Subject: Re: SS hardware material limitations
Date: Mon, 12 Jan 2009 10:33:01 -0800
From: John Worden <worden_j@ligo-wa.caltech.edu>
To: Dennis Coyne <coyne@ligo.caltech.edu>
CC: Calum Torrie <torrie_c@ligo.caltech.edu>, Mike Zucker <mike@ligo.mit.edu>, Fred Raab <fjr@ligo-wa.caltech.edu>, Riccardo DeSalvo <desalvo_r@ligo.caltech.edu>, Rainer Weiss <weiss@ligo.mit.edu>, John Worden <worden@ligo.caltech.edu>, Dennis Coyne <coyne@ligo.caltech.edu>

```
Dennis;
Yes, we agreed to allow the use of fasteners made with free-machining
stainless steel (of the 303 and 303Se variety).
John

Dennis Coyne wrote:
> John,
> The "official" reply from the Vacuum Review Board (VRB) on the
> question of the use of free-machining stainless steel, T080044-00, is
> posted on the VRB log in the wiki (item 7):
> http://lhocds.ligo-wa.caltech.edu:8000/advligo/System-wide_Information/VRB
>
>
> However, I recall speaking with you, about agreeing that SS "hardware"
> (bolts, nuts, washers) were exempt from the 303 restriction due to
> their small size. Would you please confirm in an email reply and then
> I'll amend the VRB's official response to include this exemption for
> hardware.
> Thanks,
>   Dennis
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-------- Original Message --------

Subject: Re: L080042: Is 303 stainless steel acceptable in the LIGO Vacuum system?
Date: Mon, 28 Apr 2008 09:38:35 -0700
From: John Worden <worden_j@ligo-wa.caltech.edu>
To: Michael Zucker <zucker_m@ligo.mit.edu>
CC: Norna Robertson <nroberts@ligo.caltech.edu>, raab_f@ligo-wa.caltech.edu, Dennis Coyne <coyne@ligo.caltech.edu>, Rainer Weiss <weiss@ligo.mit.edu>, Mike Zucker <mike@ligo.mit.edu>, Riccardo DeSalvo <desalvo@ligo.caltech.edu>, Janeen Romie <janeen@ligo-la.caltech.edu>, Calum Torrie <c.torrie@physics.gla.ac.uk>, Justin Greenhalgh <J.Greenhalgh@rl.ac.uk>

Refer to L080044 (This document).In response to L080042: Is 303 stainless steel acceptable in the LIGO Vacuum system?

The VRB response is:

1. Existing 303SS already installed at LLO will remain in HAM6 until we need to remove the septum or install ADL. At this time the 303SS parts are replaced at LLO.
2. All 303SS parts (if possible) for LHO will be remade with cost and schedule impact addressed via CCB.
3. E960050 receives an edit to remove 303SS from the acceptable materials list.

John Worden for the VRB.

Michael Zucker wrote:

> Aha! OK Norna, I just read OMC, not OMC SUS!
>
> Now I know where to look, is it the upper mass block D060491 under the assembly D070024? the small hardware clamps associated? or the tablecloth? or all of the above? The edrawing doesn't show the material callouts.
>
> I suggest calling up ASAP to figure out where they are on each questionable part and, exercising some judgment, have them order new material for those not started. Also might consider just remaking (in parallel) those already begun or completed, if necessary going to another shop so as not to compete with ourselves. Is this job still with Mike Gerfen over in CES or did we sub it out?
>
> I'll presume we (maybe I?) can prepare a defensible CCB for the cost impact. Schedule impact needs to be estimated, but even if the delay proves to be unjustified by the present issue, there's always finite chance of some unrelated delay.
>
> Not that we should wish for one...
>
> Thanks for clarifying,
>
> mike

On Apr 25, 2008, at 2:30 PM, Norna Robertson wrote:

> Dear Mike and colleagues
>
> Just to clarify - the 303 stainless part(s) in the OMC is(are) in the top mass above the double pendulum. So even if Sam changed the silica bench to correct the problems mentioned below, the 303 would NOT get replaced unless and until we have replacement metal parts for the *whole* suspension.
> Here is the status. We are are currently underway in making the LHO suspension using 303 - which of course we can halt and change to another steel if that is the wisdom of this group, noting this will hit cost and schedule. (For your info we have been informed by the workshop that 304 takes considerably more effort to work - I think someone questioned this.) We have no plans at present to remake the LLO suspension until it is done as part of the Advanced LIGO project and that is not scheduled for another year or two (e.g. final design review is currently scheduled May 2009 and production of all HAM suspensions starts April 2010). So changing the schedule to produce another OMC suspension for LLO without 303 in it before then would require availability of $$ and effort earlier than planned.
>
> Norna
>
> At 10:23 AM 4/25/2008, Michael Zucker wrote:
>
>> John-
>>
>> Yes I think that's a possibility we can't rule out. It's a
>> while away yet, but may be needed for S6 (for example, if we get into
>> VIRGO-like troubles with phase noise
>> from the window). As you know, we've been pretty religious about
>> allowing no vacuum
>> compromise in HAM6, precisely to maintain this option.
>>
>> We've had other issues with this OMC , it being the
>> "prototype" (thermal actuator fell apart, mirrors got contaminated, one
>> mirror mount shifted while curing...) but things got patched up one
>> way or another and it hung in there.
>> Nevertheless, there are now enough parts laid in to set up a
>> replacement if we wish.
>>
>> I'm thinking to ask Sam to proceed along those lines and replace the
>> prototype on L1 before we consider
>> removing the window. I'll send him a note.
>>
>> Mike
>>
>> On Apr 25, 2008, at 1:03 PM, John Worden wrote:
>>
>>> Fred,
>>>
>>> Isn't it possible that we remove the septum window sooner rather
>>> than later - say the window becomes the limiting component either
>>> for commissioning or sensitivity reasons in S6? In this case the 303
>>> material would be exposed to our main volume. I don't know if this
>>> is a concern given the quantities.
>>>
>>> john
>>>
>>> Fred Raab wrote:
>>>
>>>> Thanks, Dennis. It seems like the best action is to remove 303 SS
>>>> from the approved list, fix drawings that spec it (red lines should
>>>> be a good start, but rev's in reasonable time would be needed to
>>>> ensure it does not creep back in). It sounds like the stuff already
>>>> in the main vacuum volumes is small amounts, if any, and we should
>>>> not worry about that. It'll come out by 2011. As for the OMC parts,
>>>> I propose we waiver them, so as not to delay testing of DC read- out. My understanding is that eventually the HAM 6 septums will

go
away, but the current OMCs will go away first. We might want to
replace those parts later if the OMCs were ever recycled for use in
our main, highest-quality, vacuum volumes.

Fred

Dennis Coyne wrote:

> Some replies to your questions below
>
> Fred Raab wrote:
>
>> Dennis,
>>
>> Do we already have significant 303 SS components in the LIGO
>> vacuum system?
>
> The only components composed of 303 (or "300 series" without
> knowledge of the specific grade) in order system at present to my
> knowledge are parts of the OMC and (as Mike points out) possibly
> SS fasteners.
>
>> Presumably the LLO OMC has already gone through a bake oven?
>
> Yes.
>
>> Can the oven be baked clean now?
>
> As Rai suggests we can bake our ovens with air in the chamber to
> oxidize the S, Se and P.
>
>> >From your note, it appears that we would have good reason to
>> exclude 303 SS from the list of acceptable materials. One could
>> imagine a waiver for the septum-separated HAM with OMC for now.
>> My feeling is that LIGO faces a bigger risk right now from the
>> OMC and DC detection not working out well than from irreversible
>> vacuum contamination of HAM 6. My concern is whether we can still
>> prevent future contamination of the rest of the vacuum system or
>> have we already "bought the pig".
>
> I think we can prevent future contamination of the main vacuum
> volumes if we act now. As for the isolated HAM6 volumes, there may
> be some contamination but the vapor pressure is fairly low. Worst
> case scenario is we consider a bake of the HAM6 chamber with air
> in it to oxidize the S, Se and P. However, this is perhaps overkill.
>
>> Is there any indication that we had positive evidence when the
>> list was made up that 303 was OK, despite what O'Hanlon and SLAC
>> say?
>
> Mike gave you his recollection in a separate email.
> In addition to O'Hanlon and SLAC:
>
> 1) Phil Danielson, "Choosing the Right Vacuum Materials", R&D
> Magazine, April 2003.
> "Free-machining alloys such as 303 SS contain sulfur (S), but the
> vapor pressure of the S is too high for high vacuum systems."
> http://www.vacuumlab.com/Articles/VacLab36.pdf
>
> 2) The Advanced Light Source (ALS) Vacuum Policy and Vacuum
> Guidelines for Beamlines and Experiment Endstations, section 5:
> http://www-als.lbl.gov/als/quickguide/ vacuumpolicy.html#anchor9792047
> Lists this note under "marginal" materials: "Stainless Steel: SS

containing excessive amounts of sulfur or selenium must be avoided."

3) HASYLAB Vacuum Guidelines for Beamlines and Experiments
Prefers 316L and 316LN. Does not prohibit other stainless steels,
other than by the statement "Standard UHV-compatible materials
have to be used for all vacuum components."
http://hasylab.desy.de/infrastructure/vacuum_group/guidelines/vacuum_guidelines/index_eng.html

Fred

Dennis Coyne wrote:

LIGO-L080042-00

To the Vacuum Review Board (VRB):

The current LIGO Vacuum Compatible Materials List:
http://www.ligo.caltech.edu/docs/E/E960050-B/E960050-B.pdf
states that 303 stainless steel is acceptable. However,
O'Hanlon's "A User's Guide to Vacuum Technology" clearly states
that 303 should be avoided in UHV applications due to the
addition of sulfur, phosphorous or selenium. In addition, the
SLAC Vacuum Department Guidelines for Vacuum Systems:
http://lhocds.ligo-wa.caltech.edu:8000/advligo/UHVWelding?action=AttachFile&do=get&target=SLAC-I-007-12004-001-R1.pdf
states in section 3.1:
"Types 303, 303S, and 303Se contain excessive amounts of sulfur
or selenium and are not acceptable."
It should also be noted that all of our welded vacuum chambers
are comprised of 304L.

It might be argued that this prohibition is very likely due to
high vapor pressure and diffusion associated with these alloys
when baked to clean up the parts. Since we do not plan to do in- situ vacuum baking we might in principal get around this
restriction by baking in air and performing FTIR cleanliness
certification tests (rather than RGA tests). Could this approach
be acceptable? A rather simplistic analysis (given below)
indicates that this may not be acceptable. The exceedingly high
vapor pressures for the elements Se, S and P suggest that even
at room temperature there is a risk of plating optics to
significant thickness in short durations.

Can we permit (continue to permit) 303 and 303 Se stainless
steel into the LIGO vacuum system? One of the parts of the LLO
OMC is comprised of 303 and others were called out as 300
series. Presently we are machining parts for the LHO OMC. Can we
accept these parts? Should we remove 303 and 303 Se from the
LIGO UHV approved materials list? This decision will have
significant consequences since there are many drawings which
call out 303 or 300 series SS and their are many prototype parts
which have used 300 series SS. Moreover the machining costs for
302, 304 or 316 are higher.

I request a rapid reply from the VRB given the time critical
nature of the OMC work for enhanced LIGO.

The simplistic analysis follows:
The composition of 303 stainless steel is as follows (source is
matweb.com):
Carbon, C <= 0.150 %
Chromium, Cr 18.0 %
Iron, Fe 69.0 %
Manganese, Mn <= 2.00 %
Molybdenum, Mo <= 0.600 %

Nickel, Ni 9.00 %
Phosphorous, P <= 0.200 % [very small amount]
Silicon, Si <= 1.00 %
Sulfur, S >= 0.150 % [*Note no upper limit*; I couldn't find typical values]

The percentages of the three elements of concern in the composition of 303 SE is as follows:
Phosphorous, P <= 0.200 %
Selenium, Se <= 0.150 %
Sulfur, S <= 0.0600 %
All rather small percentages.

The percentages of the three elements of concern in the composition of 303 MA is as follows:
Phosphorous, P 0.0400 %
Sulfur, S 0.140 %
Also all rather small percentages.

For the purpose of getting some idea of the potential harm of these three elements in a 303 SE alloy, let's assume ~0.2 % maximum amount by weight. N.B.: This does not allow a 303 SS, which is likely (?) to have a few % of S.

The vapor pressures of sulfur, phosphorous and selenium (over an elemental solid) are:
Po = {1e-10 Se, 2.0e-6 S, 2.0e-10 Pred) torr @ 20C
Po = {2.0e-3 Se, 2.0 S, 5e-2 P} torr @ 200C
N.B.: I've used red Phosphorous in these calculations. White Phosphorous has a vapor pressure even higher than Sulfur. I'm not sure which allotropic form to use in the calculation.

The vapor pressure of these elements over a 303 alloy (with c = % Se, % S, %P by Wt.) will be lower than the vapor pressure over a solid of the pure element. For an ideal solution (alloy), with low molar fraction, the vapor pressure lowering is given by Raoult's law:

p = x(Z) * Po, where x(Z) is the mole fraction of the element Z
x(Z) ~ c*Ar(Fe)/Ar(Z), where Ar is the relative atomic mass.

Ar(Z) = {78.96 Se, 32.066 S, 30.97 P, 55.845 Fe)
x(Z) = {1.4e-3 Se, 3.5e-3 S, 3.6e-3 P}
p = {1.4e-13 Se, 7.0e-9 S, 7.2e-13 P} torr @ 20C
p = {2.8e-6 Se, 7.0e-3 S, 1.8e-4 P} torr @ 200C

Although I could not find a reference for the vapor pressure of these elements over an alloy, I did find a reference for Fe vapor pressure over a solid Vanadium-Fe alloy (Myles & Aldred, "Thermodynamic Properties of Solid Vanadium-Iron Alloys", J of Physical Chem, v68, n1, Jan 1964). At ~10% molar fraction, the vapor pressure is in close agreement with Raoult's law; The non- ideal deviation further lowered the vapor pressure. The vapor pressure will also be lowered by any diffusion rate limited process through the condensed solid alloy.

On the issue of physical vapor deposition of Z onto our optics (and other chamber surfaces), the mass rate of evaporation is given by the Hertz-Knudsen equation (H. Lee, _Fundamentals of Microelectronics Processing_, 1989):

V [g/cm^2/s] = 5.834E-2 * Sqrt(M/T) * p

where T is in K, the molecular weight M = {78.96 Se, 256.5 S, 123.9 P} and where p is the partial pressure (torr)
V = {4.2e-15 Se, 3.7e-10 S, 2.7e-14 P} gm/cm^2/s @ 20C

V = {6.7e-8 Se, 3.0e-4 S, 5.3e-6 P} gm/cm^2/s @ 200C

The maximum deposition rate, assuming direct free-molecular streaming (no adsorption/desorption), disregarding solid angles and view factors, and no condensation rate-limited processes (if any), i.e. worst case, is then given as

rd = V*As/(Pi * r^2)
where As is the source area and r is the distance to the target (optic). Assuming a ~10^3 cm^2 source area at a distance of ~10 cm from the optic, then
rd = {1.3e-14 Se, 1.2e-9 S, 8.6e-14 P} gm/cm^2/s at 20C
rd = {2.1e-7 Se, 9.4e-4 S, 1.7e-5 P} gm/cm^2/s at 200C

With a Z density of {4.79 Se, 2.07 S, 1.82 P} gm/cc this corresponds to a maximum deposition rate of
{2.8e-15 Se, 5.8e-10 S, 4.7e-14 P} cm/s at 20C
{4.4e-8 Se, 4.6e-4 S, 9.3e-6 P} cm/s at 200C

If we assume a maximum tolerable thickness of ~1 nm (about 1 monolayer), then the minimum time to achieve this layer is
{9922 Se, 0.05 S, 590 P} hr at 20C
~1 sec at 200C
Obviously the vapor evolution (including diffusion out of the parent alloy) and deposition process is much more complicated, but this simple and conservative analysis leads me to the following conclusions:

* The use of any 303 or 303-SE stainless steel should not be permitted in the LIGO vacuum system. (Perhaps in small quantity for compelling reasons and for applications distant from optics it could be permitted with a waiver.)
* We must not bake 303 SS in a vacuum bake oven; This will cause contaminate the oven and likely contaminate subsequent loads.

Dennis

OTHER EMAILS:

-------- Original Message --------

Subject: Re: L080042: Is 303 stainless steel acceptable in the LIGO Vacuum system?
Date: Fri, 25 Apr 2008 07:15:43 -0700
From: Riccardo DeSalvo <desalvo@ligo.caltech.edu>
To: Dennis Coyne <coyne@ligo.caltech.edu>
CC: john Worden <worden_j@ligo-wa.caltech.edu>, Rainer Weiss <weiss@ligo.mit.edu>, Mike Zucker <mike@ligo.mit.edu>, Fred Raab <raab_f@ligo-wa.caltech.edu>, Norna Robertson <nroberts@ligo.caltech.edu>, Janeen Romie <janeen@ligo-la.caltech.edu>, Calum Torrie <c.torrie@physics.gla.ac.uk>, Justin Greenhalgh <J.Greenhalgh@rl.ac.uk>
References: <481104E7.4070907@ligo.caltech.edu> <Pine.GSO.4.56.0804250009540.8270@ligo.mit.edu>

```
For what it is worth, I asked a few questions, and so far I found
nobody in Virgo that recalls having used 303 in Virgo.
Apparently they staid out of it, either because they knew of, or
because of luck.  It is no a common steel in any case.
```

R

-------- Original Message --------

Subject: Re: L080042: Is 303 stainless steel acceptable in the LIGO Vacuum system?
Date: Fri, 25 Apr 2008 00:24:51 -0400 (EDT)
From: Rainer Weiss <weiss@ligo.mit.edu>
To: Dennis Coyne <coyne@ligo.caltech.edu>
CC: John Worden <worden_j@ligo-wa.caltech.edu>, Mike Zucker <mike@ligo.mit.edu>, Fred Raab <raab_f@ligo-wa.caltech.edu>, Riccardo DeSalvo <desalvo@ligo.caltech.edu>, Norna Robertson <nroberts@ligo.caltech.edu>, Janeen Romie <janeen@ligo-la.caltech.edu>, Calum Torrie <c.torrie@physics.gla.ac.uk>, Justin Greenhalgh <J.Greenhalgh@rl.ac.uk>
References: <481104E7.4070907@ligo.caltech.edu>

Dennis and others,
I also did not realize that 303 had sulfur, phosphorous and selenium in it. It would be sensible to remove 303 from the list of vacuum compatible materials but I see no reason to panic.

The vapor pressure of the three constituents at room T when diluted by their fraction in the base material is small and does not pose a problem. The most serious problem could arise in contamination of the bakeout ovens. A simple fix, should an oven have been contaminated, is to run the oven at bake temperature filled with some air (oxygen). Phosphorous and sulfur will oxidize into volatile compounds that are easily removed by pumping or entrapment into the gas. Selenium will form a non volatile oxide which may leave some dust behind.

RW

-------- Original Message --------

Subject: Re: L080042: Is 303 stainless steel acceptable in the LIGO Vacuum system?
Date: Thu, 24 Apr 2008 21:38:51 -0400
From: Michael Zucker <zucker_m@ligo.mit.edu>
To: Dennis Coyne <coyne@ligo.caltech.edu>
CC: John Worden <worden_j@ligo-wa.caltech.edu>, Rainer Weiss <weiss@ligo.mit.edu>, Mike Zucker <mike@ligo.mit.edu>, Fred Raab <raab_f@ligo-wa.caltech.edu>, Riccardo DeSalvo <desalvo@ligo.caltech.edu>, Norna Robertson <nroberts@ligo.caltech.edu>, Janeen Romie <janeen@ligo-la.caltech.edu>, Calum Torrie <c.torrie@physics.gla.ac.uk>, Justin Greenhalgh <J.Greenhalgh@rl.ac.uk>
References: <481104E7.4070907@ligo.caltech.edu>

Dennis-

I basically follow your logic and agree, it seems an unnecessary risk for new hardware.

I'd balk at yanking out stuff already installed, because honestly there seems to be a lot of conservatism (e.g., barring direct line of sight to an optic, every other surface should be a perfect pump for these metals, right?).

It's harder to evaluate the response for parts currently in fab, depends on details. For future construction, I think just redlining "don't care" or "generic 300 series" drawings to change the material designation would be reasonable, as long as everyone sending out for bids knows ahead of time to do it.

Did we get a (recent) direct indication shops would really charge a premium for calling out 304?
My impression is these days the fabrication advantage of free-machining alloy should be less important. Tools are much tougher now, machines more rigid. Screw machines might still enjoy longer tool life, but I wonder if we ever make enough of anything to care (OK maybe barrel nuts... but anything cuts better than nitronic 60!)

Speaking of which, if I'm not mistaken, our SS fasteners are usually thread-rolled of nominal 18-8, which is supposed to be loose-rated 304. But that would be worth checking.

304 (not L) is also generally cheaper and stronger, which could partly offset any fabrication penalty (316's neither cheaper nor stronger, of course).

I am assuming anything to be welded is always spec'd 304L or 316, with 308 or 316 rod, is that right?

Bottom line, I'll buy into your recommendations, and raise you two:

* put out an all-call to the engineering group see what we have in
fabrication that uses 303 (and
how much/how long it would take to remake in 304)

* check fasteners too

Mike