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Sent:	Monday, March 02, 2015 9:07 AM
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Subject:	Docket ID NRC-2014-0209

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Dear NRC,

While being contacted by AREVA for procurement of nonmetallic insulation material for the next EPR in construction in France, I discovered your project to revise the NRC RG 1.36.
Unfortunately, it seems that the delay to be sure that comments will be taken into account is already over ... but it also seems that according the Federal Register (October 6, 2014) Notice, "*comments and suggestions in connection with items for inclusion in guides currently being developed [...] are encouraged at any time*."

Some parts of US NRC Guidance documents have been almost "copy/paste" inside Nuclear Power Plants construction rules used by AREVA in France, and therefore their content and evolution are of concern for us.
So please find hereunder some comments on your draft revision of RG 1.36, i.e. document draft regulatory guide (DG), DG-1312.

As a general comment, I do appreciate that RG 1.36 goes under revision. Such an old (1973, so more than 40 years) guide, based on outdated standards, was not convenient for use today.
On the other hand, such guidance is welcomed since standards (whether ASTM or ISO or European (EN) ones) are not always complete enough for defining the acceptable procedure for material procurement.

For nonmetallic insulation used on austenitic steel, one of the basic standards is ASTM C795 "Standard Specification for Thermal Insulation for Use in Contact with Austenitic Stainless Steel", which was first published in 1977.
This ASTM C795 is mainly based on 2 other standards were some testing are defined: ASTM C692 (first edition in 1971) and ASTM C871 (first edition 1977).

If the acceptance procedure of nonmetallic insulation products regarding stress corrosion cracking (SCC) defined in ASTM C795 is similar, in its concept, to the existing RG 1.36, some big differences exist.
For instance:

- The chemical analysis procedure to determine the ion concentrations of leachable chloride, fluoride, sodium, and silicate is loosely defined in RG 1.36, while ASTM C795 refers to a more detailed one (ASTM C871);
- Until last edition of ASTM C795 (in 2008, reapproved after the 5 years review in 2013), the Fluoride leached was not taken into account for the acceptability of nonmetallic insulation, while it was in RG 1.36;
- The acceptance of a lot is defined in ASTM C795 as (to sum it up) "pre-production test according ASTM C692 passed, and chemical analysis (according to ASTM C871) of all specimen in a lot sampling (for the delivered lot) shall be in the acceptable area of Figure 1 of C795";
- The acceptance of a lot is defined in RG 1.36 as (to sum it up) "a preliminary qualification phase consisting of pre-production test (similar to ASTM C692) passed with, in parallel a chemical analysis within the acceptable region of Figure 1, and a subsequent production phase test with only chemical analysis within the acceptable region of Figure 1 with no analysis result (Cl+F and Na+SiO3) deviation of more than 50% of the results from the qualification phase".

The proposed revision solved most of the above differences.
But it seems that the "50 % deviation" criteria, replaced in DG-1312 by "any statistically significant variation" triggered some comments.

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If I can agree that a criteria such as "any statistically significant variation" cannot really help to decide if the tested product can be or not accepted, I am also opposed to the simple maintain of the "50 % deviation criteria", which is not based on scientific grounds and can create real problems!

A lot of scientific studies on the SCC phenomenon has been conducted, and one of the last ones has been published in 1997, prepared by K.M. Whorlow & al. (Tutco Scientific Corporation).
One could have expected that this study, Effects of Fluoride and Other Halogen Ions on the External Stress Corrosion Cracking of Type 304 Austenitic Stainless Steel (ref NUREG/CR-6539), could have been used as a base for the revision of the RG 1.36, but (despite the inclusion of Fluoride measurements which was already the case in 1973's RG 1.36), I've not been able to find real implementation of the conclusions of it.
For instance, the fact that "the mole ratio of sodium-to-silicate had an effect on the inhibition of chloride-induced ESCC", that "many ESCC cracks were not visible unless the test coupon was flattened, re-bent, and examined using dye penetrant and 30x magnification", or that "ESCC cracking with fluoride, bromide, and iodide was not as severe as with chloride", are not taken into account in this revision.

Of course, the NUREG/CR-6539 study goal was not to define the acceptability criteria of the production lot.
But from this study, we can conclude that depending the "pass" criteria used when testing according ASTM C692 (number of steel coupons that have cracks, size of the cracks, exact type of steel used, etc.), the parallel results of chemical analysis according C871 can lie inside or outside the "Karnes' diagram".
In other words, the test procedures are sensitive to variation in different parameters, so acceptance criteria needs maybe to be defined according such uncertainty.

It seems strange, for me, to qualify some tons of insulation products by just analyzing some grams of it.
The ASTM C390 defines "Standard Practice for Sampling and Acceptance of Thermal Insulation Lots", and I agree with the comment saying that this C390 role should be more emphasized in RG 1.36 revision.
ASTM C795 §11 on "Acceptance and retests" is based on "*the average analysis of the two specimens taken from any sample*" of a lot sampling (as defined by C390).
Furthermore, ASTM C795 clearly states that "*Physical and chemical changes can occur when thermal insulation, various binders, or adhesives, or a combinations thereof, are heated. Insulation materials are often exposed to process temperatures that are sufficient to cause changes.*" And that "*A variety of acids and ionic chemical solutions are known to induce metal pitting, hydrogen embrittlement, intergranular corrosion and stress corrosion cracking on sensitized austenitic stainless steel. The results of Test Methods C692 corrosion test are expected to indicate if there is an untested agent in the insulation that will induce cracking.*"

I propose:

- that the number of minimum specimen to be tested according ASTM C871 (for "production testing" as mentioned in DG-1312) should be stated in the revision of RG 1.36 (maybe based on ASTM C390 Table 1),
- that the acceptance criteria should be the ones in §11 of ASTM C795-08,
- and that the use of ASTM C692 should be limited, as a "preproduction test" of unknown product, to only check if such unknown product could induce SCC.

Two last comments:

ASTM C871-11 §13 "Precision and Bias", clearly demonstrate that the "50 % deviation criteria" is nothing than a "best guess". On the Chloride measurement alone, a round robin organized among more than 7 different labs, showed that the "*standard deviation as a percentage of the mean value*" can be as high as 24 % for low measured values of Cl (less than 20 ppm). In other words, this means that a very <u>same</u> material can be measured at 6 ppm of Cl in one lab, and at 11 ppm in another lab ... I let you do the math if you apply the "50 % deviation criteria" ...
In Germany, there is a criteria named "AS-Quality", based on leachable Cl measurement (according European measurement standard EN 13468, similar to ASTM C871) which requires that "*the average values of the 6 tested*

samples ≤ 10 mg/kg insulant" and "*individual measurements ≤ 12 mg/kg insulant*". This means that AS-Quality insulation products are often in the range where the "50 % deviation criteria" cannot be ensured.
This also means that there is a high risk of rejection of "acceptable" material if this 50% criteria is applied (in other words, need to re-do an almost 3 months long and expensive ASTM C692 test ... with a new sample where the measured Cl could be again challenged later)!

The French Nuclear Plants manufacturer AREVA has defined a "*chemical analysis procedure*" based on the EN 13468 , but with some change in it (I underline the main change):
"*A lixiviation test in water is performed on the insulating material, according to the standard RCCM F6423 Annex F V.B (with water at 95°C during 1/2h in a PTFE beaker). The test is not performed in a boro-silicated glass container as recommended in the standard EN13468 as it may lead to sodium and silicate release.*"
The ASTM C871 §9.3 and 9.4 state (I underline): "*Quantitatively transfer the mixture to a tared 1-L stainless steel or borosilicate beaker, rinsing with distilled or DI water. Bring to boiling and maintain at the boiling point for 30 ± 5 min.*"
This means that among the parameters that might have an influence on the chemical analysis, the kind of container used by the lab can have an influence.
We have such experience, where a same product sees variations of far more than 50 % of measured quantity of Na or SiO_3 ions, while still lying in the acceptable zone of the Karnes' diagram.

For all those reasons, it is important for us that the "50 % deviation criteria" appearing in the RG 1.36 shall be revised. Maintaining as such, this criteria will not improve safety, but will lead to delays and over expenses (adding re-tests according ASTM C692) to accept "acceptable" materials.

Sincerely,

Yves DUDOGNON
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