

Subject: [Fwd: Re: Zn vapor pressure]
From: Dennis Coyne <coyne@ligo.caltech.edu>
Date: Tue, 11 Nov 2008 11:09:08 -0800
To: Dennis Coyne <coyne@ligo.caltech.edu>

The VRB never officially (in writing) agreed with my proposed ban of high Zn alloy aluminums, such as 7075, but unofficially agreed. Below is some of the email dialog/correspondence on the issue (plus one more recent email on the same topic).

Dennis

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

Subject:Re: Aluminum alloys in vacuum

Date:Thu, 21 Jun 2007 08:03:46

From:Dennis Coyne <coyne@ligo.caltech.edu>

To:Ken Mason <kmason@ligo.mit.edu>

CC:Mike Zucker <mike@ligo.mit.edu>, Andy Stein <astein@ligo.mit.edu>, Brian Lantz <blantz@stanford.edu>

References:<467825BB.6030302@ligo.mit.edu>

Ken,

According to typical UHV practice Zinc- and cadmium-bearing metals and alloys are not considered UHV-compatible. The 7000 series have a considerable % of Zn and should be avoided. However the real problem appears to be low 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 could in principal get around this restriction. If you can avoid the 7000 series aluminum alloys and simply substitute with an acceptable aluminum alloy, this would be best. If you can't, then we can consider a waiver.

Note that many acceptable alloys contain some small levels of Zn and Cd. For example, 6061 is considered an acceptable UHV alloy by O'Hanlon (A User's Guide to Vacuum Technology) and it contains a maximum of 0.25% Zn. The composition specification for 2024 aluminum alloy is:

| Component | Wt. % |
|--------------|------------|
| Al | 92.2 - 95 |
| Cr | Max 0.1 |
| Cu | 3.7 - 4.5 |
| Fe | Max 0.2 |
| Mg | 1.2 - 1.5 |
| Mn | 0.15 - 0.8 |
| Other, each | Max 0.05 |
| Other, total | Max 0.15 |
| Si | Max 0.15 |
| Ti | Max 0.15 |
| Zn | Max 0.25 |

Copper is the principal alloying element. As a group, the 2000 series, wrought alloys are noteworthy for their excellent strengths at elevated and cryogenic temperatures, and creep resistance at elevated temperatures. O'Hanlon gives as acceptable UHV aluminum examples 4043, 5052 and 6061, but does not mention the 2000 series. However, I don't see any reason why 2024 would not be acceptable in the LIGO vacuum system. (The LIGO vacuum compatible materials list, [E960050](#), lists 2024 as acceptable.) Substituting 2024 for those BSC parts which are currently designated as 7075, and which are not stress or hardness critical, should be acceptable.

The 2024 tempered alloy is susceptible to stress corrosion cracking. Our environments are benign, but moisture from the air in storage under the constant pre-load should be looked at if this is the maximum service stress condition that caused ASI to choose the high strength of 7075.

I've attached detailed property data for 2024, 7075 and 6061 (probably more than you'll ever need).

Dennis

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

Subject:Re: Zn vapor pressure

Date:Sat, 10 Feb 2007 20:54:28 -0500

From:Michael Zucker <zucker_m@ligo.mit.edu>

To:Dennis Coyne <coyne@ligo.caltech.edu>

CC:John Worden <worden@ligo.caltech.edu>, Ken Mason <kmason@ligo.mit.edu>, Rainer Weiss <weiss@ligo.mit.edu>, brian Lantz <blantz@stanford.edu>, Riccardo DeSalvo <desalvo_r@ligo.caltech.edu>

References:<4a6d6be04a4d4cf1a79ec32a432ec60c@ligo.mit.edu>
<6.1.0.6.2.20070210105553.02535b80@acrux.ligo.caltech.edu>

Thanks Dennis this is great. Was cogitating along the same lines but spent the day in the emergency room with Lil (another horseback accident, fortunately not serious). Rai and I have come up with room temperature VP's between $2e-13$ and $1e-15$ for various temperature extrapolations of handbook tables and curves.

I will look at your numbers more carefully but I wonder about two things;

1) I think your expression for the VP over an alloy has to be correct for the liquid, but finite diffusion mobility in the solid has to force surface depletion after some time (maybe this is the "non-ideal deviation" in the iron alloy paper?). For a precipitation-hardening alloy the mobility is by definition low at ambient temperature. I suppose such depletion will be enhanced in the air bake, as long as it does not approach the solution temperature (300 C I think). In any case, more conservatism.

2) On the other hand I'm not sure how I feel about a nm thick layer (actually, I don't know how to calculate the effect of a metallic layer on a dielectric mirror; any thoughts?). Supposing we might still want to insure no line of sight to the HR surfaces, if that is feasible. Fred Dylla suggested plating as a possible means of containment (dunno if this stuff will take electroless nickel or maybe silver?).

For non-line of sight I think other surfaces should adsorb the stuff effectively, especially the cold traps and ion pumps.

BTW I stared at the beast Friday night with Rich and the cross sections look enormous, I would be surprised to see parts at high stress (just scaling, say, the cross-section of the alloy spring mount against the cross-section of the maraging spring element it supports; maybe 25 or 30:1). Interesting to see what the FEA shows.

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

Subject:Re: Zn vapor pressure

Date:Mon, 12 Feb 2007 11:48:24 -0800

From:John Worden <worden_j@ligo-wa.caltech.edu>

To:Dennis Coyne <coyne@ligo.caltech.edu>

CC:Michael Zucker <zucker_m@ligo.mit.edu>, brian Lantz <blantz@stanford.edu>, Ken Mason <kmason@ligo.mit.edu>, Rainer Weiss <weiss@ligo.mit.edu>, John Worden <worden@ligo.caltech.edu>, Riccardo DeSalvo <desalvo_r@ligo.caltech.edu>

References:<4a6d6be04a4d4cf1a79ec32a432ec60c@ligo.mit.edu>
<6.1.0.6.2.20070210105553.02535b80@acrux.ligo.caltech.edu>

Dennis,

Sorry I did not get back to you on Friday - I was at home and not checking voicemails.

My vacuum book "A User's Guide to Vacuum Technology" by John F O'Hanlon has vapor pressure curves which give similar values at 20C, 120C and 300C of $\ll 10^{-11}$ torr, 1×10^{-8} torr, and 2×10^{-3} torr respectively. Note that the chart does not go below 10^{-11} torr.

The reference for the vapor pressure curves is:

"from RCA Review; 30, p285, June 1969, Vapor Pressure Data for the Solid and Liquid elements, by R.E. Honig and D.A. Kramer."

If there are parts which could be mistaken for 6061 does anyone know of a simple test to screen for 7075 prior to heating in our ovens?

I think we have a small stock of 7075 in our machine shop so it is possible that home made parts have already passed through our ovens.

John

On Feb 10, 2007, at 19:54, Dennis Coyne wrote:

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> Mike,
> Thanks for looking up Zn vapor pressure with temperature. The best
> sources that I found for elemental Zn vapor pressure in the
> temperature range of our interest was:
> 1) McKinley & Vance, "The Vapor Pressure of Zinc between 150C and
> 350C", J of Chem Phys, v22, N6, June 1954:
> Log(Po) = -7198/T+9.664, with Po in torr and T in K for the range 150C
> < T < 350C
> So, at 120C (our nominal Aluminum bake temperature) Po = 2E-9 torr and
> at room temperature (22C) it is ~2E-15 torr (by extrapolation). At a
> high bake temperature of say 300C, Po = 1E-3 torr.
>
> 2) Aldred & Pratt, "Vapor Pressures of Zinc, Cadmium, Antimony, and
> Thallium", J of Chem & Engineering Data, v8, n3, July 1963.
> Log(Po) = -6651/T+8.843, with Po in torr and T in K for the range 300C
> < T < 400C
> So, at 120C, Po = 8E-9 torr and at room temperature (22C) it is ~2E-14
> torr (by extrapolation).
> The above Clapeyron-Clausius equations are, of course, consistent with
> your AIP handbook.
>
> On the issue of the vapor pressure of an alloy, the Zn vapor pressure
> over 7075 Aluminum (6% Zn by Wt.) should be lower than the vapor
> pressure over pure Zn. For an ideal solution (alloy) the vapor
> pressure lowering is given by Raoult's law:
> p = x(Zn) * Po, where x(Zn) is the mole fraction of Zn.
> x(Zn) ~ 0.06*Ar(Zn)/Ar(Al) = 0.025, where Ar is the relative atomic
> masses.
> So, p = 5E-17 torr at 22C and 6E-11 torr at 120C.
> Although I could not find a reference for Zn vapor pressure over a
> 7000 series aluminum, I did find a reference for Fe vapor pressure
> over a Vanadium-Fe alloy (Myles & Aldred, "Thermodynamic Properties of
> Solid Vanadium-Iron Alloys", J of Physical Chem, v68, n1, Jan 1964).
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> At ~10% molar fraction, the vapor pressure is in close agreement with
> Raoult's law; The non-ideal deviation further lowered the vapor
> pressure.
>
> On the issue of physical vapor deposition of Zn 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):
> $V = 5.834E-2 * \text{Sqrt}(M/T) * p = \{1.3E-18 \text{ gm/cm}^2/\text{s at 22C and } 1.3E-12$
> $\text{ gm/cm}^2/\text{s at 120C}\}$
> where the molecular mass of Zn, $M = 65.38$. 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
> $rd = V * As / (\text{Pi} * r^2)$
> where As is the source area and r is the distance to the target
> (optic). Assuming a $\sim 1\text{m}^2$ source area at a distance of $\sim 1\text{m}$ from the
> optic, then
> $rd = \{4.0E-19 \text{ gm/cm}^2/\text{s at 22 C and } 4.2E-13 \text{ gm/cm}^2/\text{s at 120C}\}$
> With a Zn density of 7.14 gm/cm^3 , this corresponds to a maximum
> deposition rate of $\{5.6E-20 \text{ cm/s at 22C and } 5.9E-14 \text{ cm/s at 120C}\}$. If
> we assume a maximum tolerable thickness of $\sim 1 \text{ nm}$ (about 1 monolayer),
> then the minimum time to achieve this layer is $\{5.7E4 \text{ yr at 22 C, } 470$
> $\text{ hr at 120C and only } 25 \text{ hr at 150C}\}$. Obviously the vapor deposition
> process is much more complicated, but this simple (and I think
> conservative) analysis gives some comfort.
>
> It seems clear to me that:
> • The use of 7075 Alu in our vacuum system at room temperature is
> acceptable. However, we should prohibit it's use in the future without
> prior approval based on a compelling reason.
> • We should not bake 7075 Alu in a vacuum bake oven; While likely
> not to cause significant contamination at 120C to subsequent loads, if
> the oven has hot spots, or a unplanned excursion to higher
> temperatures, the oven could contaminate subsequent loads.
> • Air baking 7075 Alu at 120C should be acceptable (no risk of
> contaminating the air bake oven due to operation at 1 atm)
> Dennis
>
> At 01:35 PM 2/9/2007, Michael Zucker wrote:
>> Rai straightened me out. According to the AIP Handbook the
>> vapor pressure of elemental zinc is $\sim 1e-3$ torr at 300 C, *not* 300 K
>> as someone recalled.
>> At 295 K the vapor pressure would be more like $1e-14$ torr
>> (extrapolating a thermal exponential from the handbook table).
>> Solution in
>> a stable alloy should further suppress this by orders of magnitude.