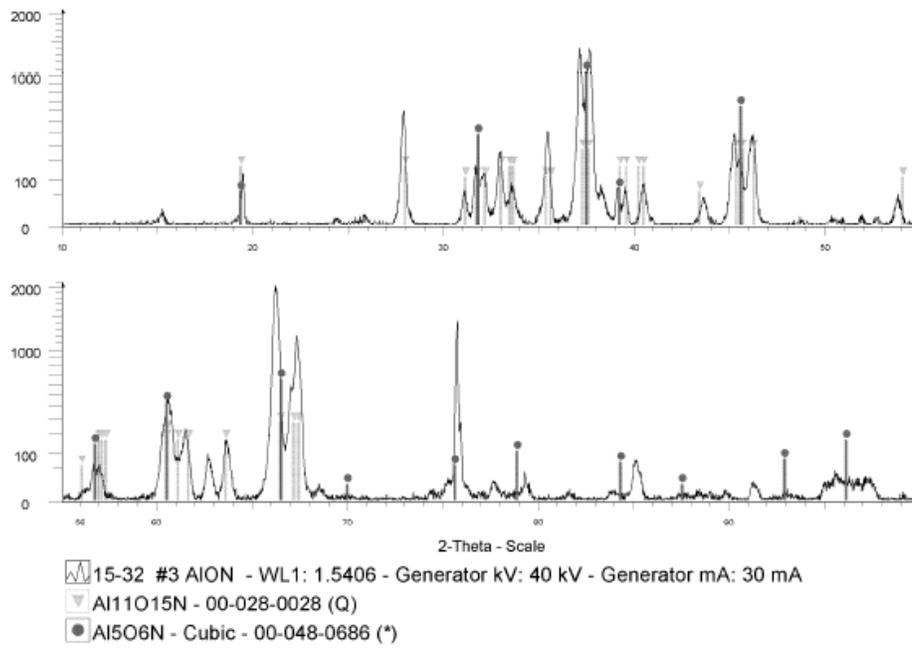


Figure 45 XRD of sample batched at 32.0 mol% AlN with 15.0 wt% AlPO₄ added, fired at 1950°C with a 28.0 mol % AlN settling powder

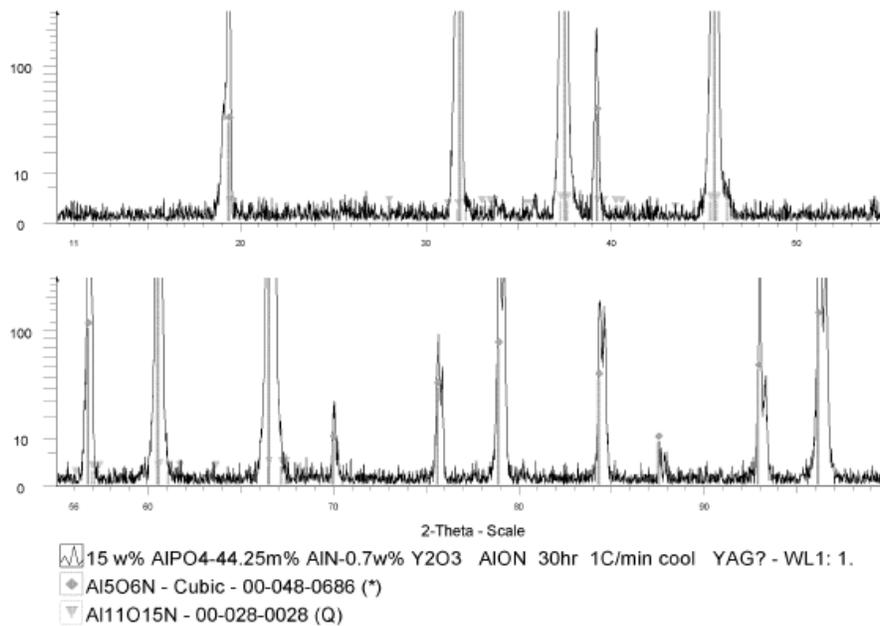


Figure 56 XRD of sample batched at 44.25 mol% AlN, fired at 1950°C with a 28.0 mol % AlN settling powder