

INTRINSICALLY TAMPER INDICATING CERAMIC SEAL (ITICS)

Juan A. Romero, Heidi A. Smartt, Charles A. Walker, Barry D. Schoeneman, David L. Zamora, Thomas M. Weber

Sandia National Laboratories, Albuquerque, NM USA

Daniel Krementz, George Weeks, Kyle S. Brinkman, Adrian Mendez-Torres
Savannah River National Laboratory, Aiken, SC USA

ABSTRACT

Sandia National Laboratories and the Savannah River National Laboratory are collaborating on an effort to research and develop the technologies required to build and test a tamper indicating device (TID) made of ceramic materials whose intrinsically unique surface structure provides the ability to uniquely identify the device. Various coatings are being investigated to improve tamper resistance. The proposed seal will also include the ability to securely identify the seal and to verify its integrity in-situ using electronic and cryptographic methods. A hand held verifier is under development, which will provide for in-situ verification. This seal is intended to replace the ubiquitous metal cup seal used by both domestic and international security organizations for loop and wire Safeguards and Security applications. The need to replace the current seal has been articulated by both domestic and international security experts.

INTRODUCTION

Seals are used in nuclear verification regimes to determine that material is neither introduced nor removed from a tamper-indicating container or that unattended monitoring equipment is not tampered with. Additionally, seals provide a unique identity (a tag) for the sealed container or item. Seal criteria include reliability, tamper-indication, in-situ verification to reduce inspection effort, ease of evaluation of results, ease of conclusiveness, and ease of use [1].

The metal seal (Figure 1) is the most common seal used by the International Atomic Energy Agency (IAEA) today. It is a single use, metal wire loop seal that must be verified at the IAEA. A unique ID is obtained by imaging random scratches on the inside surface of the metal cap and comparing the images before installation and after removal.

The need to replace metal seals, used both by the IAEA and other security organizations, has been articulated and this project researches advanced capabilities to this end (Figure 2). Some of these capability improvements include in-situ verification; improvements for ease of use by incorporating wire-cutting features into the seal or application tool; multiple levels of tamper indication via a frangible seal body, surface coatings, and low power electronic tamper indication; unique identification through active means (electronics, verified in-situ through a hand held reader) and passive means (non-reproducible surface features).

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Figure 1: Size comparison of a metal seal (left), rapid-prototype mockup of the ITICS (right), and a U.S. quarter (bottom).

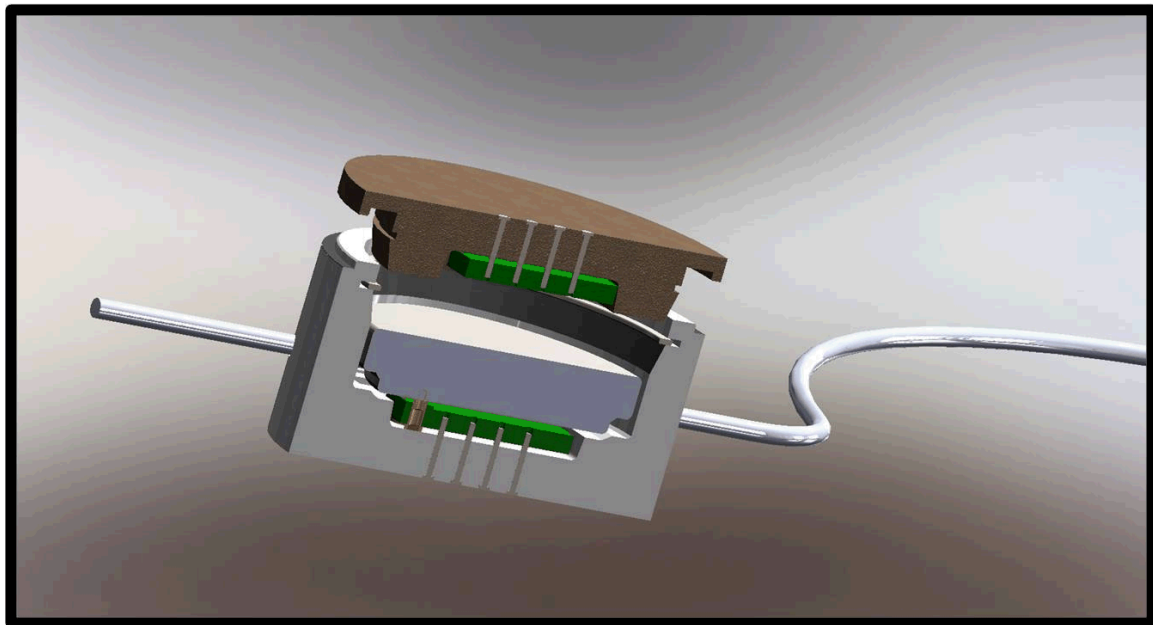


Figure 2: Cross section of the Intrinsically Tamper Indicating Ceramic Seal (ITICS).

CERAMIC/SEAL BODY

The ceramic seal body is designed to have two components prior to application. To apply the seal, both ends of the seal wire are threaded through the bottom component. The top component is then pressed against the bottom component to capture the wire and establish continuity to the electronics. Seal closure and retention are achieved by a mechanical snap ring and an adhesive that is activated during closure. A seal application tool is under development that will ensure radial alignment of the seal electronics, apply

even clamping force to the seal mating surfaces to facilitate adhesive activation, and cut the seal wire. Seal wire cutting using cutting edges in the seal itself is also being considered. Seal body development has been an iterative collaboration between SRNL and SNL. The effort has included collaboration on design details concerning seal closure, clearances for the adhesive, electrical wiring options, seal wire capture, and seal wire cutting approaches. After the electronics have been incorporated into the seal and an initial vulnerability review has been performed, a second iteration of the seal body design will be performed.

SRNL researched ceramic materials for the seal body and selected alumina, zirconia and Macor™ to provide a range of desired properties. Properties considered included tensile strength, frangibility, manufacturability and availability. Seal body test pieces of the three materials have been procured to support electronics integration, coatings research and seal mechanical properties. If all three materials prove to have acceptable performance as a seal body material, alumina is the desired material based on its compatibility with brazing (see brazing discussion below).

BRAZING [2]

In order to implement a self-contained wire cutting capability in the seal itself, a metal cutting edge will be brazed on the ceramic. The distinction to note is the difference between brazing and soldering. Brazing is a joining process whereby a filler metal is heated above 450°C and distributed between two or more close-fitting parts by capillary action. Soldering occurs at temperatures below 450°C. Materials being brazed do not melt which occurs in a welding process. The feasibility of using furnace brazing processes for tungsten carbide cutting edges will depend on the ceramic materials selection. SRNL initially chose alumina, zirconia, and Macor™ as candidate ceramic materials. SNL fabricated brazed test samples for strength testing and wire cutting force studies. Strength is dependent on the difference in thermal expansion coefficients between tungsten carbide and the ceramic material chosen.

From the initial strength results, alumina (97Ag-1Cu-2Zr/WC) performs best, although all systems exhibit signs of stress at the ceramic/tungsten carbide braze interface (Figure 3, Figure 4, Figure 5). Macor™ substrates are shown to be too weak to accommodate the interface stresses resulting in delamination along the entire joint length. Zirconia substrates exhibit higher stresses than alumina substrates.

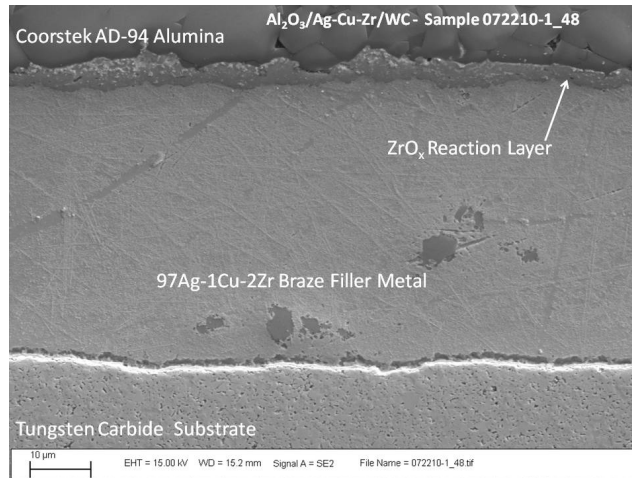


Figure 3: Alumina-tungsten carbide braze section.

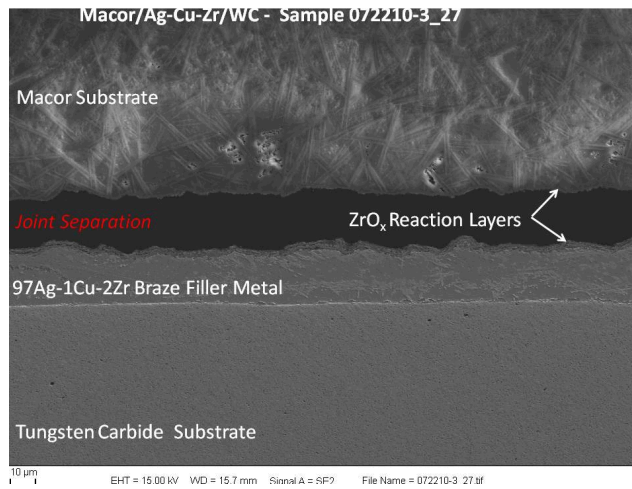


Figure 4: Macor™-tungsten carbide braze section.

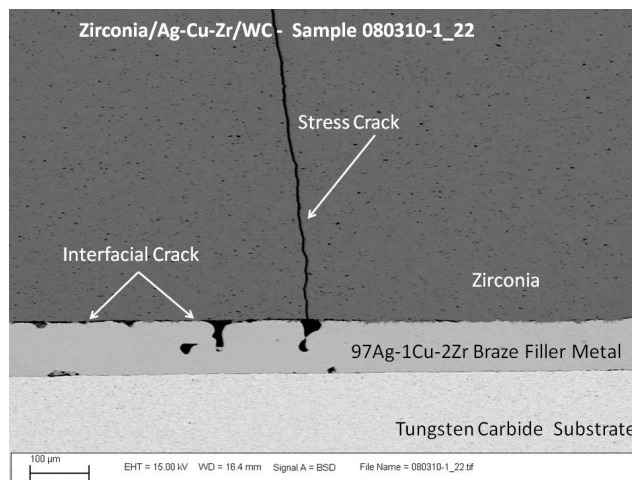


Figure 5: Zirconia-tungsten carbide braze section.

The next step in the brazing research is to fabricate test samples for shear testing, fabricate test samples and fixturing to determine required wire cutting force, and to look more closely at coefficient of thermal expansion (CTE) matched carbide materials. Based on these tests, SNL will optimize the brazing processes and joint design.

ADHESIVES

The adhesively bonded faces (Figure 6) have the function of providing a secondary method of preventing intrusion, as well as providing a barrier to the external environmental or liquids. Adhesives must be identified which exhibit the following characteristics: ability to bond with seal materials, provide robust packaging or delivery scheme within the seal, attain adequate bond strength with no “in field” surface preparation, bond only at the time of installation, possess long shelf life, and maintain bond strength in a broad range of ambient conditions.

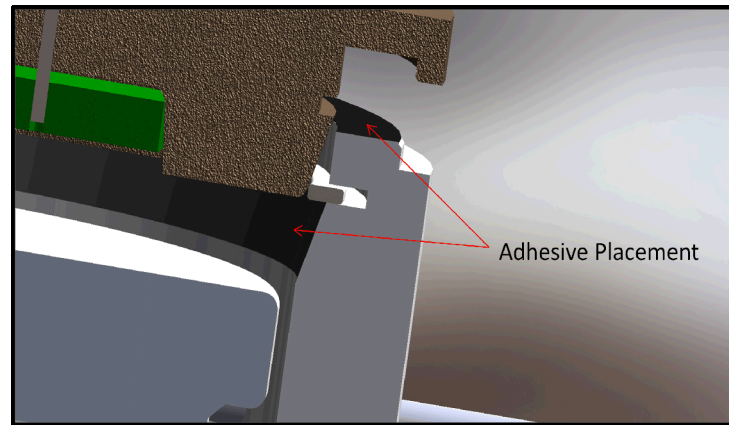


Figure 6: Adhesive seal placement.

Three adhesive systems have been tested thus far – (1) 3M VHB, an acrylic based pressure sensitive adhesive (PSA) serving as a baseline, (2) Avery Dennison (AD) S8755 PSA unfilled, and (3) AD S8756 PSA unfilled, 0.25 micron $\text{Al}(\text{OH})_3$. All three PSA films were 0.002 inch thick and used to bond two alumina disks together for tensile strength testing. These are off-the-shelf materials and are not considered structural films. All alumina samples were pencil blasted, acetone rinsed, ultrasonic rinsed in isopropyl alcohol, and blow-dried using dry nitrogen. To obtain optimal bonding using the 3M VHB, samples were clamped using 30 lbf followed by a 2 hour cure at 150°F. The 30 pound force was chosen based on an initial estimate for the cutting force required to trim the seal wire. The 2 AD films were processed at room temperature after being bonded with ~30 lbf which was reduced to a lower constant force in the bonding fixtures overnight. This was done to simulate actual clamping conditions the seal would see in service. All samples were tested at room temperature with a pull rate of 0.05 in/min. Table 1 summarizes the pull test results.

Table 1: Adhesive Bond Strength Results.

Material	Average Stress To Failure (psi)	Std. Dev. (psi)
3M VHB	81.41	42.72
AD S8755	65.06	22.37
AD S8756	44.67	11.02

The most notable aspect of the results is the large standard deviation within the groups. These materials rely on applied force to form the bond between materials. Using a low clamping force produces non-uniform bonding. Based on results for required wire cutting force, an applied force of approximately 100 lbf will be required. This force is a more realistic bonding force which will reduce the standard deviation in bond strength. As none of the three previously tested adhesives performed as desired, another adhesive system is currently being developed and will be tested soon.

ELECTRONICS

The seal body contains two microcontroller units (MCU) with protected memory authenticating each other using AES-128 secret key cryptography. The keys are actively managed in SRAM to mitigate persistence. Randomly timed messages are passed between MCU's for verification of seal integrity. If the MCU's are separated, lose power, or receive a low battery voltage warning during deployment, the secret keys are destroyed and replaced with a default set of keys. Future versions will destroy the keys upon seal body tamper as well.

A hand held seal reader securely identifies the seal in-situ and verifies its integrity. Messages provide information regarding the status of internal components. The MCU's log interrogations using a relative time stamp.

A fully functioning desktop version has been built and is being tested. The actual size electronic boards (Figure 7) are being procured to install in the prototype seal bodies.



Figure 7: Battery and MCU board size relative to US quarter .

The next steps are to integrate the actual populated boards and connection pins into an initial prototype ceramic body, validate functionality of the electronics package and communication, and modify the electronics based on initial findings.

COATINGS

Research is being performed on conductive and fluorescent coatings and dopants that can be used as tamper indicators. Conductive coatings Indium Tin Oxide (ITO), IrO_2 , and RuO_2 have been applied to samples of Macor™, alumina, and zirconia by thin film deposition. Initial results indicate that the sheet resistance of ITO is sensitive to modifications to the surface that may be indicative of a tamper attempt. One of the desirable features of ITO is its transparency, which makes it compatible with Laser Surface Authentication (LSA). LSA maps the surface roughness of a material, thus providing a unique identifier. An initial study using Laser Surface Authentication (LSA) to uniquely identify test billets made of zirconia, alumina, and Macor™ was successfully conducted. This study shows these materials are compatible with LSA. The study also shows that the surface needs to be protected with a transparent coating (ITO, for example) to prevent duplication of the surface structure.

Fluorescent coating research is being performed in support of synthesis and development of fluorescent seal coatings. To incorporate fluorescent species on or into the surface of ceramic specimens, fluorescent functional coatings were synthesized by chemical solution deposition methods based on silica gel (tetra ethyl orthosilica TEOS). The R&D work on this task has focused on the methodology to prepare coatings with fluorescent centers. Two ionic dopants (Er^{+3} and Cr^{+3}) have been identified. Er-TEOS sol-gels have been successfully synthesized. Analysis of the Er sol-gels has revealed discreet visible emission peaks for given specific ultraviolet and visible excitation wavelengths. Techniques to deposit the sol-gels on ceramics are currently being developed. Once deposition techniques of the fluorescent coatings are determined, research will begin on application of multi-layer conductive and fluorescent coatings. The fluorescent coating will be applied to the ceramic, followed by the conductive coating and the multi-layer coatings will be characterized to ensure that the properties of the coatings do not interfere with each other. Studies to confirm compatibility with LSA will also be performed.

NEXT STEPS

After the individual tasks are completed, an initial prototype seal will be fabricated by the end of FY11. This prototype seal will be submitted for initial vulnerability assessment and results will be used to guide the next iteration in the development of the prototype seal.

ACKNOWLEDGEMENT

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REFERENCES

1. B. Richter, "Containment and Surveillance – Status and Perspectives," ESARDA Course on Nuclear Safeguards and Non-Proliferation, 2008.
2. C.A. Walker and V.C. Hodges, "Comparing Metal-Ceramic Brazing Methods," *Brazing Journal*, 87 (10), 2008.