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Cavitation-erosion resistance of 316LN stainless steel in mercury containing metallic solutes

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ABSTRACT

Room temperature cavitation tests of vacuum annealed type 316LN stainless steel were performed in pure mercury and in mercury with various amounts of metallic solute to evaluate potential mitigation of erosion/wastage. Tests were performed using an ultrasonic vibratory horn with specimens attached at the tip. All of the solutes examined, which included 5 wt% In, 10 wt% In, 4.4 wt% Cd, 2 wt% Ga, and a mixture that included 1 wt% each of Pb, Sn, and Zn, were found to increase cavitation-erosion as measured by increased weight loss and/or surface profile development compared to exposures for the same conditions in pure mercury. Qualitatively, each solute appeared to increase the tenacity of the post-test wetting of the Hg solutions and render the Hg mixture susceptible to manipulation of droplet shape.

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1. Introduction

The spallation neutron source (SNS) generates neutrons via interaction of a pulsed (60 Hz) 1.0 GeV proton beam with a liquid mercury target. The high energy pulses are expected to give rise to thermal-shock induced pressure waves in the Hg which, after reflection from the container surfaces, will result in negative pressure transients and cavitation in the target liquid [1,2]. Some of the energy released during the collapse of the cavitation bubbles will be manifested in a jetting action of liquid at extreme velocity that can potentially erode the adjacent containment material.

Based on a favorable combination of factors, including resistance to corrosion by Hg, well-characterized behavior in a neutron radiation environment, and retention of acceptable ductility after irradiation to moderate displacement doses, 316LN stainless steel was selected as the target container material [3]. However, previous tests have indicated that annealed 316LN is susceptible to potentially significant pitting and erosion damage resulting from cavitation in Hg using a split Hopkinson pressure bar (SHPB) apparatus [4], in-beam exposures [5,6], and a vibratory horn [7–11]. Of particular concern is the observation that pit depths resulting from relatively brief SHPB and in-beam exposures (order of 10–200 cavitation pulses/events), if linearly extrapolated over the anticipated target service life of several hundred million pulses, would prematurely threaten the integrity of the target containment.

It has been shown that the cavitation-erosion resistance of annealed 316LN may be improved by hardening the material via surface treatments and/or cold-working [10,11]. Further, cavitation-erosion resistance of alternate materials may be somewhat superior to 316LN [10], but these alternate materials may exhibit less desirable or less well known properties for other aspects of the target service requirements.

It has also been noted that dissolving a significant amount of metallic elements into Hg leads to changes in surface tension characteristics of the liquid. In one particular experiment [12], it was observed that a 4 wt% addition of alloying elements (primarly 1% each of Pb, Sn, and Zn along with smaller amounts of other elements) increased the surface tension about 35% compared to pure Hg. Further, droplets of the modified Hg formed at the end of a capillary tube were observed to become much larger and to stretch longer prior to breaking compared to the pure Hg counterparts, and the authors suggested the alloyed liquid appeared to be on the path to semi-solid behavior. If this would be the case, cavitation behavior in highly alloyed Hg might be expected to be significantly different than that in pure Hg, particularly if the apparently semi-solid behavior of the alloyed Hg caused cavitation bubbles to collapse at lower velocities, thereby imparting less potential erosion damage to nearby containment surfaces.

In contrast to previous work [10,11], which examined cavitation-erosion in Hg as a function of the physical properties of potential containment materials and various surface treatments, the concept under investigation here considers Hg composition as a potential variable to mitigate cavitation-erosion damage in a given structural material. While there are no doubt practical issues associated with alteration of the composition and fluid properties of the target Hg (such as pumping characteristics, neutron performance, and isotope generation/waste handling), the purpose of this laboratory effort was to compare cavitation damage on 316LN produced by pure Hg with that produced by Hg with various alloying additions.

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2. Experimental

Binary phase diagrams [13] for Hg in combination with all other elements for which data were available were examined in the search for readily soluble alloying additions to Hg. Diagrams were located for Hg in combination with 74 different elements (although a few of the diagrams were calculated rather than observed/measured). Among these 74 elements, ambient temperature solubility in Hg was essentially nil for a large number of them and at least 1 wt% for only eleven elements. Of these eleven, three (Cs, Rb, and Tl) were dropped from further consideration in these screening tests due to handling dangers in the pure state – primarily the potential for explosive oxidation. Of the remaining eight soluble elements, Bi and Mg were deleted from the list of potential solutes because lab experiments suggested they oxidized too readily and presented very sluggish dissolution kinetics. The remaining six elements appear in Table 1 along with other information about each potential solute.

It has been observed [12,14–16] that metals dissolved in Hg tend to have an extremely high activity in the atomic state and are therefore relatively reactive with even trace oxygen in the environment. As a result, oxides of the solute metal tend to form quickly as a scum on the surface of the host Hg, and these oxides may also catalyze the oxidation of Hg itself. This effect is very readily apparent visually, as nominally clean/pure Hg has a very shiny silver surface whereas alloyed Hg exposed to air exhibits a dull gray surface layer that appears to encase the Hg. The scum may be somewhat self-limiting in that its formation slows or precludes further reaction with air unless regularly disturbed (e.g., by stirring). In the absence of air (for example, within inert gas chambers), the oxidation reaction is drastically retarded and perhaps even eliminated on a practical time scale. To minimize the oxidation of alloying elements in Hg, the test vessel used in these experiments included a feature permitting the surface of the Hg to be flooded with inert gas during solution preparation and testing.

In addition to the cover gas requirement, a test vessel with a relatively small volume of Hg was deemed useful in order to limit the total amount of solute necessary to make the test solutions. Previous cavitation tests in this laboratory utilized ~750 ml pure Hg within a wide-mouth stainless steel dewar, but a glass vessel with a working volume of ~50 ml was fabricated for these experiments. Due to the limited volume of Hg in the test vessel, specimen surfaces were immersed only to a depth of approximately 2 mm for the test exposures. (Immersion depth and solution volume are variables with some influence on the quantitative results of cavitation tests – see Ref. [16] for additional details).

Because of the intense energy/work associated with a vibratory horn cavitation test, the solution test temperature tends to increase during sonication, particularly within a limited solution volume. To maintain a constant temperature of the Hg bath during testing, the glass test vessel was fabricated with a double wall to permit circulation of coolant around the Hg bath to maintain a constant Hg test temperature of 30–31 °C. Taking into consideration the desire for an inert gas cover, reduced volume, and cooling capability, the vessel and test arrangement schematically depicted in Fig. 1 was used for all the tests presented here.

All cavitation-erosion tests were performed using a titanium vibratory horn and the general test methodology described in ASTM G-32 [17]. The working face of each test specimen had a surface area of 180 mm^2, and the specimen was attached to the vibratory horn via a threaded shank. Photos of the vibratory horn and representative specimens are given in Ref. [10]. The horn tip oscillated at a fixed frequency (20 kHz) and was set to generate a peak-to-peak vibrational amplitude of approximately 25 μm. The rapid reciprocating displacement induces the formation and collapse of cavities in the liquid near the specimen surface, and cavitation-erosion damage from collapsing cavities can be quantified by measurement of specimen weight change and/or erosion depth as a function of exposure time. The baseline test condition included immersion of the working surface of the specimen to a depth of about 2 mm in approximately 30 ml of Hg, and a Hg test temperature maintained at approximately 30 °C.

Nominally pure Hg (filtered through cheesecloth until the luster associated with the Hg surface was very bright/shiny; chemical analysis detected only ~85 ppb Ag and ~100 ppb Si above detection limits) was used for baseline testing and for alloying. Solutes examined in this set of screening tests included pure Hg with 5 wt% In, 10 wt% In, 4.4 wt% Cd (solubility limit), 2% Ga (solubility limit), and a mixture – following the example in Ref. [12] – that included 1 wt% each of Zn, Pb, and Sn. Other details associated with preparation of Hg solutions appear elsewhere [16].

The test specimens were machined from a block of 316LN stainless steel that was also used as a source for specimens in previous testing [10,11,16]. The composition of the 316LN is given in Table 2. All specimens were machined from the original cross-rolled plate material such that the few inclusions present were oriented parallel to the test face. Following machining and light sanding of the test surface on 800 grit paper, the specimens were loosely wrapped in Ta-foil and vacuum annealed at 1020 °C at 10^{-4} Pa (10^{-6} torr) or less for 1 h, followed by cooling to less than 300 °C in about 1 h.

Post-exposure cleaning of the specimens consisted of ultrasonic cleaning sequentially in (1) an aqueous solution containing dissolved sulfur species to chemically bind Hg, (2) distilled water, and (3) reagent grade acetone, followed by forced air drying. After cleaning, specimens were weighed and examined with an optical microscope to determine the average cavitation-erosion profile

Table 1
Potential solutes for Hg in cavitation-erosion testing

Element	Ambient solubility in Hg (wt%) [13]	Relative dissolution kinetics[a]
Zn	~1	Sluggish
Ga	~2	Rapid
Cd	~5	Rapid
In	~52	Very rapid
Sn	~1	Slow
Pb	~1	Slow

[a] $T \leqslant 30$ °C, modest agitation, argon cover gas.

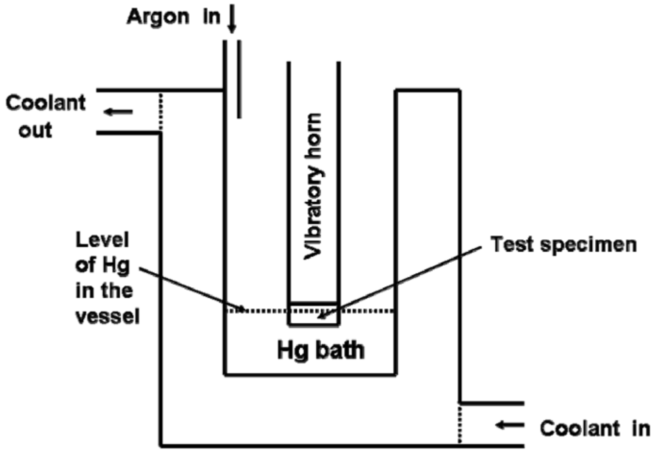

Fig. 1. Schematic drawing of the glass vessel used to contain the Hg in the present experiments. The specimen is on the tip of the vibratory horn with the test surface submerged about 2 mm into the Hg bath. Actual dimensions include an internal vessel diameter of 5 cm and a 1 cm gap between the bottom of the Hg containment and the test specimen surface.

Table 2
Composition of master heat of 316LN stainless steel from certified mill report

Element	wt%	Element	wt%
C	0.009	Cr	16.31
Mn	1.75	Ni	10.20
P	0.029	Mo	2.07
S	0.002	Co	0.16
Si	0.39	Cu	0.23
N	0.11	Fe	Balance

(utilizing the calibrated fine focus feature on at least seven random but regularly spaced fields of view at 400×) and assess potential pitting. In addition, selected specimens were also examined with the scanning electron microscope.

3. Results and discussion

3.1. Data interpretation

The evaluation reported here is intended primarily to compare cavitation-erosion damage among identical specimens in pure Hg and in modified Hg for a fixed test configuration. As such, it is a screening test only. In particular, the reader should recognize there is no precise correlation between the damage rate/intensity produced at the tip of the vibratory horn and at the surface of the Hg target container at the SNS target facility. Other details of data interpretation – generally less significant to the present investigation – appear in Refs. [10,11].

3.2. Testing in pure mercury

A series of three 1-h sonication exposures was performed on four identically prepared specimens of vacuum annealed 316LN stainless steel in pure Hg. The weight loss results for annealed 316LN in the baseline test conditions are shown in Fig. 2.

Consistent with previous data [10,11], the weight change as a function of time typically extrapolates to zero weight change at a slightly positive exposure time (rather than zero). This can be generally explained by considering the cavitation-erosion process to require an incubation time in which micro-cracks must be initiated and coalesce/propagate prior to resulting in bulk material loss [7,18]. As a result, it is common for mass loss in the initial exposure period to be somewhat lower than in subsequent exposure periods of similar duration, with an approximately constant mass loss developing after the initial hour or two of exposure [10,11].

Following sonication, the specimen surfaces exhibited crudely hemispherical craters that may or may not partially overlap with other nearby craters. Fig. 3 is representative of this observation among specimens in this investigation. Examination at higher magnification (series of scanning electron micrographs in Fig. 4) reveals that the cavitation-erosion process apparently involves metal removal in small, progressive stages that has some similarities to micro-cracking/tearing on a small scale. While the spacing of craters was relatively uniform over the specimen surface, it is clear that some feature of the cavitation process tends to focus damage such that crater formation rather than completely uniform removal of material is encouraged. Similarly, Young and Johnston [9] noted that cavitation damage in some liquid metals (e.g., Na) tends toward very general surface attrition while damage in Hg, like that observed here, tends toward formation and deepening of individual craters.

The surface profile measurement is essentially an assessment of the average depth (rim to bottom) of the craters across the specimen surface. Neither the diameter of the craters, their position relative to the original specimen surface, nor the fraction of the surface represented by craters, is assessed by the profile measurement – these factors would tend to present themselves in the mass loss measurement. Increased profile depth generally correlates with increased mass loss for these specimens, but there are minor exceptions among closely spaced data points. As a rule, the surfaces of the specimens tested in baseline conditions were remarkably uniform in that the average profile was very consistent across the entire specimen. However, as exposure time increased, each specimen exposed in pure Hg developed some (as few as two, as many as six) scattered 'pits' on the surface. This pattern has been observed in prior testing [9–11] and, like the previous experience, the 'pits' observed here were generally hemispherical but wider and deeper (both by a factor of 1.5–3 times) than the nominal crater dimension following 3 h of sonication. The mechanism of formation of these 'pits' is not clear, but it is possible that they are related to overlapping craters, the presence of inclusions

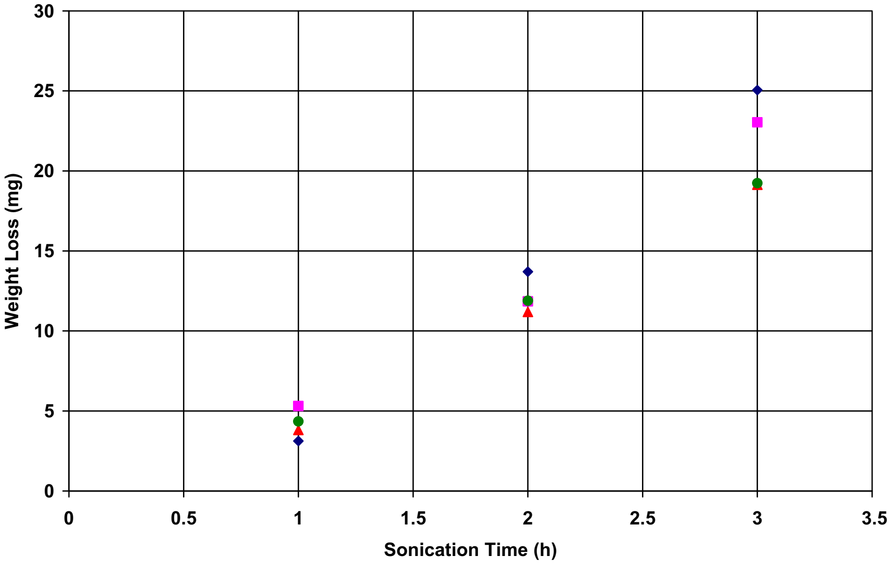

Fig. 2. Weight loss of annealed 316LN specimens as a function of sonication time. Note that in some cases, the data points representing the four individual specimens overlap sufficiently to prohibit clear distinction.

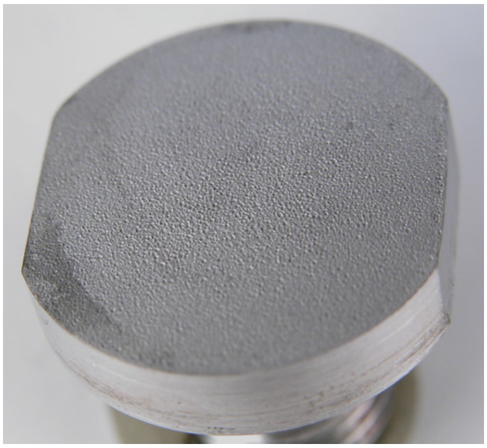

Fig. 3. Vacuum-annealed 316LN stainless steel cavitation specimen following three hour sonication in pure Hg at room temperature. The actual-size diameter of the specimen is 16 mm.

intersecting the exposed surface, or rare/random Hg wave conditions in which constructive interference generates an unusually large cavitation pulse.

The average surface profile for annealed 316LN as a function of sonication time in pure Hg is given in Fig. 5. Notice that the intercept of the profile trend line is greater than zero. Interpretation is hampered by a lack of very short-time exposure data, but the positive intercept implies localized erosion damage of susceptible areas occurs very quickly. This explanation is consistent with previous results [4–6,18] indicating the development of localized surface roughness and shallow pits after a very few pressure cycles. The positive intercept also implies that there is a minimum size/profile of surface damage associated with the onset of measurable weight loss, suggesting a minimum damage extent related to some aspect of the test material (e.g., grain size or sub-cell size) and/or a property of the cavitating fluid.

The post-test appearance of the pure Hg in all cases included the slow accumulation of a dark gray/green or black scum floating on the top that appeared to be test debris. Such material accumulates on the Hg surface independent of the test material (observed for sonication of 316LN as well as other materials [10,11]), and is easily skimmed off the surface with cheesecloth. This material has not been analytically identified, but it seems likely that it represents the interaction of Hg and air on the material removed from the specimen surface during cavitation-erosion. A similar looking material was found floating on the post-test Hg following an extended thermal convection loop test of 316L stainless steel at the outset of the SNS compatibility studies, and the dark green/black scum was found to be primarily amorphous but containing Fe, Cr, Ni (the main components of stainless steel) along with Hg and O [15].

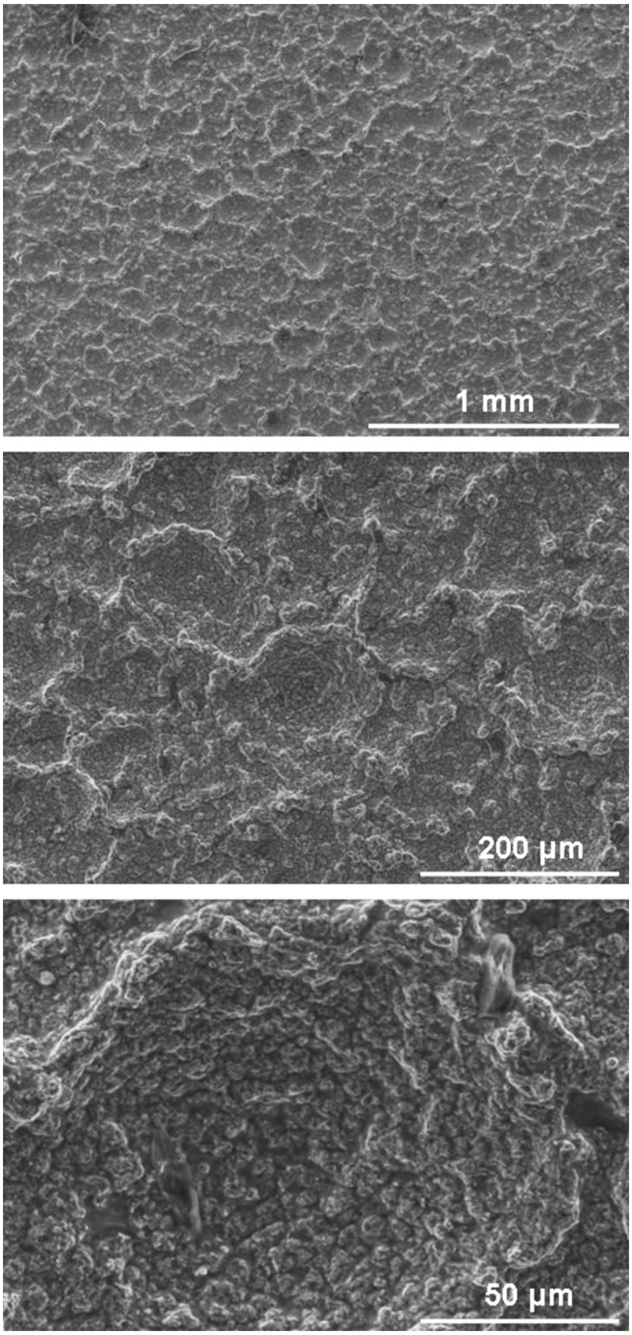

Fig. 4. Backscattered electron images of the cavitation-erosion damage observed on annealed 316LN specimens following three hours of sonication in pure Hg at room temperature. Magnification increases in sequence from the top photo to the bottom photo.

3.3. Addition of In to Hg

Indium is by far the most soluble element in mercury at near-ambient temperature. A roughly rectangular piece of In (cut from a cast ingot, sized for the desired mass) was added to the Hg within the glass test vessel under argon cover gas flow. The Hg quickly and completely wetted the In, and appeared to pull the solid In down into the liquid via the surface tension associated with complete wetting. Within 2–3 min at room temperature, the Hg had completely dissolved the requisite amounts to generate solutions with 5 wt% and 10 wt% In.

Upon dissolution, the surface luster of the mixture immediately changed from the shiny metallic appearance of the pure Hg to a somewhat more dull/flat silver. In addition, the liquid seemed almost 'encased' by a thin, pliable silver/gray scum that moved readily with the liquid. Unlike pure Hg, which can be poured from a glass or plastic bottle with no residual scum, or 'tailing' [12], adhering to the container, the Hg–In mixtures tended to leave a small (compared to the mass of In added) amount of scum on the bottom/side surfaces of containers when the remainder was poured off. Aggressive stirring prior to pouring seemed to minimize the amount of tailing left behind, but it is impractically difficult to completely eliminate the residual material.

Consistent with the observations of Wilkinson [12], the alloyed Hg seemed to take on properties similar to a semi-solid or a gel in

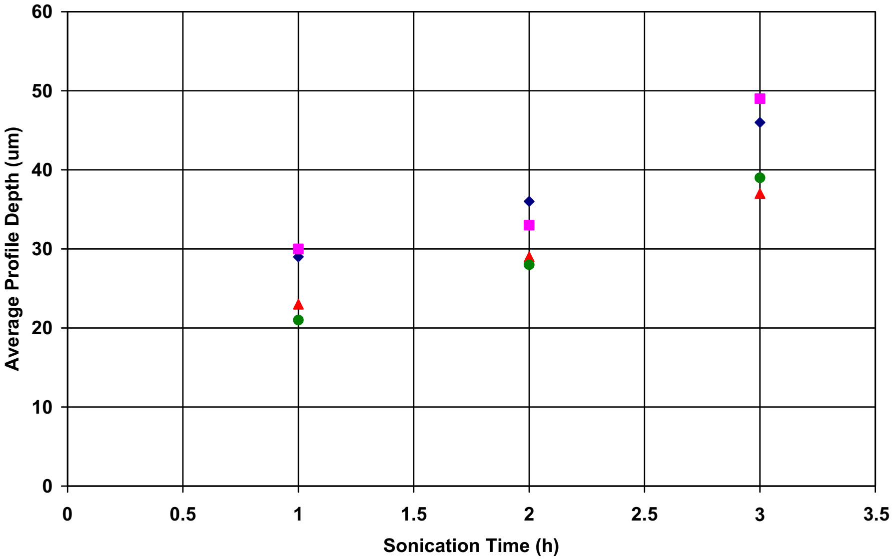

Fig. 5. Surface profile development for annealed 316LN specimens as a function of sonication time in pure Hg.

Fig. 6. Bead of pure Hg on the left assumes and maintains a roughly hemispherical shape despite probing with a plastic rod, while the same size bead of Hg–10 wt% In on the right has its shape readily manipulated. A plastic rod was dragged through the center of each bead just prior to this photograph. Actual bead size in each case was approximately 15 mm diameter.

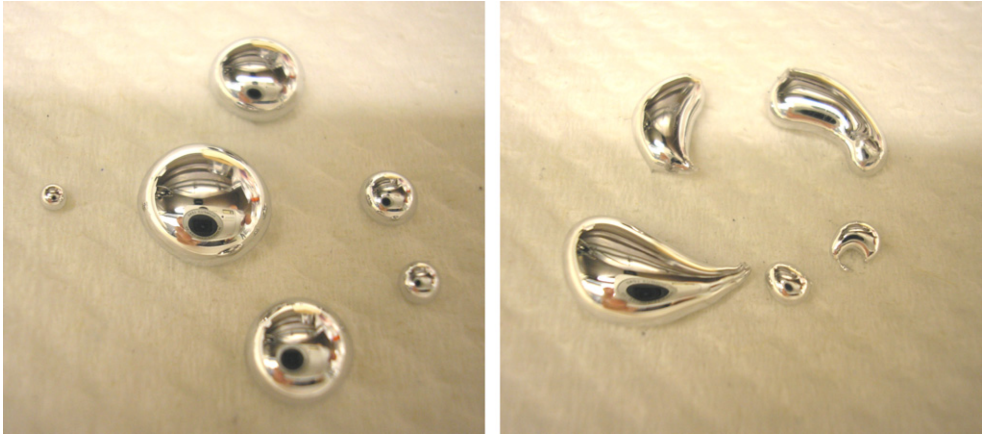

Fig. 7. Small beads of pure Hg on the left resist manipulation and maintain roughly spherical shapes, while the same size beads of Hg–10 wt% In on the right can be readily shaped. Actual diameter of beads ranged from 2 to 10 mm.

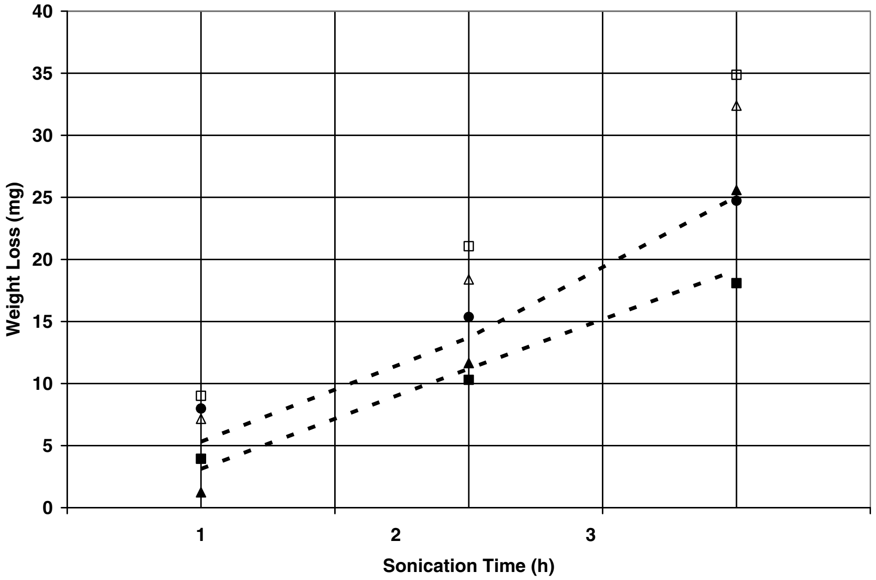

Fig. 8. Weight loss as a function of sonication time for annealed 316LN in pure Hg (range of data bounded by dashed lines) compared to Hg–5 wt% In (solid data points) and Hg–10 wt% In (open data points).

that the droplets could be elongated slightly upon dispensing from a small diameter nozzle and modest-size droplets could be shaped and segmented easily, which is all but impossible for droplets of pure Hg. Figs. 6 and 7, comparing pure Hg beads with Hg–10 wt% In beads, are representative of this behavior which was observed for all alloyed Hg in this investigation. The authors suspect that the bead-shaping behavior is not a bulk property of the Hg alloy but is due to the surface oxidation reaction that leaves the exposed surface of the Hg alloy bead rich in metallic oxide species, and the relative 'stiffness' of this layer promotes the shaping of the droplets. Indirect evidence supporting this concept includes the observation that a syringe inserted into the beads of alloyed Hg and used to remove fluid from the center of the bead leaves a 'skin' of silver/gray residual material – not readily pulled into the syringe – that appears not unlike a deflated balloon. The sub-surface Hg within the bead may indeed have a higher surface tension than pure Hg due to the alloying, as measured by Wilkinson [12], but surface tension measurements were not carried out in this work. If the surface tension indeed increased as a result of alloying, cavitation-erosion damage might be expected to increase. The reason is that increased surface tension implies an increased driving force to collapse a cavitation bubble, and the increased force might be expected to increase the velocity/force associated with the jetting action of liquid that results.

Weight change as a function of exposure time for the cavitation specimens in Hg with 5 wt% In and 10 wt% In compared to pure Hg is shown in Fig. 8. The data indicate that 5 wt% In generated cavitation-erosion wastage very similar to pure Hg (weight change data falls more-or-less within scatter band for the pure Hg data), while cavitation in 10 wt% In was decidedly more aggressive in terms of weight loss. In particular, note that the apparent incubation time for weight loss seems to be reduced for Hg–10 wt% In,

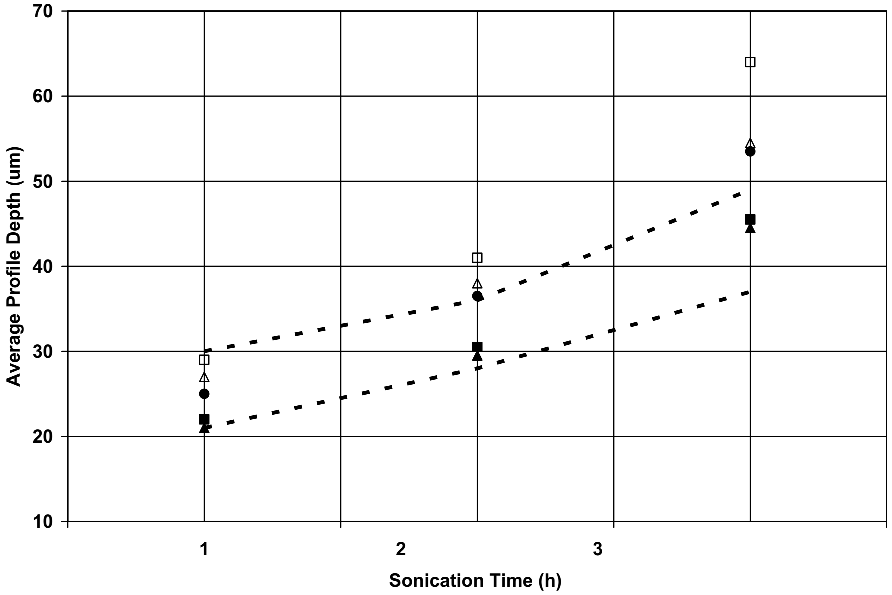

Fig. 9. Average profile depth as a function of sonication time for annealed 316LN in pure Hg (range of data bounded by dashed lines) compared to Hg–5 wt% In (solid data points) and Hg–10 wt% In (open data points).

and at extended exposure times the rate of weight change is also somewhat higher for the Hg–10 wt% In mixture compared to pure Hg. Consistent with the weight change data, the development of the average profile shown in Fig. 9 also suggests that Hg–10 wt% In is somewhat more aggressive than pure Hg.

While not quantitatively assessed, the addition of In to the Hg also seemed to make for more tenacious post-test wetting of the specimen. Increased wetting was evidenced by the need for longer post-test cleaning times in the Hg removal step. Representative appearance of the cleaned post-test specimens exposed to Hg + 10 wt% In is given in Fig. 10. Comparison with Fig. 4 reveals that the cavitation-erosion damage process is very similar in pure Hg and alloyed Hg, but the damage profile (depth of the craters) is more defined in the alloyed Hg for the same exposure time. It is also interesting that very few of the 'pits' observed on the specimens exposed to pure Hg were observed on specimens exposed to Hg alloyed with In; of the five total specimens exposed in mixtures of Hg + In, three had no pits and another exhibited only one pit. When the pits were present, however, they were indistinguishable from those observed on other specimens of 316LN exposed in pure Hg.

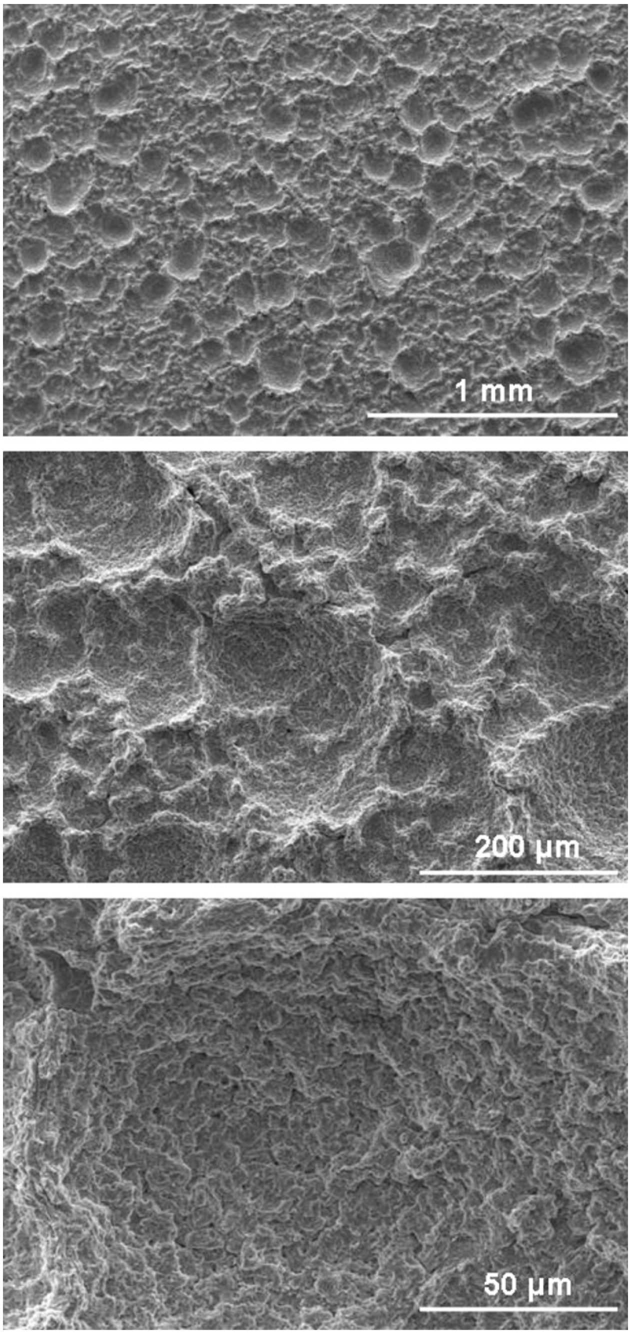

Fig. 10. Backscattered electron images of the cavitation damage observed on annealed 316LN specimens following three hours sonication in Hg–10 wt% In at room temperature.

Table 3
Qualitative results of sonication tests of annealed 316LN in pure Hg compared to other Hg compositions examined in this study

Hg composition	Weight loss result compared to pure Hg	Surface profile result compared to pure Hg
5 wt% In	~Unchanged	~Unchanged
10 wt% In	~50% increase	~50% increase
4.4 wt% Cd	~15% increase	~40% increase
2.0 wt% Ga	Slight decrease	~25% increase
1 wt% each Pb/Sn/Zn	~50% increase	~50% increase

3.4. Other alloying additions to Hg

Alloying additions of 4.4 wt% Cd, 2 wt% Ga, and a combination of 1 wt% each of Pb, Sn, and Zn were also investigated. In summary, each solution was found to increase either the weight loss or the depth of the surface profile (or both) compared to testing in pure Hg. Details associated with the preparation of Hg mixtures and the test results can be found in Ref. [16], and Table 3 contains generalized results from these cavitation tests for ease of comparison.

4. Conclusions

Various metallic solutes were added to mercury in an attempt to change the properties of the resulting liquid metal cavitation medium in a way that might reduce cavitation-erosion damage on annealed 316LN stainless steel. Specimen weight change and profile development as a function of exposure time was compiled for fixed test conditions and compared as a function of the composition of the Hg test medium. Test compositions included pure Hg as well as Hg containing: (a) 5 wt% In, (b) 10 wt% In, (c) 4.4 wt% Cd, (d) 2 wt% Ga, and (e) 1 wt% each of Pb, Sn, and Zn added together.

Compared to pure Hg, mixtures with 10 wt% In and the combination of 1 wt% each of Pb–Sn–Zn significantly increased cavitation-erosion damage as evidenced by significantly greater weight changes and surface profiles – both larger by ~50% – and resulted in a greater rate of change following 3 h of exposure. Solutions including 5 wt% In and 4.4 wt% Cd resulted in weight changes only slightly greater on average than those observed in pure Hg, but both produced deeper surface profiles, by about 15% and 40%, respectively, following three hours sonication. Only the solution with 2 wt% Ga exhibited a slightly lower weight loss than that observed in pure Hg after 3 h exposures, but the depth of the surface profile of the specimens so exposed was somewhat greater than for specimens in pure Hg. Based on observation of the cleaned post-test surfaces at a range of magnifications in the scanning electron microscope, the mechanics of cavitation-erosion appear to be the same for pure Hg and alloyed Hg, but perhaps more intense in the latter.

It was observed that alloyed Hg droplets could be shaped and manipulated, suggesting semi-solid properties and perhaps a liquid in which cavitation bubbles would collapse with less intensity than in pure Hg. However, for the conditions and solutes examined here, alloying of Hg did not mitigate cavitation-erosion damage on annealed 316LN stainless steel and, in fact, seemed to intensify damage for several Hg alloy compositions. It appears that the semi-solid properties of the Hg are relegated to the near surface regions of the fluid, caused by a high fraction of oxides in the surface layer, and that increased surface tension of the bulk (sub-surface) fluid apparently dominates the cavitation-erosion behavior as evidenced by greater cavitation-erosion damage (due to increased intensity of bubble collapse). A potential area of interest for future work includes study of the properties of the oxide-rich layer of alloyed Hg exposed to air and whether this semi-solid, wetted onto the test surface of interest, could mitigate cavitation-erosion damage.

Acknowledgements

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