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Development of a Ceramic Tamper Indicating Seal: SRNL Contributions

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Abstract

Savannah River National Laboratory (SRNL) and Sandia National Laboratories (SNL) are collaborating on development of a Ceramic Seal, also sometimes designated the Intrinsically Tamper Indicating Ceramic Seal (ITICS), which is a tamper indicating seal for international safeguards applications. The Ceramic Seal is designed to be a replacement for metal loop seals that are currently used by the IAEA and other safeguards organizations. The Ceramic Seal has numerous features that enhance the security of the seal, including a frangible ceramic body, protective and tamper indicating coatings, an intrinsic unique identifier using Laser Surface Authentication, electronics incorporated into the seal that provide cryptographic seal authentication, and user-friendly seal wire capture. A second generation prototype of the seal is currently under development whose seal body is of Low Temperature Co-fired Ceramic (LTCC) construction. SRNL has developed the mechanical design of the seal in an iterative process incorporating comments from the SNL vulnerability review team. SRNL is developing fluorescent tamper indicating coatings, with recent development focusing on optimizing the durability of the coatings and working with a vendor to develop a method to apply coatings on a 3-D surface. SRNL performed a study on the effects of radiation on the electronics of the seal and possible radiation shielding techniques to minimize the effects. SRNL is also investigating implementation of Laser Surface Authentication (LSA) as a means of unique identification of each seal and the effects of the surface coatings on the LSA signature.

Introduction

Tamper indicating seals are used in nuclear verification regimes to verify that material is not diverted from a container or that unattended monitoring equipment is not tampered with. A seal must be reliable, cost-effective, easy to use, and must provide both tamper indication and unique identification (ID). In-situ verification of seals is also a desirable feature.

Metal loop seals are commonly used by the International Atomic Energy Agency (IAEA) and other security organizations. Metal seals are single use seals whose unique ID is made by a labor intensive process of imaging a random scratch and solder pattern on the inside surface of the seal. The unique ID images must be compared before seal installation and after removal.

The Ceramic Seal presents a new concept in low cost seal design. This seal brings together a number of technologies enabling a level of security that has previously been unavailable for applications where metal loop seals are currently used. Following are some of the significant improvements over previous passive seals:

- Frangible ceramic seal body
- Functional coatings to identify drilling and cutting attacks
- Intrinsic unique identifier using Laser Surface Authentication
- Electronics internal to the seal that provide in-situ cryptographic seal authentication
- User-friendly seal wire capture[1]

An alumina ceramic base material for the seal body was selected to present a strong but brittle/frangible structure that will break rather than open after installation. Alumina ceramics are difficult to repair without leaving some telltale indication.

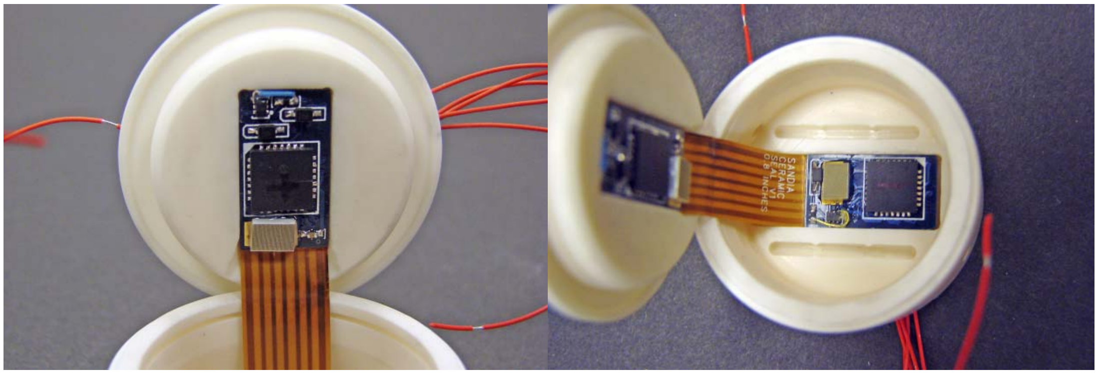

Figure 1: Generation I Ceramic Seal Assembly and Electronics (photos provided by SNL)

SRNL is performing this research and development in collaboration with Sandia National Laboratories (SNL). Figure 1 shows the Generation I Ceramic Seal with electronics. The alumina body was developed by SRNL and the electronics were developed by SNL. The Generation II Ceramic Seal is currently being developed that incorporates feedback from the SNL Vulnerability Review (VR) team and which has a body made of Low Temperature Co-Fired Ceramic (LTCC). The LTCC material being used is an alumina based ceramic that is built up in layers prior to firing. The LTCC body allows passive electronics and tamper indicating features to be incorporated into the seal body.

Ceramic Seal Mechanical Design

For development of the Generation I Ceramic Seal, several ceramic seal body materials were fabricated and evaluated, including Macor machinable ceramic, zirconia ceramic and alumina ceramic. Macor was determined to be too fragile for this application. Zirconia and alumina have comparable material properties in that they both have adequate flexural strength while having a frangible ceramic nature which aids in tamper indication. Alumina was chosen because it was most compatible with the brazing research being performed by SNL.

The design of the Generation I Ceramic Seal (Gen I) body includes a closure mechanism employing a snap ring that is protected by a tortuous path integrated into the interface between the two seal halves, called the seal cap and the seal body. Part of the concept for Gen I was use of an application tool to be used for alignment of the seal halves for proper electronics functionality, closure of the seal, and seal wire cutting. Testing was performed to test these functions and to determine whether an alumina seal body could be used as a shear surface for seal wire cutting. Figure 2 shows the Application Tool Test Mechanism being compressed using a commercial clamp. This testing proved the ability of the alumina seal body to be used as a shear surface for stainless steel seal wire and the ability of the tool to perform all of the application functions, although the seal wire was not always fully sheared. Also, the gripping force required to close the clamp is greater than desired. [2]

Figure 2: Application Tool Test Mechanism in Commercial Clamp

The Gen I seal design required greater than hand force to insert the seal wire into the seal body and it was thought to be desirable to cut the seal wire flush with the seal body. Feedback from the Sandia VR team led to alternative seal wire routing and the option to leave a small length of wire exposed outside the seal. The Gen II seal design incorporates alignment features and the new seal wire routing allows manual closure of the seal. Due to these factors, the Gen II seal eliminates the need for an application tool.

One of the features of the Ceramic Seal that is critical to the operability and security of the seal is seal wire routing. Tying or crimping of the seal wire is not desirable because of the variability inherent to these wire securing techniques. Several seal wire routing options were designed and tested. The left photo in Figure 3 shows the Gen I seal wire routing in a zirconia seal body, along with a cap, battery, and a quarter for a size reference. The center and right photos in Figure 3 show plastic rapid prototypes of the two primary Gen II seal wire routing options. The seal wire routing design was changed due to VR team input and also to allow the seal to be assembled without an application tool. The wire routing depicted in the right hand photo had the best seal wire retention results. In addition, SNL tested both polymer coated stainless steel wire rope and low E guitar string as possible seal wires. The guitar string was able to be secured in the seal more effectively than the coated stainless steel wire rope.

Figure 3: Left, Gen I Wire Routing. Center & Right, Gen II Wire Routing Options. Photos courtesy of SNL & SRNL.

Other than seal wire routing and use of the LTCC manufacturing process, several other changes were made to the seal mechanical design for Gen II. For the Gen I seal, alignment of the cap and body was achieved by the application tool and retained by an adhesive to be developed by SNL. For Gen II, alignment features were added to the interface between the cap and body so that alignment does not rely on the performance of an adhesive. In addition, an o-ring was added to the internal interface between the cap and body which serves to pull the cap and body together and is an environmental barrier for the internal electronics.

Coatings Development

SRNL has researched functional coatings both for protection of the exterior ceramic surface for Laser Surface Authentication (LSA) and for tamper indication. LSA reads the reflections of a red laser off of the surface of the ceramic seal to develop a unique ID of the seal surface. Transparency of the coating in the red visible light wavelength range is required for the coating to be compatible with LSA. Both fluorescent and electrically conductive coatings have been investigated for tamper indication.

The ceramic materials used for the seal body and cap are electrically insulating. Application of an electrically conductive coating to the surface of the Ceramic Seal was proposed to allow mapping of surface sheet resistivity/ conductivity which would be sensitive to cracks, holes or cuts indicative of tampering. Indium tin oxide (ITO), ruthenium oxide, and iridium oxide were chosen for investigation. The change in resistivity/ conductivity with tampering was approximated by scratching the films using a diamond tipped blade. Electrical characterization of the coatings was performed using the van der Pauw method. The resistivity of the ITO coated samples was found to be the most sensitive to surface scratches. This property, in addition to the optical transparency of ITO, makes ITO a promising candidate as a conductive tamper indicating coating. However, when Ceramic Seals are installed in a facility, it is likely that inadvertent scratches could result in false tamper indications using this technique. Conductive coatings could be used for tamper indication on the interior surfaces of the seal, but this would require additional development that could not be supported in this project. After the first year of this project, conductive coatings research was suspended due to operational concerns.

Fluorescent coatings applied to the seal body could provide the inspector with a quick and easy method to inspect the seal integrity. The surface could be inspected using a UV flashlight to check for defects in a continuous coating which could indicate cutting, drilling or other methods of seal penetration that would trigger in-depth seal verification. Extensive research has been performed by SRNL for development of a rugged fluorescent coating that will remain stable in an outdoors environment. Alumina and silica transparent sol-gel films and sputtered ITO films were considered as matrix materials for the prospective fluorescent dopants. ITO was eliminated first as an option because the sputtered layers are so thin that it would be impractical to deposit enough layers to obtain adequate fluorescence. Alumina sol-gel films were found to be more stable than silica sol-gel films when combined with fluorescent dopants. In addition, the chemical and thermal expansion compatibility between alumina coatings and the alumina ceramic seal body made this the host material of choice for fluorescent dopants. Terbium and erbium were selected as fluorescent dopants due to the capability of excitation in both the UV and visible regions.

UV-Vis absorption spectroscopy measures the attenuation of light after it passes through a sample and was used to determine the transparency of coatings to determine whether they are suitable for application to the seal. Figure 4 shows a comparison between an uncoated quartz slide and a slide coated with alumina sol-gel (Al_2O_3) doped with 0.1mol Tb. This figure demonstrates the transparency of the doped coating, which indicates that it can be used as a protective coating for LSA.

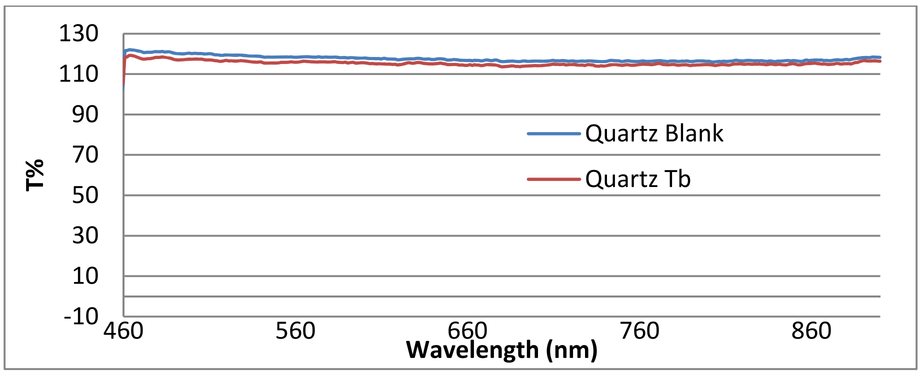

Figure 4: Transmittance measurements comparison between quartz and Tb coated sample

To increase the fluorescence of the coating, various factors including dopant concentration, use of sensitizers, and annealing temperature were studied. It was found that higher concentrations of dopant do not necessarily result in greater fluorescence, apparently due to a phenomenon called "quenching" where neighboring dopant atoms interfere with the photoluminescence process (see Figure 5). Incorporation of the optical sensitizer gadolinium (Gd^{3+}) was studied in an attempt to prevent this quenching process. A terbium/ gadolinium solution was examined in an ethanol environment. Ratios of Tb^{3+}:Gd^{3+} of 1:10, 1:20, and 1:30 were compared to Tb^{3+} only. Photoluminescence (PL) measurements of these solutions showed that the addition of gadolinium did not increase photoluminescence; in fact, photoluminescence decreased as the gadolinium ratio increased.

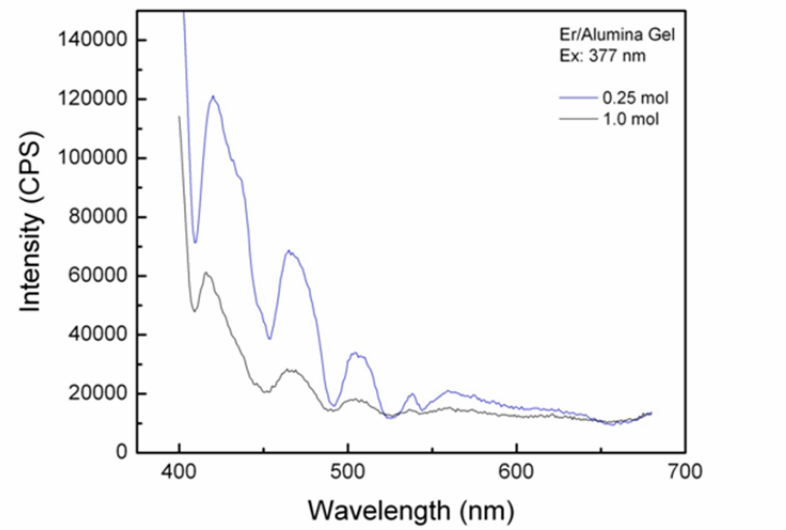

Figure 5: Photoluminescence spectra of Alumina gel doped with Er under different concentrations

PL measurements of samples with annealing temperatures of 400°C, 600°C, and 800°C were compared to samples that were not annealed. It was found that photoluminescence intensified as the annealing temperature increased. 800°C is close to the processing temperature of LTCC and could adversely affect the integrity of the seal body, so 600°C is being used for samples currently being studied. Another interesting phenomenon discovered is that alumina samples from the Ceramic vendor AstroMet fluoresced red when excited with 365 nm light. After discussing this with AstroMet, it was determined that the red fluorescence was caused by chromium impurities in the alumina substrate. The red can be clearly seen through the doped alumina coating (see Figure 6).

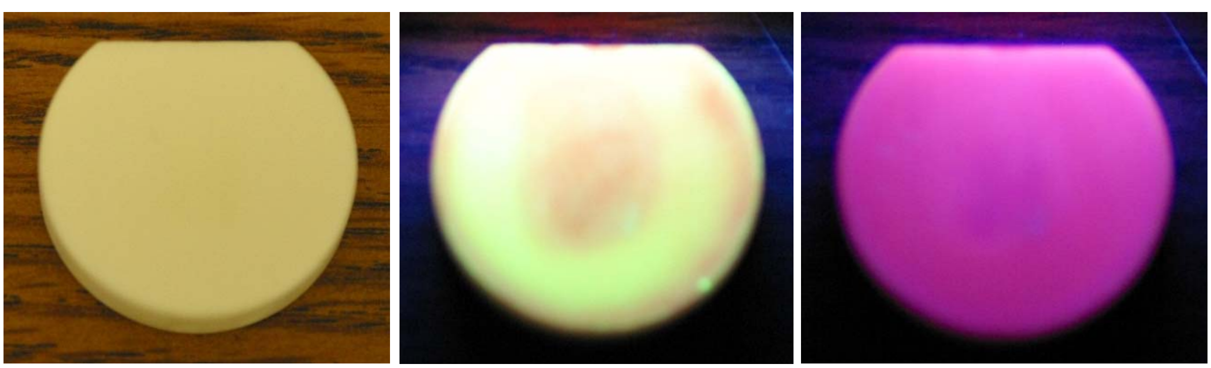

Figure 6: AstroMet sample coated w/ Tb doped Al_2O_3.
Left – room light, center - 254 nm (Tb coating), right - 365 nm (Cr substrate)

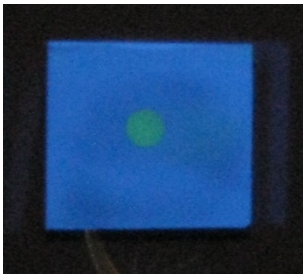

Figure 7: LTCC sample w/ fluorescent spot

SRNL has utilized a spin coating technique to apply sol-gel coatings to the samples. This technique works well for applying coatings on flat surfaces, but not for cylindrical surfaces such as the side of the seal body and cap. To apply coatings over the entire exterior surface of the seal, SRNL subcontracted Tetramer Technologies to use a spray coating technique. Figure 7 is a LTCC sample illuminated with 254 nm UV light. The spot in the center of the sample is spray coated by Tetramer with a Tb doped alumina coating. The surrounding blue color is the natural color of the LTCC material.

Radiation Shielding Study

SRNL performed modeling of Ceramic Seal exposure to radiation fields to determine whether additional shielding of the Ceramic Seal is needed to protect the functionality of the electronics components.[3] IAEA Recommended Doses and Fluences were used for the evaluation. It was found that the Ceramic Seal does not provide significant shielding for high energy photons and fast neutrons. Shielding of high energy photons is not practical due to the weight and thickness required to provide adequate shielding. Shielding of thermal neutrons is feasible; simulations show nearly 100% attenuation when employing samarium, gadolinium, or cadmium coatings at a thickness of 30 microns.

Before a recommendation to pursue thermal neutron shielding applications can be given, it is necessary to identify the likely radiation environments in which the Ceramic Seals will be deployed. A serious attempt should be made to investigate potential applications of the seal such that characterization of the appropriate radiation field can be used to either direct or curtail development of neutron shielding efforts.

When dealing with electronic devices such as those in the Ceramic Seal, ionization damage can vary widely for devices of the same type that are produced by different manufacturers. Future work, regardless of neutron shielding development, should therefore include experiments designed to test the exposure limits of operable, completed seals. This is the best way to assign perceived risk to the seal as a function of radiation type, energy, and amount. No such experiments have been performed in order to focus efforts on completion of tasks in support of completion of the Gen II LTCC seal prototype.

Laser Surface Authentication (LSA) Studies

SRNL is investigating the use of LSA as a technique to obtain a unique ID of the seal prior to installation of a seal in the field. The LSA technology was developed by Ingenia Technology and uses a small flatbed scanner and proprietary software to scan the surface of an item. The variations in the reflections from the scanner lasers are detected and processed to form a unique "fingerprint" of the surface being scanned. This would be used as a verification technique on a random sampling of seals that are returned to a laboratory supporting the seal inspector's organization.

In the LSA system the number of bits matched divided by the number of bits tested gives the bit match ratio (BMR). Scans which match should give a BMR greater than 65% and scans which don’t match give a BMR of 50–55%. However a BMR greater than 80% is desirable to indicate that the compared scans correspond to the same sample.[4]

During initial trials of the LSA technique, the LSA system was unable to uniquely identify Gen I alumina Ceramic Seal bodies on a consistent basis. Discussion with Ingenia technical support led to investigation of the impact of surface roughness on LSA performance. To determine the compatibility of the LSA technology with the Ceramic Seal, several variables were examined including surface roughness, manufacturing processes that produced the surface roughness, sample placement, and comparison of coated samples with uncoated samples.

In Figure 8 the bars represent the average roughness of the samples and the circles represent the average BMR of the samples when compared with themselves. The lines extending above and below the circles represent the range of BMR values for each sample. Figure 8 shows that both surface roughness and manufacturing process effect BMR. A surface roughness greater than 1 micron is desirable. It was also found that manufacturing processes such as surface grinding result in greater BMR variability because the parallel lines produced by the grinding cause the BMR to be greater when the surface is scanned perpendicular to the lines rather than parallel to the lines. Surface modification using a polishing wheel produces random marks that will help to maintain a consistent recognition and eliminates the effect of the scanning direction. It also helps to create a more random surface suitable for unique ID. Since the surfaces of the seal to be LSA scanned are flat, the polishing process can be performed on a production scale at an affordable cost. Studies of sample placement on the LSA scanner indicate that the placement of the sample must vary no more than 1 mm or 0.5° to obtain consistently acceptable BMR values.

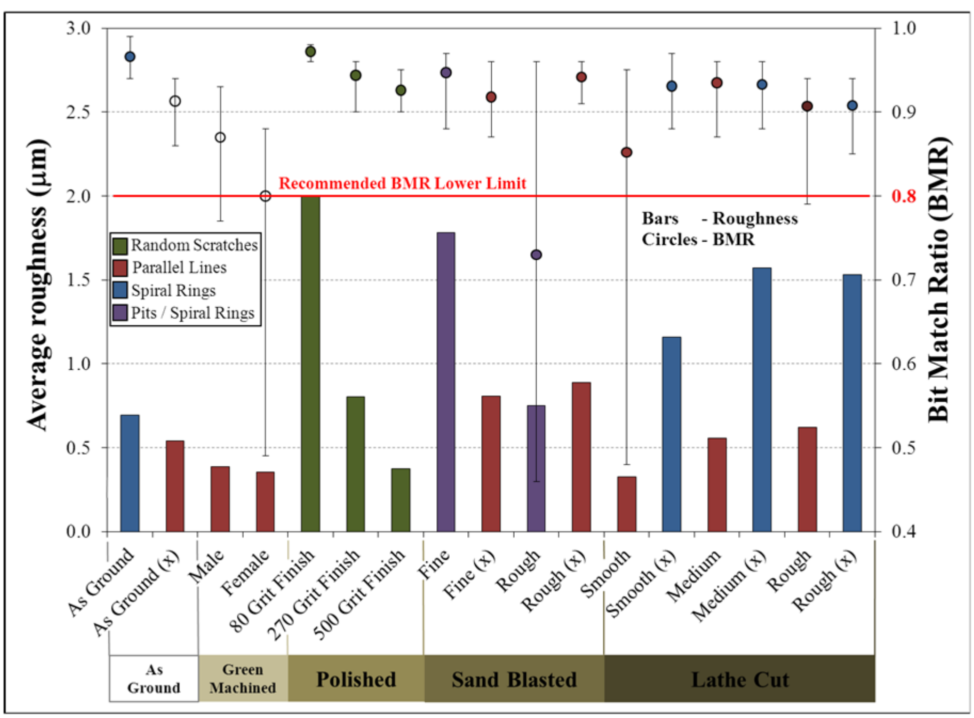

Figure 8: Average roughness and corresponding BMR for ceramic samples with different manufacturing processes.

Compatibility of the tamper indicating coatings with the LSA technique was also investigated. Comparison of uncoated ceramic samples with the same samples coated by SRNL resulted in BMR values below 0.6, suggesting that the coating is interfering with recognition of the samples. However, scans of an uncoated alumina sample compared with the same sample coated with ITO showed that the BMR increased between the uncoated samples and the ITO coated samples when the ITO coating was made more transparent by annealing. This indicates that some of the underlying features of the ceramic surface are recognized in the scans. If the unique ID of the seal is a result of a combination of the specific application of the coating and the underlying seal surface, the unique ID could be even more difficult to replicate.

Conclusion

The Gen II Ceramic Seal is currently being fabricated by SNL and will be sent to SRNL for application of tamper indicating coating by Tetramer Technologies and initial LSA scanning by SRNL. The seal will then be sent back to SNL for a vulnerability review of the entire seal. SRNL and SNL have proposed to NA-22 a continuation of the Ceramic Seal development effort, preparing the seal for commercialization by production and functional testing of multiple prototypes and also development of a hand-held reader for in-situ initialization and verification of the seal electronics. Improvement of the coatings would also be researched, including investigation of incorporation of unique ID properties into the coatings.

Acknowledgement

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