SURFACE DECORATION OF STAINLESS STEEL FOR LBE FLOW MEASUREMENT BY ULTRASONIC TECHNIQUES

Kenji Kikuchi*, Masao Tezuka**, Shigeru Saito*, Hiroyuki Oigawa*, Yasushi Takeda***

* Japan Atomic Energy Research Institute, Tokai-mura, Ibaraki-ken 319-1195 Japan, E-mail: kikuchi@popsvr.tokai.jaeri.go.jp
** Sukegawa Electric Co., Ltd., Hitachi-shi, Ibaraki-ken, 317-0051, Japan
*** Hokkaido University, Sapporo-shi, Hokkaido, 060-8628, Japan

ABSTRACT

Ultrasonic techniques were applied to lead bismuth eutectic (LBE). LBE melts at 124.5ºC. Below 400ºC the austenitic stainless steel is not easily corroded in a saturated oxygen content and LBE often adheres on the steel. However, adhered LBE can be removed by blushing easily. So LBE was not bonded strongly to the steel. When the steel is submerged into LBE, LBE will contact with the steel except for the interface among LBE, gas and metal where the surface energy controls the shape of the free surface in LBE. It is supposed that LBE will transmit ultrasonic wave into LBE through the contacting area. However, the ultrasonic echo was too low to detect from the steel container filled with LBE. The measurement was improved by coating the interface between the steel and LBE with the SnPb solder. After an immersion test the steel surface was covered with thin LBE layer. The thickness of the layer is only 10 to 20 µm. So it will not disturb the flow pattern where UVP is applied. Sn was not detected by X ray analyses. This is an evidence how the steel was wetted in LBE and how the ultrasonic wave transmitted thougl the interface of LBE and the steel.

Keywords: LBE, Ultrasonic wave, Wetting, Austenitic stainless steel, Solder, X ray analyses

INTRODUCTION

Lead bismuth eutectic (LBE) is a potential target for spallation neutron source as well as a coolant material of the sub-critical fast reactor in the concept of the nuclear transmutation system driven by the high-energy proton accelerator.[1] In the R&D of the target system it has been found that a local flow affects the corrosion-erosion rate of the materials and the thermo-mechanical design of the target window where the incident protons bombard. For example, the projected electrode of electro-magnetic flow meter in the flow channel was eroded under the flowing LBE. Material samples for corrosion test were eroded locally. Dissolved elements of the structural material into LBE at the hot leg deposit in the form of polycrystals at the cold leg as a result of mass transfer mechanism in the closed channel.[2] The target window disturbs LBE flow and heat transfer is deteriorated. Ultrasonic method is expected to be a powerful tool to visualize a flow pattern in the particular area of LBE components in order to optimize the flow distributions. It can reveal the flow pattern in the other application such as the liquid mercury.[3] So we applied this technique to LBE.

There are technical issues to be considered the ultrasonic method techniques applied to LBE. It is a boundary property on the ultrasonic wave penetration between the vessel and LBE. Firstly, working temperature should be higher than 124.5ºC, melting point of 45Pb-55Bi(w%) eutectic alloy. Oxygen has a very important role for handling LBE at high temperature. Oxygen dissolves into LBE. Dissolved oxygen makes Fe oxide on the surface of the steel. Saturated oxide in LBE reacts with Pb and makes PbO.[4] It is inevitable that Pb will be oxidized at high temperature without control of the oxygen content in LBE. Though the corrosion test of the austenitic stainless steel the oxide is too small to detect under 400ºC. But over 450ºC the stainless steel was corrosion-eroded and the oxide layer was made.[5] The surface of stainless steel was covered with solidified LBE even after draining out of the container over 450ºC. Secondly, wetting property may affect the measuring efficiency by the ultrasonic method. If the LBE container is not wet, supersonic sound will be reflected at the boundary between the metal container and LBE. Then the echo signal from the LBE will not be given.

EXPERIMENTAL METHOD

Fig.1 illustrates the ultrasonic echo measurement in order to know the intensity of the reflected ultrasonic wave at the interface of the vessel and materials and/or reflector in LBE. Materials used are water and LBE. Test temperatures are RT for water and 140ºC for LBE, respectively, both in air. The reputation frequency of ultrasonic probe used is 4MHz, type TH4-5-8 [6]. The ultrasonic probe is attached to the cylindrical container with 80mm diameter made of the austenitic stainless steel or acrylic resin with using a matching medium. A reflector plate made of stainless steel is set up in LBE in order to reflect the ultrasonic waves. Several types of the vessel are used. The vessel surface is round because of a part of cylinder and an edge surface of the ultrasonic probe is flat. In order to avoid mismatching of the face-to-face contact, one vessel surface is flattened by machine process at the outer side surface in order to examine the contacting quality. But the internal surface is still round. Ni coated the internal surface of one vessel. Ni is expected to cover the surface roughness after the machining of manufacturing process and to avoid the surface from the oxidation. The internal surface of another vessel was coated by Sn-Pb solder.

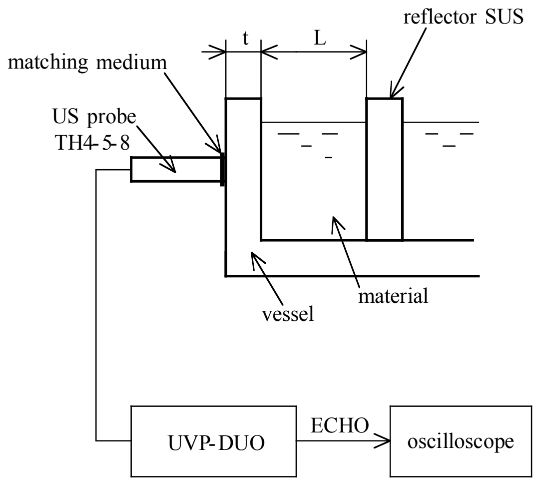

Fig.1 Illustration of ultrasonic echo measurement.

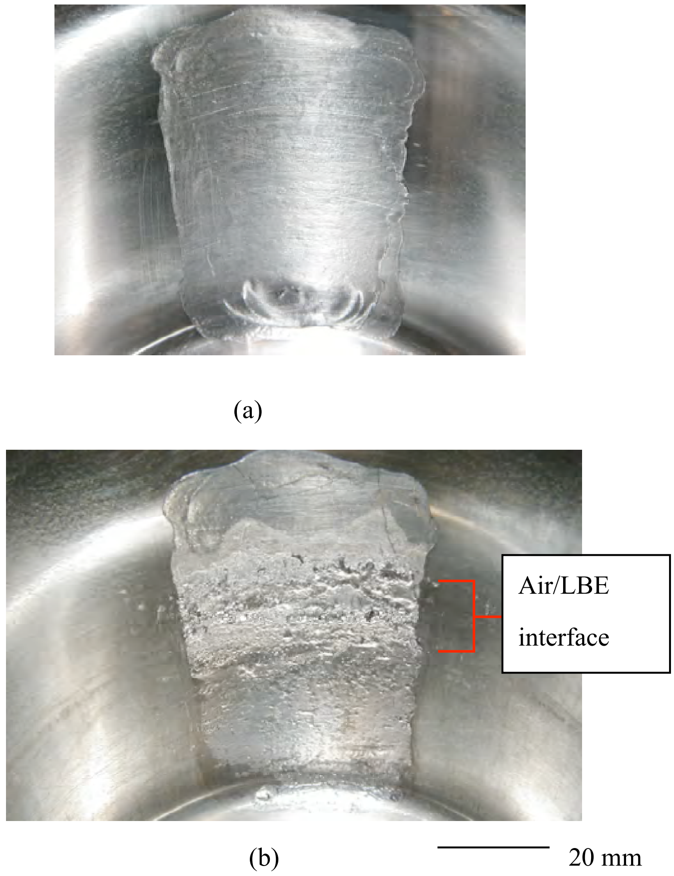

Fig. 2 Optical microscope of internal surface of the vessel (ID8). The vessel diameter is 8 cm. (a): Image before est, (b): Image after test.

RESULTS

Table 1 summarizes the results of the echo intensity measurement. The echo intensity E(x) for IDX (X=2 to 8) is defined by the relative value to the reflected wave height at ID1. In the definition it is also considered that the wave height is proportional to the input voltage.

E(x)=20Log[wave height at IDX / wave height at ID1]

The echo intensity from the vessel without any treatment and the reflector plate in LBE was too low to measure (ID5). On the other hand, when the supersonic probe was directly submerged into LBE from the top free surface (ID4) in Fig. 1, the high intensity of the echo signal was detected from the bottom plate. Then the probe was attached to the flattened part of the vessel, but the echo signal was still too low to detect (ID6). Ni coated vessel also did not help obtain the echo signal (ID7). The echo signal was high enough to measure only in the solder coated vessel (ID8). At a time of sixty hours melting LBE in the vessel the echo signal was measured sufficiently. In the process there are twelve cycles of LBE processing. LBE was melted, kept 140oC during five hours and then solidified. For water the echo intensity was the highest among the measurements when it directly submerged into water(ID1). When the measurement was done from outside the acrylic resin (ID2), the echo intensity is still very high. However, the wave height became small from the outside of stainless steel (ID3).

Table 1 Echo intensity of ultrasonic waves

ID	Vessel	Material	Echo intensity, dB	Wave height	Input voltage,V	Temperature,℃	Echo detection
1	None	Water	0	1.8	30	RT	Good
2	Acrylic resin	Water	-3.5	1.2	30	RT	Good
3	SUS	Water	-23.5	0.12	30	RT	Bad
4	None	LBE	-8.5	0.7	30	140	Good
5	SUS	LBE	-	0	150	140	Bad
6	SUS (flattened)	LBE	-39.1	0.1	150	140	Bad
7	SUS(Ni coating)	LBE	-39.1	0.1	150	140	Bad
8	SUS(Solver coating)	LBE	-25.1	0.5	150	140	Good

Note: Wave height is proportional to input voltage.

Fig.2 shows the optical microscope images of the internal surface of the vessel (ID8) before and after 60 hrs exposures to the melting LBE. SnPb solver was coated partly in the vessel (a). Coated material still existed. Geometrical feature of coated area looks same in both images. The surface became rough, especially, at the interface of air and LBE. It should be noted that the echo intensity was high at the coated area but too low to detect at the uncoated area as experienced inID5. Uncoated area was not wetted by LBE.

DISCUSSION

At relatively low temperatures where the austenitic stainless steel is not corroded, ultrasonic method cannot be applied for measurement of LBF flow because the echo intensity will be too low to measure. However, the surface of the vessel is decorated by SnPb solver, this techniques will be applied. Question is why? Key issue is the wetting property between LBE and the vessel material. The wetting property is related to the surface energies at the contacting area ob LBE. If the steel surface is not wetted, the gaseous layer may exist between LBE and the steel where ultrasonic waves will not transmit to LBE because of the different impedance of the material. Although after the test (ID8) there was material remained on the wall as shown in Fig.2 (b) there was no evidence with regards to the wetting property in LBE.

So we did test to examine a change of surface state of coated solver and LBE by submerging the reflector plates into LBE for sixty hours. Three plates made of SUS316 were prepared. One is a standard sample without any use in LBE. The other two specimens were submerged into LBE during 10 and 60 hrs, respectively. Fi.3 shows the optical microscope images of three plates. Figs.3 (a), (b) and (c) show the plates coated with the SnPb solver. SnPb solver was coated on the three plates in the same manner. Fig.3 (d) shows the plate (b) submerged into LBE during 10 hours. Fig 3(e) shows the plate (c) submerged into LBE during 60 hours. Surface morphologies of (d) and (e) look different from their originated images of (b) and (c), respectively. The cross sections of the plates were examined by the X-ray analyzer. It was found that Sn disappeared in the plates of (d) and (e) but Bi was detected in the remained materials on the plates as shown in Fig.4. The melting point of Sn Pb eutectic and LBE are 183°C and 124.5 °C, respectively. It is, however, thought that bismuth diffuses into coated SnPb and makes LBE in the solidified material on the plates. Optical microscope observation of the plate cross-section showed that thickness of coated layer was 500mm but reduced to 10 to 20 μm after immersion in LBE. Fig.5 showed the photos of the cross section. The thickness of the coated materials on the plates was 500 mm. After immersion in LBE it reduced to 10mm by 60 hrs. Conclusively the coated material is so wetted that the supersonic sound can be transmitted at the interface of the steel and LBE.

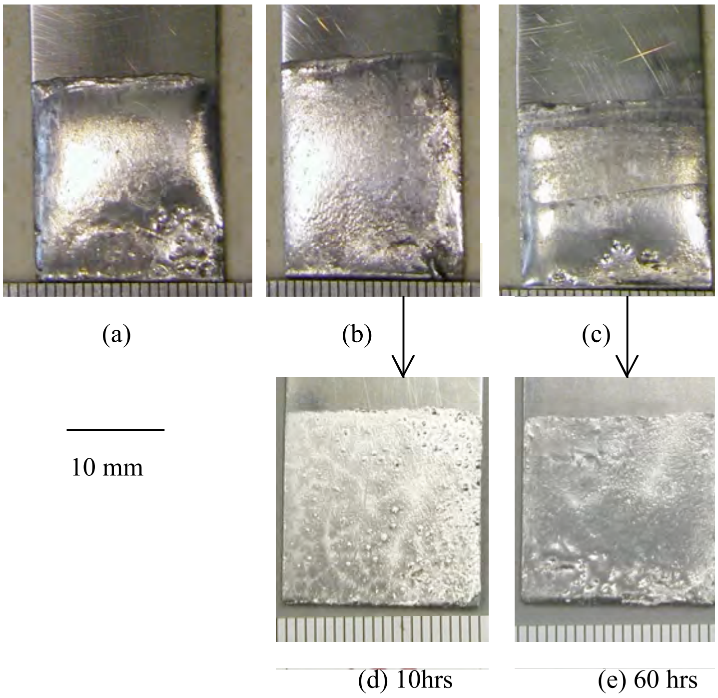

Fig.3 Submerged test of coated plates into LBE.

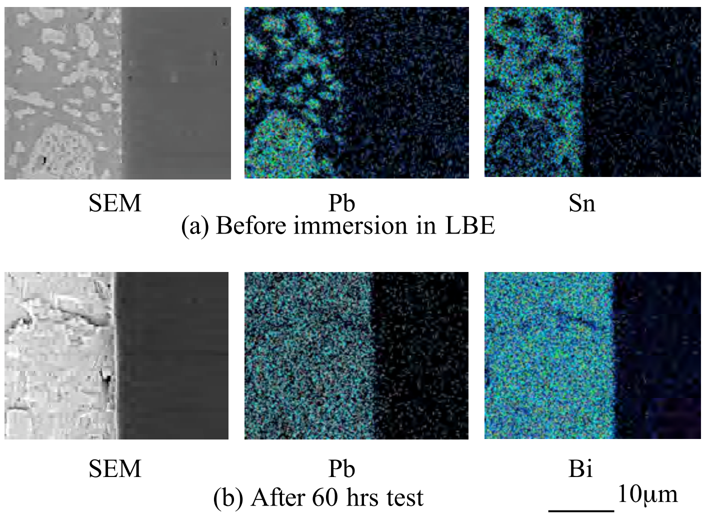

Fig.4 X-ray analyses of cross-section of coated SUS 316.

CONCLUDING REMARKS

The ultrasonic wave property in the steeland LBE was examined at 140ºC in air. SnPb solder makes possible to detect the sonic wave by enhancing wetting property of the steel. SnPb eutectic disappeared from the plate surface and PbBi eutectic covered the materials on the SUS316 plate. The thickness of the covered PbBi layer was 10 to 20µm.

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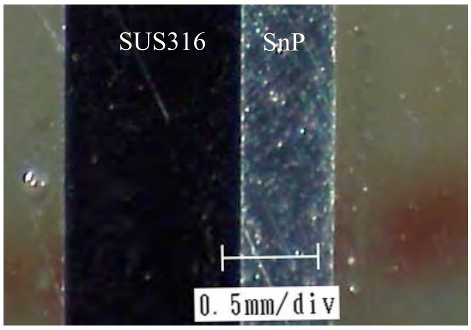
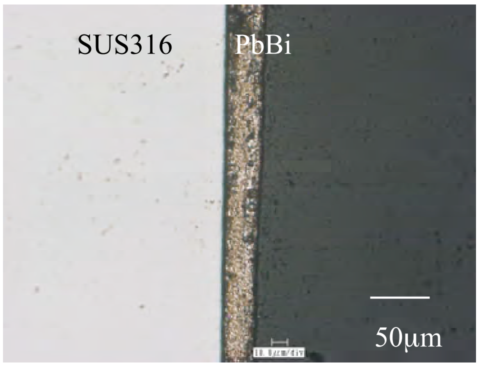


(a) Before immersion



(b) After 60 hrs test.

Fig.5 the cross-section of the plates.